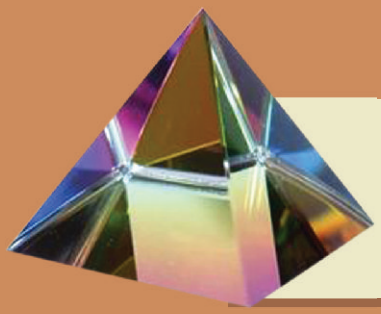

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A Comparative Study on the Levels of Stress and the Coping Strategies used by Female Arthritis Patients (40-60 years) Pre and Post Surgery

Sneha Patel and Krishnakali Bhattacharyya

ABSTRACT

When the individual finds she has to undergo a surgery for arthritis she perceives stress and stress calls for some coping. The aim of the present study is to compare the level of perceived stress prior and post surgery and the coping strategies used before and after an operation for arthritis in female patients. The sample consisted of 30 voluntary participants aged between 40-60 years. One day prior to the operation they were given a questionnaire on stress (Perceived Stress Scale) and a self prepared questionnaire on coping strategies to fill. This was repeated the day after their operation. The obtained results were analyzed by finding out the mean, standard deviation, and t value. It was found that there is a significant difference in the level of perceived stress prior and post surgery among female arthritis patients. The level of stress decreased significantly after surgery. There is significant difference in the use of coping strategies also.

Keywords: Stress, Perceived Stress, Operation/Surgery, Coping Strategies, Arthritis

Introduction

Arthritis is a chronic progressive autoimmune disease, mainly categorized by the inflammation of joints, due to the attack of immune cells on synovial tissue of joints, leading ultimately to pain and irreversible damage. Such a patient experiences, fatigue, weakness, along with unbearable pain in their joints, which are also major symptoms of stress, thereby affecting their health and well-being, along with daily life activities. The prevalence of arthritis is 1-2% worldwide, of which the ratio of prevalence of the chronic illness between females and males is 3:1. On consulting a team of doctors, it was found that of these patients, 0.02-0.03% of the patients, undergo surgery. Many studies conducted have shown a close relationship between psychological distress and deteriorating health amongst arthritis patients. This study aims to assess the level of stress undergone by female arthritis patients (40-60 years) before and after surgery, and the coping strategies used by them. The study was conducted on a sample of 50 female patients of which only 30 patients completed the two questionnaires provided. It was found that the level of stress is higher prior to operation as compared to post operation. It was also found that the female patients tended to use more of defense oriented coping strategies, as compared to task oriented coping strategies.

Methodology

The study was conducted on the middle aged female arthritis patients residing in Kolkata, who were to undergo surgery. In total 50 patients were approached– out of which 30 patients participated, and completed questionnaires. Blood pressure was measured and taken into account, since it is one of the symptoms of stress. The patients were also asked to fill in two questionnaires, Perceived Stress Scale and a self-preparatory questionnaire, before and after surgery. The results were compared and analyzed.

Result and Discussion

Arthritis is one of the chronic illnesses, that is on the rise. The disease in simple terms means inflammation of joints, which leads to pain, problem in physical mobility, rise in stress levels, and leads to further difficulties faced by the individual, especially when a person is a middle aged individual, when he or she is already going through a transitional phase from young adulthood to old age, a period, when he or she is prone to get many chronic illnesses. Arthritis is found more in women than in men, and when individuals need to undergo an operation for the illness, it not only leads to increase their level of stress which leads to various physiological reaction in their body such as increase in weight, increase in blood pressure and increase in blood sugar levels. When this happens they begin to employ various coping strategies.

It has been revealed that there is a close linkage between stress and the decreased activity in an arthritis patient, especially on how the stress increases the problems faced by an arthritis patient through their immunity system, leading to increase in pain, fatigue and functional disability. Female participants were approached while they were

admitted to nursing home with their ailment on the day prior to the surgery. In this context it may be said that all the patients were overweight and suffered from hypertension. First they were administered Perceived Stress Scale to measure their level of stress. Analyzing the results it was found that the perceived stress level was high among the patients prior to surgery (Table 3), however it was reduced significantly after surgery as reflected in the obtained t value. This may be because of the successful operation has reduced the constant pain, care given by the medical staff and the physiotherapy provided. This also may be because of the thought that they will not be a burden on their family, friends and colleagues any more. Lack of mobility is a major source of stress which is significantly reduced after the successful operation and the medical care provided. This reassures the patient of carrying out her daily life activities as she can do earlier.

The results of the present study found a significant decrease in the perceived stress level from pre to post operation period. 27

Table 1: Showing Mean, Standard Deviation, Range and t Value of prior and post surgery of stress level

Conditions	Mean	Standard Deviation	Range	t Value	H_0
Prior Surgery	29.4	2.859	12	16.796**	Rejected
Post Surgery	26.4	3.689	15		

** ($p<0.01$)

Taking into account the significant features which have been noted in the scores of the questionnaire, for example, in the question In the last month how often have you been upset because of something that happened unexpectedly, the total stress prior to the operation is 70, and post operation is 57. This may be because majority of the female patients were not expecting themselves to be suffering from a chronic ailment as arthritis, let along being it at such a severe stage that they would have to undergo a surgery. This alone is a bid unexpected news for them, which acts as a stressor, creating distress among the patients. Though on the other hand, the successful operation may be one of the reasons behind the decrease in stress level, along with the participants expecting medical treatments and care, after the surgery. In the question In the last month how often have you felt nervous and 'stressed' the level of stress is higher in the case of pre-operation as compared to pos-operation. This may be because, prior to the surgery, the participants may have been nervous regarding the operation and apprehensive about the success of it. Increasing pain and lack of mobility adding to the joint pain, leading to stress before surgery may have been replaced by the hope of mobility after surgery. The post operation care and treatment provided by the medical staff and the increase in the number of visitors after operation might have also added some positive effect. For the question…In the last month how confident have you felt about your ability to handle your personal problems, the level of stress prior to surgery is higher as compared to post surgery. The female arthritis patients may be facing this due to the inflammation of joints leading to immense pain and lack of mobility, along with fatigue, and distress. But it may be the successful operation has significantly made them optimistic about reduction of their pain, and the medical care and treatment, along with physiotherapy has thrown a ray of light with scope of mobility.

Secondly they were administered a self prepared questionnaire to measure the usage of coping strategies by the female arthritis patients. Analyzing the results it was found that the coping strategies are highly used in the case of pre-operation stage (Table 3, 4 and 5), however it was reduced significantly after surgery as reflected in the obtained t value.

Table 2: Showing Mean, Standard Deviation and t Value of defense oriented coping strategies used prior and post surgery

Conditions	Mean	Standard Deviation	t Value	H_0
Prior Surgery	47.07	3.123	20.578**	Rejected
Post Surgery	32.63	3.24		

** p value<0.01

Table 3: Showing Mean, Standard Deviation and t Value of task oriented coping strategies used prior and post surgery

Conditions	Mean	Standard Deviation	t Value	H_0
Prior Surgery	31.37	2.639	10.617**	Rejected
Post Surgery	20.4	2.823		

** p value<0.01

The word 'coping' implies 'an individual's behavioral and cognitive attempts to manage stress' (Lazarus and Folkman, 1984).[8] The coping strategies used by the female arthritis patients are of two types – task oriented coping strategy and defense oriented coping strategy.[6] As it can be observed from the results (Table 3 and 4), the defense oriented strategies are used more than task oriented strategies in both the cases. A study conducted by T. Covic et. al . reported that arthritis patients tend to cope up with arthritis by resting and worrying, clearly showing the use of defense oriented mechanisms rather than the use of task oriented strategies.

It has been observed that in both cases, the participants tend to use defense oriented coping strategies. From the data collected it has been observed that prior to surgery they employ coping strategies such as accepting sympathy and understanding of others, telling themselves many things to make themselves feel better, fantasizing things to make themselves feel better, spending time with family and friends and praying more than earlier. A.C. Jones, K. Kwoh and his colleagues[7] found that amongst the different races, the defense oriented strategies, especially in form of hope and prayers were more prevalent among the African-Americans, than the white Americans. As compared to this, in the present study the post surgery patients used techniques such as accepting somebody's sympathy and understanding, telling themselves things to feel better, keeping in touch with their feelings and letting them go, spending time with family and friends, taking out their anger on others, and feeling more tired than they used to making them fall to sleep more often. In both the cases, it has been seen, that social support is an important factor even when the female arthritis patient is using defense oriented coping strategies to cope with stress. 'Social support' can be broadly defined as the interaction or the resources provided by others to help the individual cope with the stressor. The effective coping to the rising stress, in arthritis, requires external sources of social support, such as medicines, medical facilities, and post-surgery therapeutic facilities, along with a proper diet, and family support for the emotional needs of the patient. Most studies conducted have shown the positive effects of social support on an arthritis patient, the availability and approach of the social support has an effect on the psychological distress of the patient, reducing his stress level.[9]

Regarding the task oriented strategies; they were used lesser as compared to defense oriented coping strategies (Table 4 and 5). However majority of the patients never blamed themselves for their suffering. Analyzing the results the present study it was found that the coping strategies are highly used in the case of pre-operation stage, however it was reduced significantly after surgery as reflected in the obtained t value (Table 6).

From the data collected in the study at hand, it has been observed that prior to surgery, the respondents have used strategies like making plans of action to follow step by step, doubling their efforts knowing what has to be done, letting their family and friends help out, and exercising and meditating to relax and vent out their stress. In a study conducted on 59 women suffering from arthritis, the extent of the perceived stress, social support, coping with disease was explored to understand physical, psychological and social

adjustment in the female arthritis patients. It was found that perceived stress was a better predictor of level of positive and negative emotions of the individual. It was also found that a cognitive behavioral therapy to facilitate a patient's adjustment could be used for the management of stress to enable adaptive coping strategies and utilization of social support resources, amongst the people suffering from arthritis.[3]

There is a decrease in the use of task oriented coping strategies after operation. Prior to operation they reported to educate themselves for instance to tackle the problem. The response to this statement in coping strategy questionnaire changed in post operation period. After operation, the patients mostly coping strategies such as making plans in action to follow step by step, tackling the problem at hand, knowing what has to be done and doubling their efforts to tackle the problem, letting the family and friends help them out and diligently following the instructions of the doctor.

In a study conducted by P.P. Katz on 511 arthritis patients, it was found that accommodation, active remediation, social support and perseverance were mainly used coping methods while coping with stress due to arthritis and doing daily activities.[6] A study conducted on fifteen arthritis patients by T. Uhlig et. al. explored the effect of tai chi method of exercise on the health and stress of the participants. It was found that the arthritis patients experienced improved mobility, improved health, balance, and less pain during exercise and daily life along with stress reduction helping them to cope up with stress.[10]

Table 4: Showing Mean, Standard Deviation and t value of overall Coping Strategies used prior and post surgery

Conditions	Mean of Overall Coping Strategies	Standard Deviation of Overall Coping Strategies	t Value	H_0
Prior Surgery	78.43	3.765	25.578**	Rejected
Post Surgery	52.96	4.542		

** p value<0.01

Overall, it can be observed that the female patients use more of coping strategies prior to surgery, which decreases significantly (Table 5 and 6). After operation it may be said that since they undergo a higher level of stress prior to operation as compared to post operation, they use more coping strategies before surgery as compared to after surgery. In a study conducted on 26 people of the age group 42-78 years by Y. Iwasaki, and group, various coping strategies used by middle aged and older people revealed that the arthritis patients used various coping strategies such as keeping oneself active and busy, educating oneself about arthritis, socialization, spiritual coping, helping others and doing leisurely activities helped to cope with the distress.[4] In another study conducted it was found that leisure helps to positively divert the mind of the arthritis patients helping them to cope with stress, along with rejuvenation and renewal, by promoting life balancing facilitating resilience, and capacity to proactively cope with the stress due to the illness.[4] Thus, the study conducted reveals that the female arthritis patients do undergo through distress. It is further revealed that there is a significant difference in the level of stress undergone pre-operation and post operation. A significant difference in use of coping strategies, were also observed from pre operation to post operation stage. It was however noted that in both pre and post operation stages the respondents used defense oriented coping strategies more than task oriented coping strategies.

Conclusion

Chronic illness and operation brings about stress. When a woman hears that she is suffering from a chronic ailment and has to undergo an operation, she cannot put off her stress. This study has been conducted to compare and analyze the level of stress, before and after operation among female arthritis patients (40-60 years old). The result of the present study reflects that, the female arthritis patients undergo stress. Moreover, the study found that there is a significant difference in the level of stress undergone by the participants, pre and post surgery. The level of stress is has reduced post surgery, which may be due to the successful operation and medical care and treatment provided.

Stress calls for coping mechanisms It can be concluded that with undergoing through stress due to arthritis, the participants have employed both task oriented and defense oriented coping strategies. The study reveals that there is a significant difference in the use of coping strategies by the female arthritis patients, where they have used more coping strategies prior to surgery, as compared to post surgery, due to the fact that they experience higher level of stress prior to surgery as compared to post surgery.

Overall, it has been seen that the female arthritis patients who volunteered for the present study have shown significant reduction in their stress level after operation. Their nature of using coping strategies also changed from prior to post operation showing the use more of defense oriented coping strategy as compared task oriented coping strategy.

Reference

1. Carson R.C., Butcher J. N., Mineka S. and Hooley J.M. "Abnormal Psychology, Chapter 5: Stress and Adjustment Disorders". 13th Edition. Pp. 150
2. Covic T., Adamson B. and Hough M. "The impact of passive coping on Rheumatoid Arthritis pain." Rheumatology. Volume 39. (2000) pp. 1027-1030.
3. Curtis R., Groarke A.M., Coughan R., and Gsel A. "Psychological Stress as a predictor of psychological adjustment and health status in patients with rheumatoid arthritis" Patient Education and Counselling. Volume 59(2). (2005) pp. 192-198.
4. Iwasaki Y. and Butcher J. "Coping with arthritis among Middle-Aged and Older Women and Men with Arthritis." The International Journal of Psychosocial Rehabilitation. (2004)
5. Iwasaki Y., MacTavish J. and MacKay K. "Building on strengths and resilience: leisure as a stress survival strategy." British Journal of Guidance and Counselling. Volume 33(1). (2005) pp. 81-100
6. Jones A.C., Kwoh K., Groeneveld P.W., Mor M., Geng M., Ibrahim S.A. "Investigating Racial Differences in Coping with Chronic Osteoarthritis Pain" Journal of Cross-Cultural Gerontology. Volume 23 (4). (2008) pp 339-347.
7. Katz P.P. "Use of self-management behaviors to cope with rheumatoid arthritis stressors". Arthritis and Rheumatism Volume 53(6). (2005), pp.: 939-949. [http://www.ncbi.nlm.nih.gov/pubmed/16342099]
8. Proefschrift, Evers A.W.M. "Predictors of Disease Outcome in Rheumatoid Arthritis: The Role of Illness Cognitions, Coping and Social Support." Pp. 10 – 17.
9. Tong A., Jones J., Craig J.C., and Singh-Grewal D. 'Children's experiences of living with juvenile idiopathic arthritis: A thematic synthesis of qualitative studies.' Arthritis Care and Research.Volume–69(9). (2012)pp. 1392-1404. [http://www.ncbi.nlm.nih.gov/pubmed/22504867]

To Study the Impact of Sex Education on Sex Awareness among Adolescent Girls

Niketa Chokhani & Krishnakali Bhattacharyya

ABSTRACT

Adolescent is an evolutionary process of human development which commences biologically with changes as the physiology of pubis and completes psychological with ultimate organization of sexuality. The present study aimed to determine the effect of sex education on sex awareness among adolescent girls, by studying 162 girls between 15-18 years from two Bengali medium government aided school and an English medium private school of Kolkata who have given permission to conduct the study. Finding out that the girls being studied have high attitude (positive) towards sex education by administering Sex Attitude Scale by Dr. Usha Mishra, the study proceeded to enquire about their awareness and knowledge regarding various issues relates to sex, sexuality, and self-defense (pretest) through a self-prepared questionnaire. Then a program on sex education was introduced through a power point presentation and discussion. The impact of this program was studied by administering the self-prepared questionnaire on awareness and knowledge for the second time. It was found that the program which intervened between pre and post test has a significance effect on all the three groups of girl being studied. Thus it reflects the urgency of sex education among adolescents' girls.

Keywords: Adolescent, contraception, menstuatration, puberty, relationship abuse, self-defence tips, Sex Education, sexually transmitted disease, womens' legal rights.

Introduction

WHO defines adolescence as the period of life between 10-19 years. It is a period of rapid physical and biological changes, which may lead to confusion, tension, frustration, and feeling of insecurity. Adolescence is said to be a period of stress and strain. It is the period in which foundations can be laid for a long and healthy life. Adolescence is a prime time for health promotion and for establishment of healthy behavior that will influence health in later years.[1] It is a time of sexual exploration and incorporating sexuality into one's identity. They wonder whether they are sexually attractive how to behave sexually and what the future holds for their sexual lives. Most adolescents eventually manage to develop a mature sexual identity, even though, as adults can attest, there are always times of vulnerability and confusion along life's sexual journey.[5] Therefore lack of accurate information and the absence of proper guidance and counseling may lead to various behavioral and reproductive health problems.

Sex education is an educational program aimed at promoting individuals fulfillment in personal living and in his family and social relationship by integrating sexuality into the total personality. Sex is a very sensitive subject still considered to be taboo in Indian society and a topic not to be discussed openly perhaps between husband and wives. 3 Absence of proper guidance and counseling may lead to various behavioral and reproductive health problems. Most of the parents and school authorities feel that imparting sex education means making children promiscuous, that it let them think of sex as permissible and leads to experimentation.[4]

Hypothesis:

H_0: There is no significance impact of Sex Education program on adolescent girls.

H_1: There is significance impact of Sex Education program on adolescent girls.

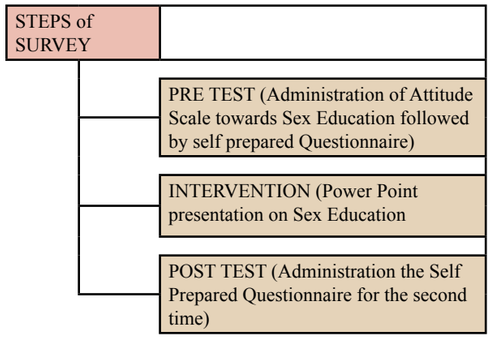
Methodology

The present study aimed to determine the effect of sex education on sex awareness among adolescent girls, by studying 162 girls between 15-18 years from two Bengali medium government aided school and an English medium private school of Kolkata who have given permission to conduct the study. Two questionnaires were used namely the Attitude towards Sex Education Scale by Dr. (Smt.) Usha Mishra (pretest), to study the attitude of the students towards sex education, and a self prepared questionnaire to enquire about their awareness and knowledge regarding the various issues relates to sex , sexuality, and self defence (pre-test). Then a program on sex education was introduced through a power point presentation (intervention) and discussion was followed. The impact of this program was studied by administering the self-

prepared questionnaire (post test) on awareness and knowledge for the second time.

Method of Data Analysis: The data collected by administering Attitude towards Sex Education scale is analyzed using Mean and Standard Deviation.

To analyze the data collected through self prepared questionnaire the following the Test of Significance of Difference of Proportion was used.[2]



Results and Discussion

The present study dealt with the impact of sex education on sex awareness among adolescent girls. For this the attitude toward sex education was determined first, following which a self prepared questionnaire twice was being administered: intervened by a power point presentation on sex education accompanying discussion. The results obtained are discussed in details under two major headings as follows-

Results obtained from attitude towards sex education scale: Accepting sex education is a prohibited and tabooed subject for discussion in India, probing into the matter necessitates in the first instance to know the respondents attitude regarding the area. The results obtained from the present study is given in the table below:

Table 1: Showing the Mean, Standard Deviation and Stanine score of Attitude Scale towards Sex Education

Group	N	Mean	Standard Deviation	Stanine	Comment
1A	60	100.5	11.17	7	High
1B	60	105.9	6.80	7	High
2	40	113.3	20.85	8	High

- From the above table it is seen that all the three groups of girls have mean value above 100 in attitude scale, which can be interpreted as high as the norm, i.e., Stanine score is 7 to 8. This further indicates that they have positive attitude towards sex education.
- Taking into account individual data sheets it was found that the majority of the respondents in all the three groups "strongly agree" with the statements like "sex education helps the students to understand sex related problems", "sex education is necessary for eradicating social evils.
- Also majority of them "Disagree" with the statements like "sex education should not be an essential part of formal education at all levels", and "strongly disagree" with the statements like "there is no need of sex education before marriage", "sex education would lessen the students interest towards studies".
- These further indicate that students belonging to the three groups do not posses negative attitude towards sex education.
- Reflection of this positive attitude thus paves the way for the present study to proceed further for knowing awareness and knowledge on various aspects related to sex and sexuality of these adolescent girls.

Results obtained from self prepared questionnaire

Table 2: Showing overall Pre and Post Frequency of correct answer and "t" value

Groups	Pre Test Frequency Of Correct Responses	Post Test Frequency Of Correct Responses	"T" Value	Null Hypothesis (H_0)	Alternative Hypothesis (H_1)
1.A	1206	2002	3.2 **	Rejected	Accepted
1.B	1536	2324	3.37**	Rejected	Accepted
2	1187	1672	4.65**	Rejected	Accepted

** P (<0.01)

- It was found that their level of awareness was not up to the mark. These girls had lack of knowledge in various areas like relationship, sexually transmitted diseases, various myth related to mensturation, the ways to defend oneself and their legal rights and most importantly when to say 'NO" to avoid unwanted pregnancy.
- The study conducted also showed their preference source for their information were their mother on whom they depended for the information like growing up, body changes, and their friends for the information like about kissing and dating but it was seen that their least preferred source for their information was father.
- Study conducted showed that the program which intervened between pre and post test with the aim to impart knowledge regarding sex education has a significant effect on all the three groups of adolescent girls being studied.

Overall, the results of the present study thus reflects that a highly debatable area of education, that is sex education , if handled carefully and with passion can not only prove fruitful in bring about change in awareness can also benefit the young minds to lead a healthy life.

Conclusion

The present study finding out that the group of girls being studied have positive attitude towards sex education aimed in understanding the level of awareness among them .it was found that their level of awareness was not up to the mark. These girls had lack of knowledge in various areas like relationship, sexually transmitted diseases, various myth related to menstuaration, the ways to defend oneself and their legal rights and most importantly when to say 'NO" to avoid unwanted pregnancy.

The study conducted also showed their preference source for their information were their mother on whom they depended for the information like growing up, body changes, and their friends for the information like about kissing and dating but it was seen that their least preferred source for their information was father.

In the debates it is always argued that sex education doesn't have any impact on the knowledge and also may corrupt the mind of children. But the result of the present study conducted showed that the program which intervened between pre and post test with the aim to impart knowledge regarding sex education has a significant effect on all the three groups of adolescent girls being studied. Therefore Sex education in India is need of the hour; need to impart such education to adolescents so they can learn about sexuality without hesitation.

Reference

1. Allen, Louisa, ' Say Everything: Exploring young Peoples suggestion for imparting sexuality education" ", Sex Education, Vol 5(2), (2005), pg. 389-404
2. Gupta S.C, "Fundamentals of Statistics" Pg. no. 17.5 -17.9
3. Jayathambal,P, Devi, Nalina K. , "Adolescents Awareness on Reproductive Health Care and Sex Education", Research Highlights,(2002), pg.68-74
4. Rangaiyan, Gurumurthy, Verma, Kavi, K., "Reproductive Health, Sexual Activity and Condom Use- Knowledge among male College Students in Mumbai- 1993-1994" The Indian Journal of Social Work, (4), (2005) Retrieved from: (http://paa2004.princeton.edu/papers/41546)
5. Santrock John W., Adolesence, 11th edition, Tata McGraw-Hill .

Development of Guava gel Candy fortified with amaranth leaf powder using different gelling agents.

Ankana Sengupta, Damanjeet Kaur

ABSTRACT

The "super fruit" guava, a rich source of ascorbic acid was developed into a gel based candy and fortified using various levels of dehydrated amaranth leaf powder. Guava gel candy fortified with 10g of amaranth leaf powder was the most acceptable. Ascorbic acid content of the gel candy was significantly reduced from that of the guava pulp (223.78mg/100 g) owing to the heat processing for the preparation of the candy (31.25mg/100 g). Hence variations of gelling agents like gelatin and agar agar had been used apart from pectin, which required much lesser heat treatment to set the gel candy, thereby retaining ascorbic acid (93.33mg/100 g) & (45.7mg/100 g) respectively. The iron content of the fortified guava gel candy had significantly reduced (0.2-0.35mg/100 g), as compared to the fresh amaranth leaves (4.9mg/100 g) and dehydrated amaranth leaf powder (3.2mg/100 g) although there has been a significant increase in iron content as compared to basic gel candy. It was seen that the zinc content significantly increased on fortification. It can be concluded that the developed and fortified guava gel candies when refrigerated could be stored up to 15weeks without any spoilage.

Keywords: Amaranth, Candy, Fortification, gelling agent, Iron –rich.

Introduction

Micronutrient malnutrition (MNM) is widespread in the industrialized nations. In 2002 Iron deficiency Anemia (IDA) was considered to be among the most important contributing factors to the global burden of disease.[5]

Guava is a seasonal fruit and a popular among the people of all social strata due to its comparative low price than some other fruits.[8] Its harvesting period is short and highly perishable. Once fully ripens, its marketing quality deteriorates[2] hence, guava fruits cannot be kept for longer time unless preserved properly.[6] The development of low cost processing technology of guava is highly required, to benefit both the producer and the consumer.[4] It has wide applications in juice, nectar, pulp, jam, jelly, marmalade, slices in syrup, fruit bar or dehydrated products, as well as being used as an additive to other fruit juices or pulps and also sauces, ice cream, butter, chutney.[7]

The high content of vitamin C in the "super fruit" guava[7,8] makes it a powerhouse in combating free radicals and oxidation thus preventing many degenerative diseases and thus can be used to fortify children foods.[42] Thus, there is need to popularize more processed products of guava with the view to exploit its nutritive goodness.[1]

Leafy vegetables are the most affordable and sustainable dietary sources of vitamins, minerals and other bioactive compounds are highly perishable thus requiring special processing treatments to prevent post harvest losses which are preserved by canning, freezing, dehydration and blanched to obtain good quality products.[10]

Amaranth leaves being cheapest are available in all seasons. The high non heme iron content has been selected to provide iron security by incorporating in traditional products.[12] Ascorbic acid (AA), with its reducing and chelating properties, is the most efficient enhancer of non-heme iron absorption when its stability in the food vehicle is ensured. The promotion of iron absorption in the presence of AA is more pronounced in meals containing inhibitors of iron absorption.

There is an increasing demand for healthy products, natural and high quality, among consumers. Hence, it is important today to develop new nutritional food, maximize their nutrient content in both processing and storage and extend the shelf-life, thus to meet the requirement of the market. In this regard, the information on nutrient change in processing and storage will be of great importance.[9]

Fortification of suitable food vehicles with absorbable forms of iron is a highly desirable approach in controlling iron deficiency.[40] Nutrient losses are bound to occur during food processing & storage. The additional cost of fortification should be reasonable for the consumer.

Candies are popular products among children and adults and their versatility is visually alluring as well as pleasing to the consumer.[3] Different studies on development of fruit candies by steeping or soaking in sugar syrup, especially tamarind candy[13], amla, papaya, watermelon, pineapple, carambola rind candies have been established.[11] But information on preparation of gel candy from guava fruit is lacking.

Methodology

Guava and Amaranth leaves were purchased from local market. Pectin, Gelatin & Agar agar were purchased from Dalhousie whole sale market.

Product Development: Amaranth leaves were washed and were dried in Hot Air Oven at 50^0C for 5 to 6 hours. Three different trials of guava gel candies had been carried out to develop a basic recipe. variations were made by addition of different levels of dehydrated Amaranth leaf powder, i.e. (2.5 gm, 5 gm,10 gm, 12.5 gm, 15 gm) to Basic Recipe 3 (BR3). Variations were also made with different kinds of gelling agents, namely pectin, gelatin and agar.

Sensory Evaluation: The basic recipe candies and the fortified candies were presented for sensory analysis using a 9 point hedonic scale on the basis of appearance, color, taste, odor and overall acceptability to a panel of 20 members.

Physicochemical properties: The chemical analysis of jam was carried out by evaluation of different chemical properties, such as pH value, Titrable acidiy, TSS, Insoluble Solids, Total sugar, Ascorbic acid, Iron, Calcium and Zinc estimation. The pH value of the samples were measured with a pH meter at room temperature, calibrated prior to buffer solutions of pH value 4.0 and 7.0. Titrable acidity is calculated as *(Ta = B x 0.1 x 0.064 x 100/W)*
Where, *Ta* is titrable acidity; *B* is reading burette; *W* is weight of sample. TSS is calculated as *Grams of Total Soluble Solids/100ml Filtrate = Specific gravity -1/0.00386*. Insoluble solids are calculated as: *Percentage of fruit content = Insoluble solids in sample/ Insoluble solid in fruit x 100*. Total sugars, reducing sugar and non-reducing sugar was carried out through Lane and Eynon Method.

Ascorbic acid is determined by titrating with 2, 6-dichlorophenol indophenols and Meta phosphoric acid which is blue in alkaline solution and red in the acidic solution is reduced to colorless form.

Iron was estimated at an absorbance of 458nm by plotting standard concentration versus absorbance, using 2M HCl and Potassium Thiocyanate.

Calcium & zinc content were determined by the Complexometric Titration with EDTA.

The developed food product was stored in two different conditions in a polythene zip lock pouch to check the shelf life. It was stored at room temperature and refrigeration temperature at 4oC. The product was checked for the span of 3.5 months. The microbial load of the product was determined by staining with Malachite green and saffranin dye, which was then observed under microscope.

Statistical analysis: A t-test was conducted, which is any statistical hypothesis test in which the test statistic follows a Student's t distribution.

Results and Discussions

Three different recipes were attempted to obtain the most acceptable recipe by the panel members. BR3 obtained the highest score as compared to BR 1 and BR 2 and proved to be the most acceptable product developed. BR1 and BR2 did not have a well set and a firm texture. Addition of milk powder did not allow pectin to improve the texture, again leading to a sticky product. Basic recipe (BR3) when fortified with 10g of amaranth leaf powder obtained the highest score. BR3 fortified with 15 g of amaranth leaf powder obtained the lowest score. Excess addition of amaranth leaf powder hindered the formation of the gel structure, leading to poor flowy texture.

9 point Hedonic Scale Comparative Score of variations of gelling agents used for candy preparation revealed that FC [P] had been attributed the maximum score. Although the texture of guava candies prepared using gelatin was very well set, the main drawback is that it is obtained from a non-vegetarian source. The candies prepared using agar agar had a comparatively brittle gel like structure, with lower gel strength, leading to a low score in its texture. All the gelling agents used for candy preparation were quite acceptable.

Guava contained 240.4 mg/100 g of Ascorbic acid, which reduced to 223.78mg/100 g when made into pulp, due to oxidation of ascorbic acid.[8] Further processing of the guava pulp to develop candy at about 110 to 117^0C using pectin resulted in retention of only 25mg/100g and 31.24mg/100 g in BC [P]

and FC [P] respectively. Longer the processing time and heat treatment, higher was the ascorbic acid loss. Guava candy fortified with amaranth leaf powder aids in better retention of ascorbic acid. The ascorbic acid content in BC [G] and FC [G] resulted in the retention of 90mg/100 g and 93.33mg/100 g respectively, since gelatin required much lesser heat treatment. The BC [A] and the FC [A] also retained 45.7mg/100gm and 45.99mg/100gm ascorbic acid respectively. Statistical analysis showed that the level of ascorbic acid is not significantly higher in the fortified candy.

The moisture content of fresh guava was 82.5%, fresh amaranth leaves was 89.25% whereas that of dehydrated amaranth leaves was significantly low, i.e. 3.3%. The BC [P] had a moisture content of 37.23%, whereas the FC [P] showed moisture content of 36.69%. The moisture content of the BC [G], FC [G], BC [A] and FC [A] were not possible to estimate, since on heating, gelatin and agar agar melted, which on cooling again solidified.

Ash content of Guava yielded 1.6% of ash; fresh amaranth leaves yielded 3.13% ash, and dehydrated amaranth leaves yielded 32.83% of ash. The BC [P] and FC [P] yielded 22.5% and 22.47% respectively. Similarly, the BC [G] and FC [G] yielded 22.02% and 22.52% ash respectively. Whereas, BC [A] and FC [A] yielded 19.68% and 19.75% ash respectively, as shown in Fig 5.6, which was further used for mineral estimation. Removal of moisture during drying may have resulted in an increase in concentration of nutrients in the remaining mass.

The pH value of the guava pulp was 6.3. But the pH value of BC [P] and FC [P] resulted in a significant reduction in pH value of 3.8. The pH value of the BC [G] was 4.5 whereas; the FC [G] had a lower pH value of 3.9. Similarly, the pH value of the BC [A] was 4.9 whereas; the FC [A] had a lower pH value of 3.7.

The titrable acidity of the BC [P] was 0.95% and that of FC [P] was 0.7%. BC [G] showed the titrable acidity of 1.09%, whereas the FC [G] had 0.9% of titrable acidity. The BC [A] had a titrable acidity of 1.01% and the FC [A] had a titrable acidity of 0.99%. Total acidity must not exceed 1% since it leads to syneresis of the gel. The values of acidity found in all formulations are considered well below this limit. Fortification with amaranth leaf powder reduces the chances of gel syneresis.

Total Soluble Solids (TSS) of BC [P] was 51.8%, whereas the TSS of FC [P] was significantly higher, i.e. 81.75%. Again the TSS of BC [G] was 16.5%, whereas FC [G] was 20.34%. BC [A] had a TSS of 19.72% and FC [A] was 21.92%. The increase in the TSS in the fortified candies as compared to basic candies may be due to the addition of dehydrated amaranth leaf powder.

Results indicated that the percentage of insoluble solids in BC [P] was 25.8% whereas in FC [P] was 30.8%. In BC [G] was 8.3%, whereas in FC [G] was 10.89%. BC [A] had an insoluble solids % of 18.23 and FC [A] had 21.63%.

The total sugar content includes the reducing sugar (glucose) and the non reducing sugar (sucrose). Results showed that the BC [P] had 36.74% of reducing sugar and 9.5% of non reducing sugar, whereas the FC [P] had 36.84% reducing sugar and 9.94% of non- reducing sugar. So fortification did not bring about any significant changes in the reducing and non-reducing content of the guava gel candy. The BC [G] had 28.24% of reducing sugar and 6.18% of non reducing sugar, whereas the FC [G] had 29.88% reducing sugar and 7.02% of non reducing sugar. The BC [A] had 38.2% of reducing sugar and 6.65% of non reducing sugar, whereas the FC [A] had 36.5% reducing sugar and 6.25% of non-reducing sugar. The increase in reducing sugar is mainly due to the hydrolysis of different carbohydrates present in the gel candy.

Iron was estimated at an absorbance of 458nm. the iron content of the fresh amaranth leaves was 4.7mg/100gm, which when dehydrated by oven drying had a comparative lower iron content of 3.1mg/100gm, which may be due to the losses during oven drying process. The iron content in fresh guava as well as BC [P] was negligible. But the FC [P] resulted in an iron content of 0.35mg/100gm. The BC [G] had an iron content of 0.1mg/100gm, whereas the FC [G] contained 0.25mg/100gm. The iron content of the BC [A] was negligible whereas the FC [A] was 0.2mg/100gm. Statistically the level of iron content was significantly higher in the fortified gel candy.

Fresh amaranth leaves has 17.35% of calcium, which when dehydrated had 16.09% of calcium in it. The basic candies did not contain any calcium, whereas, FC [P] had 0.16%, FC [G] had 0.09% and FC [A] had 0.74% of calcium content.

In spite of the amaranth leaves containing appreciable amount of calcium in it [91,6]; the fortified candies did not retain calcium. This might be beneficial for the absorption of iron, after consumption since calcium inhibits the absorption of iron, as it is an inhibitor. Statistical analysis proved that content of calcium is not significantly higher in the fortified candy.

Fresh amaranth leaves contain 10.35mg/mmol of zinc, which on dehydration, retained 9.29 mg/ mmol. The BC [P] contained 1.09 mg/mmol, whereas the FC [P] had a higher zinc content of 7.65mg/ mmol. BC [G] had 2.34 mg/mmol of zinc, whereas FC [G] had 4.4 mg/mmol of zinc. BR [A] had 2.8mg/mmol and FC [A] had 3.5 mg/mmol of zinc. Statistical analysis proved that content of zinc is not significantly higher in the fortified candy.

Table No. 1- Shelf life testing of guava gel candy variations

Samples	**No. of weeks**	
BC [P]	**Room temperature**	**Refrigeration temperature**
	3-3.5	**16.5-17**
FC [P]	4.2	17.5
BC [G]	-	17.5
FC [G]	-	17.5
BC [A]	-	15.0
FC [A]	-	15.3

Basic Candy prepared using Pectin is BC [P], Fortified Candy using pectin is FC [P]. Basic candy using gelatin is BC [G] and fortified candy using gelatin FC [G]. Basic candy using Agar is BC [A] and fortified candy using Agar is FC [A].

Microbial analysis: The growth of Rhizopus sp. on the candies was revealed. Fortified candies had shown a better shelf life.

Conclusion

Candies are popular products among children and adults and their versatility is visually alluring as well as pleasing to the consumer. Heat labile Vitamin C could be retained in gelatine candies than other variations, aiding in better absorption of iron. But the iron content after fortification was lower than assumed. Nutrient losses are bound to occur during food processing & storage. Hence, Attempts to fortify candy using other iron rich sources could be further carried out, thereby critically analyzing the effectiveness of various other natural iron fortificants as well as elemental iron compounds as a fortificant, checking the bio availability of different sources in the product.

Reference

1. Hal Hiwilepo-van; et al; Kinetics of thermal degradation of Vitamin C in Marula fruit (Sclerocarya birrea subsp. caffra) as compared to other selected tropical fruits; 2012; Food Science and Technology. Vol 49(2) 188- 199.
2. Hal Hiwilepo-van; et al; Kinetics of thermal degradation of Vitamin C in Marula fruit (Sclerocarya birrea subsp. caffra) as compared to other selected tropical fruits; 2012; Food Science and Technology. Vol 49(2) 188- 199.
3. Hiremath; et al. Preparation of sapota candy. 2012; Department of Post Harvest Technology, K.R.C.C.H.Arabhavi-591310; International Journal of Food, Agriculture and Veterinary Sciences; Vol. 2 (1):107-112.
4. Hossen S; Effect of different extractions of juice on quality and acceptability of guava jelly. August 2009; Agricultural Engineer, Department of Agricultural Extension; vol 3(4): 27-35.
5. Jain Monika, et al. Iron deficiency and anaemia; 2012; Department of Food Science and Nutrition Banasthali Vidyapith, Rajasthan; Indian Journal of Fundamental and Applied Life Sciences ISSN: 2231-634; Vol. 2 (2).101 -107.
6. Jain P. K. and P. K. Nema. "Processing of Pulp of Various Cultivars of Guava (Psidium guajava L.) for Leather Production". 2007; Agricultural Engineering International: the CIGR Ejournal.Manuscript FP 07 001. Vol. IX.
7. Jain P.K., Quality of guava and papaya fruit as influenced by blending ratio and storage period; 2011; American Journal of Food Technology; Department of Horticulture, Jawaharlal Nehru Krishi Vishva Vidyalay, Jabalpur, MP 482004; India; Vol 6(6) 507 – 512.
8. Kadam Dattatreya M; et al; Evaluation of Guava Products Quality;2012; International Journal of Food Science and Nutrition Engineering; 2(1): 7-11 [47]
9. Kogulan A, L M.Saiger; Guava powder production using pilot scale spray dryer and nutrient retention study.2008; Faculty of Chemical & Natural Resources Engineering University Malaysia Pahang.
10. Mepba H. D, et al; Effects of processing treatments on the nutritive composition acceptance o some Nigerian edible leafy vegetables. 2007; Department of Food Science and Technology, Nigeria; Vol 7(1). ISSN 1684 – 5374.
11. Seema L; Enrichment of traditional foods with green leafy vegetable for iron security of adolescent girls; 2008; Department of Food Science and Nutrition. college of Rural Home Science, Dharwad University of Agricultural Sciences, Dharwad.
12. Seema L; Enrichment of traditional foods with green leafy vegetable for iron security of adolescent girls; 2008; Department of Food Science and Nutrition. college of Rural Home Science, Dharwad University of Agricultural Sciences, Dharwad.
13. Singh Dhiraj; et al. Processed products of tamarind. 2007; Department of Post Harvest Technology; Natural Product Radiance; Vol 6(4)315-321.

Effect of honey on physicochemical properties and acceptability of lemon flavoured black tea

Mukta Gupta and Sonali Ghosh

ABSTRACT

Tea (Camellia sinensis) is known as the most popular beverage in the east, arouses great interest among scientists due to its beneficial health effects. In previous research the health benefits including antioxidant potential of black tea (Camellia sinensis), Lemon (Citrus limon) and honey have been extensively reported. But nothing is reported about the effect of their combined use. The present study was aimed to evaluate effect of Honey on Physicochemical properties and acceptability of lemon flavoured black tea. Data obtained showed that the use of honey in lemon black tea not only increases the level of protein, sugar, and free radical scavenging activity but also potentiates the antioxidant activity of tea increasing antioxidant contents such as phenolics, and ascorbic acid. Honey added lemon black tea resulted in approximately increase 89% protein, 312% sugar 426% ascorbic acid and 369% anti-oxidant activity. An agar disc diffusion assay was used to assess antibacterial activity against Gram negative strain (Escherichia coli). The honey added tea samples were found to inhibit growth of the tested bacteria. Honey lemon tea creaming can be reduced by treating with tannase produced from agro-waste, which has a potential application in removal of haze. Through sensory evaluation, the acceptability of different flavoured tea is also established. Thus honey lemon tea, being a rich source of natural antioxidants, may be used in the prevention of various free radicals related diseases.

Keywords: antioxidants, Escherichia coli, honey, lemon, physicochemical properties, tannase, tea.

Introduction

Tea *(Camellia sinensis)*, is a popular beverage consumed worldwide whose health effects are still being investigated and have received a great deal of attention which could be connected with polyphenol and antioxidative activity, fighting the harmful influence of environmentally generated free radicals.[5]

In tea leaves, the following flavanols collectively known as the catechins are present:

- (+)-Catechins
- (+)-Gallocatechin
- (-)-Epicatechins
- (-)-Epicatechins-3-gallate
- (-)-Epigallocatechin-3-gallate (EGCG)[10]

During the fermentation process of black tea, Catechins are converted to complex condensation products, the theaflavins and thearubigins. They are very powerful antioxidants of black tea.[17]

The tea antioxidants are highly associated with a variety of biological effects, including anti-inflammatory, antibacterial, anti-allergic, anti-thrombotic, and vasodilator actions.[8]

Honey contains a significantly high level of antioxidants, including catalase, phenolic acids, flavonoids, carotenoids, organic acids and ascorbic acid, many of which had been reported to have health function such antioxidantive, antimutagenic, anticarcinogenic, antibacterial and hypoglycemic activities.[12]

Numerous therapeutic properties have been attributed to Lemon (Citrus limon), like anticancer, antiviral, anti-tumor, anti-inflammatory, ability to inhabit platelets aggregation, and age related macular degeneration protective against neuro degeneration.

It has been determined by animal studies that citrus limonoids and derivatives have certain biological activities that may be used as chemopreventive agents for cancer. as it inhibit tumor formation by stimulating the enzyme glutathione S-transferase (GST).[2]

As Tea is one of the most popular non-alcoholic beverages consumed, people prefer adding lemon and honey to it, which increases its palatability, taste, aroma and unknowingly adds antioxidant benefits too.

Although it has already been demonstrated that black tea, lemon and honey have antioxidant activity, nothing is reported about the effects of their concomitant use. That the use of honey potentiates the antioxidant activity of lemon flavoured black tea, which is already a mixture of two powerful antioxidants (lemon and tea), raise lipid peroxidation inhibition properties, free radical scavenging activity.[17]

In this study, those effects were evaluated in infusions of lemon-flavoured black tea with addition of honey.

Methodology

Tea preparation [17]

1 gram of each type of tea was mixed with 100ml boiling water and allowed to stand for 5 minutes. Next filtered and cooled and 8ml honey and 4 ml of fresh lemon juice was added.

Protein assay[21]

Standard protein prepared containing a range of 0.2 to 1 mg/ml BSA. Test sample was prepared by 10 times dilution and by adding Lowry reagent and Folin-Ciocalteu reagent to it. Absorbance was measured at 660nm.

Measurement of total sugar[4]

GOD/POD method was used for the estimation of sugar level. Absorbance was measured at 500 nm.

Measurement of total phenolics[13]

Folin's reagent and sodium carbonate were added to the test sample and the absorbance measured at 765 nm. Gallic acid was used as standard.

Estimation of ascorbic acid content[7]

Standard vitamin C solution and 10 drops of 1% starch solution was titrated against iodine solution, till the first sign of blue colour was seen. The tea samples were titrated in the same way. The vitamin C content was calculated using the formula: $V_1S_1=V_2S_2$.

Tannin assay[3]

Tannin assay was done using BSA, acetate buffer, Sodium dodecyl sulphate- triethanolamine buffer and ferric chloride. Standard curve was prepared with tannic acid. Absorbance was measured at 510 nm.

Evaluation of radical-scavenging activity[15]

This assay was done using the 2, 2-diphenyl-1-picrylhydrazyl (DPPH) assay. The results were expressed as the percentage of reduction (inhibition) of the DPPH according to expression (A0-At)/ A0×100, where A0 is the initial absorbance and at is the absorbance at increasing time t.

Antimicrobial susceptibility test[6]

The different tea samples were tested on the test bacteria (E Coli) using the disk diffusion inhibition method. Sterile paper discs (4mm) were soaked in the different tea samples and placed on the agar plates containing test bacteria and incubated at 37°C for 24 hrs. The results were expressed as millimeters by the mean inhibition zone diameter formed around the disk.

Production of tannase from agriculture waste and treatment on different tea samples

Substrate pre- treatment[16]- Red gram husk was used as agriculture waste. A strain of Aspergillus niger was used for the study. Spores were raised on potato dextrose agar slants sporulation medium.

Fermentation and extraction- Five gram red gram husk was moistened with 10 ml of salt solution. The contents were sterilized and inoculated with 1ml of Aspergillus niger spore inoculums and incubated at 37°C for 72 hours. Tannase was extracted by adding buffer, and PMSF crushed with mortar and pestle in cold. After centrifugation and filtration, filtrate was stored for estimation of tannase activity.

Tannase activity assay[20]- A standard curve was prepared using gallic acid. The enzyme reaction mixture was prepared by the addition of methyl gallate, citrate buffer and crude enzyme. Methanolic rhodanine and potassium hydroxide were also added. Absorbance was measured at 520nm.

Treatment of Tea with Purified Tannase[18]- To 1ml of tea 100ul of tannase (193U/ml) was added. Test tubes were then incubated in 37^0C upto 120 min. After that tubes were incubated in water bath at 50^0C for 10 min. to deactivate the enzyme.

Sensory evaluation[19]- The organoleptic evaluation of controlled and lemon flavoured honey black tea was performed by panel of 30 semi-trained members using 9-point hedonic scale.

Statistical analysis- All data are presented as the mean ± SD of three independent tests. The t test was used to evaluate the difference between treated samples and controls.

Results and Discussion

Determination of protein content- It was observed that the protein content of commercially and freshly prepared lemon flavoured black tea with honey

was increased by 81% and 89% respectively when compared with the untreated control tea ($p<0.001$). This is due to addition of honey and lemon in tea. The protein content of honey is normally less than 5.00 g/kg.[11] The total protein content of honey is depend on their botanical or geographical origin, enzymes introduced by either the bees or other substances derived from the nectar and storage time. The protein content was also increased in commercially available lemon flavoured black tea by 59% and freshly prepared lemon black tea by 34%. This may be because lemon fruit also content some amount of protein.

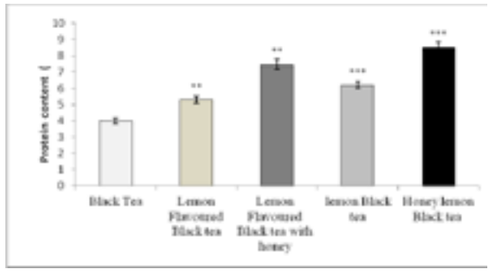


*Figure 1 - Effect of honey on Protein content in different tea samples. The experiments were performed three times, and the mean values ± S.D. (error bars) have been shown in the lower panels (***, p< 0.001 and **, p < 0.01 versus control tea).*

Total Sugar- The total sugar content was increased significantly ($p<0.001$) in commercially Lemon flavored black tea with honey and freshly prepared Honey lemon tea by 290 % and 312 % as compared with the untreated control tea. The sugar level was increased due to addition of honey, which is a good source of simple sugar. Sugar accounts for 95-99% of honey dry matter. Majority of these simple sugars are D-fructose (38.2%) and D-glucose (31.3%), which represents 85-95% of total sugars.[1] No significant changes were seen in lemon flavoured black tea and lemon black tea.

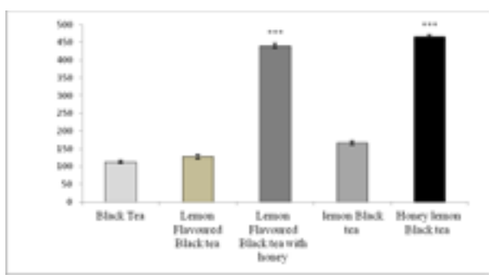


*Figure 2 - Effect of honey on sugar content in different tea samples. The experiments were performed three times, and the mean values ± S.D.(error bars) have been shown in the lower panels(***, p< 0.001 versus control tea)*

Total Phenol

It was observed that total polyphenol was increased significantly ($p<0.01$) in freshly prepared honey lemon black tea by 13% which is not very much significant as compared with the untreated control tea. As honey has polyphenol, adding it to tea further increases the total polyphenol content. The antioxidant activity of natural honeys and tea depends largely on their chemical composition, such as phenolics, flavonoids, enzymes, organic acids, amino acids, ascorbic acid, and carotenoids as well as their origins.

In the commercially bought lemon flavoured black tea and lemon flavoured black tea with honey, the Phenolic content was significantly ($p< 0.001$) lower by 39.59 %, and 52.4 % than the untreated control black tea. This may be because flavoured black teas undergo further processing which might be causing a loss in their Phenolic content.

Ascorbic Acid Assay

It was found that ascorbic acid increased significantly ($p < 0.001$) in all the tea samples. It increased in freshly prepared Lemon Black tea and Honey lemon black tea by 426.3% and 518 % as compared with untreated control black tea. Fresh lemon juice and honey was added which increased the total vitamin c content. Lemon has a wide range of antioxidants such as (ascorbic acids, limonoids, flavonoids) and acts as antioxidants by scavenging and quenching free radical generated in the body.[14]

Whereas in commercially bought Lemon flavoured black tea and Lemon flavoured black tea with honey tea, Vitamin C was increased by 110.5% and 163% respectively as lemon flavours and fresh lemon juice was added.

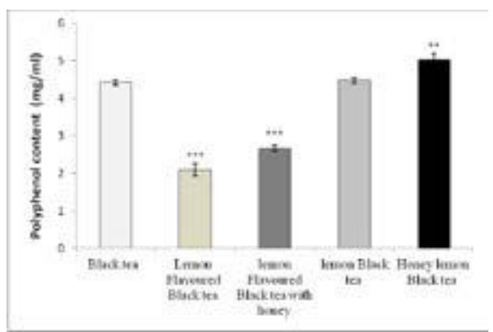


*Figure 3 - Effect of honey on total phenol content in different tea samples. The experiments were performed three times, and the mean values ± S.D. (error bars) have been shown in the lower panels (***, p< 0.001 and**, p<0.01 versus control tea).*

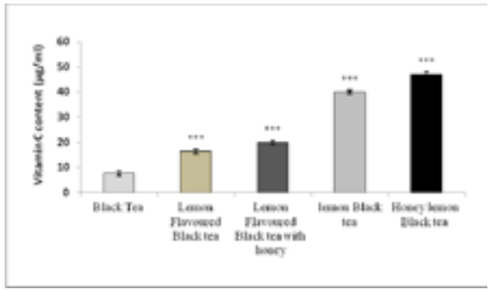


*Figure 4 - Effect of honey on the Vitamin C content in (µg/ml)in different types tea samples . The experiments were performed three times, and the mean values ± S.D. (error bars) have been shown in the lower panels (***, p < 0.001 versus control tea).*

Estimation of tannin in tea

It was found that tannin content of commercially bought lemon flavoured black tea and Lemon flavoured black tea with honey tea was decreased significantly ($p< 0.001$) by 20.9% and 22.4% in comparison to untreated control black tea. This may be due to further processing. In case of freshly prepared lemon black tea and honey lemon black tea no changes were seen in tannin content. This may be because addition of lemon and honey in black tea does not effect the tannin content of tea.

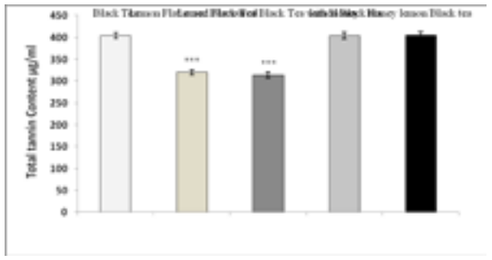


*Figure 5 - Effect of honey on tannin content in different types of tea samples.The experiments were performed three times, and the mean values ± S.D.(error bars) have been shown in the lower panels(*** , p< 0.001 and versus control tea).*

Anti –oxidant activity

It was observed that there was an increase in anti oxidant activity significantly ($p< 0.001$) in freshly prepared lemon black tea by 233% and honey lemon tea by 369% as compared to untreated control black tea. This may be due to their higher phenolic, ascorbic acid and flavonoid content which were increased due to addition of lemon and honey in tea.

Whereas in commercially bought lemon flavoured black tea and lemon flavoured black tea with honey antioxidant activity was increased by 53% and 188 % in comparison to untreated control black tea.

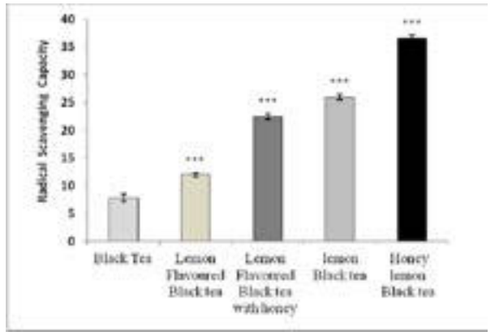


*Figure 6 - Effect of honey on Radical Scavenging Capacity in different tea samples. The experiments were performed three times, and the mean values ± S.D. (error bars) have been shown in the lower panels (***, p< 0.001 versus control*

Anti Microbial property

It was observed that there was no significant difference between the commercially bought lemon flavoured black tea and untreated control black tea. But anti bacterial properties were increased significantly ($p<0.001$ and $p< 0.05$) in both commercially available and freshly prepared lemon flavoured black tea with honey by 140%, lemon black tea by 68% and honey lemon black tea by 202% as compared to untreated control black tea. Honey has low water content, high osmolarity (high sugar content), low pH, antibiotic peptides, catalase, bee defensin-1, and production of hydrogen peroxide. All these are responsible for anti bacterial properties in honey.[2]

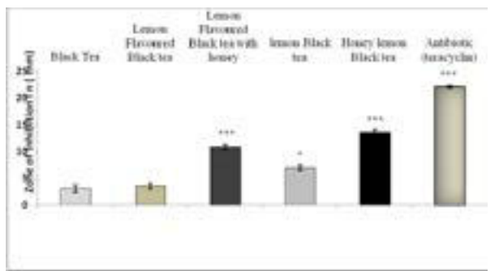


*Figure 7 - Anti Microbial property of different tea samples. The experiments were performed three times, and the mean values ± S.D. (error bars) have been shown in the lower panels (***, p< 0.001 and * p < 0.05 versus control tea).*

Removal of Haze or cream in Honey lemon tea

Honey contains Ca2+, Mg2+, colloidal matters,

protein and polyphenols which contributed to honey lemon tea creaming. The interaction between hydroxyl groups of phenolic compounds and peptide bonds of protein in forming strong insoluble polyphenol-protein complex in aqueous solution which is catalyzed by metal cations resulting in increase of haze and cloudiness.[9]

When the tea was treated with tannase, the haze was removed which was due to degallation of gallated catechines and gallaeted polyphenols which prevent their interaction with protein.

Sensory evaluation

Sensory evaluation was done to observe the acceptability of the developed tea, showed that honey lemon black tea was widely accepted as it improves the quality of tea in relation to colour, aroma, taste and overall acceptability. Error bars indicate standard deviation from triplicate determination.

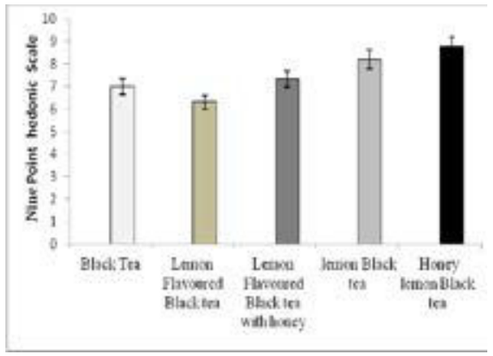


Figure 8 - Sensory Evaluation of different Tea samples based on appearance.

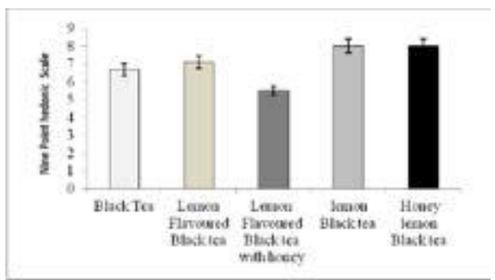


Figure 9 - Sensory Evaluation of different Tea samples based on colour.

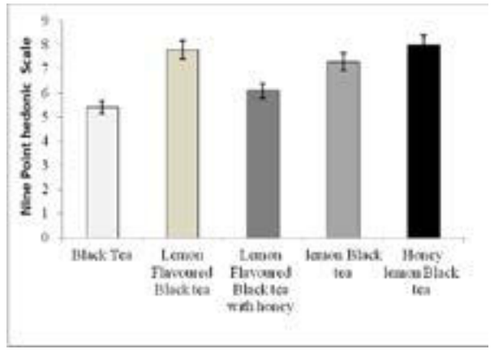


Figure 10 - Sensory Evaluation of different Tea samples based on aroma.

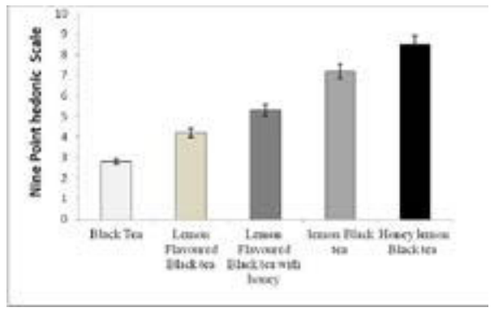


Figure 11 - Sensory Evaluation of different Tea samples based on taste

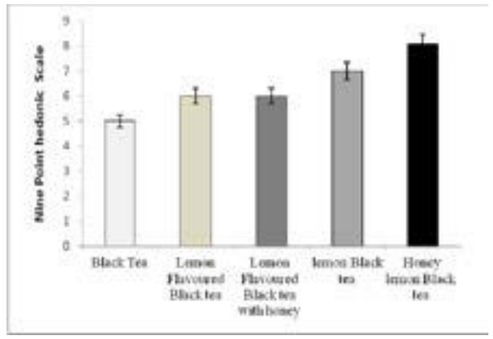


Figure 12 - Sensory Evaluation of different Tea samples based on overall rating

Conclusion

The result of the present study clearly indicates that the addition of honey in freshly prepared lemon black tea is the best antioxidant content as compared with commercially available lemon flavoured black tea and control black tea increasing the total polyphenol, ascorbic acid, protein, sugar, antibacterial including catechins, theaflavins, thearubigins and radical scavenging activity. The preparation and marketing of these teas can also be looked into and encouraged widely consumed commercially.

Further study can be done to demonstrating their antioxidant activity in vivo such as scavenging reactive oxygen and nitrogen species. They may also function indirectly as antioxidants through antioxidant enzymes, such as glutathione S-transferase and superoxide dismutase. Animal studies offer a unique opportunity to assess the contribution of the antioxidant properties of honey lemon tea.

References

1. Abdulwahid Ajibola., Joseph P Chamunorwa., Kennedy H Erlwanger. (2012) Nutraceutical values of natural honey and its contribution to human health and wealth Nutrition & Metabolism. 9-61
2. Ahmed Moussa., Noureddine Djebli., Saad Aissat., Baghdad Khiati., Meral Ünal and Salima Bacha1. (2012) Activity and Total Phenolics of Algerian Honeys and Antibacterial Effect against Gram-Negative Bacteria. J Microb Biochem Technol 4(7), 152-156
3. Ann E., Hagerman. (2002). Protein precipitable phenolics
4. Basavarajappa S., K. Raghunandan S., Hegde S.N. Physico-biochemical analysis of multifloral honey of Apis dorsata Fab (Hymenoptera: Apidae) in southern Karnataka India. Current biotica. 5(2), 144-156
5. Chung S.,Yang and Janelle M.,'Landau. (2000) Effects of Tea Consumption on Nutrition and Health. J. Nutr. 2409–2412
6. Halawani, E., Shohayeb, M. (2011) Survey of the antimicrobial activity of Saudi and some international honeys. Journal of Microbiology and Antimicrobials. 3(4), 94-101
7. Halmenstine Anne Marie. (2011) Vitamin C Determination by Iodine Titration. About.com Chemistry. The New York Times Company
8. Hao Wang. (2011) The study of the antioxidant activity of phenolic components of manuka honey. Research Commons at the University of Waikato
9. Hategekimana J., J.G. Ma., Y. Li, E. Karangwa., W. Tang and F. Zhong. (2011) Effect of Chemical Composition of Honey on Cream Formation in Honey Lemon Tea. Advance Journal of Food Science and Technology.3(1), 16-22
10. Kannan Natarajan. (2009) Tannase: A tool for instantaneous tea. Current Biotica., 1(3), 96-103
11. Khalil I., Moniruzzaman M., Boukraâ L., Benhanifia M., Islam A., Islam N., Sulaiman SA., Gan SH. (2012) Physicochemical and antioxidant properties of Algerian honey. Molecules. 17(9),199-215
12. Khan N., Mukhtar H. (2007) Tea polyphenols for health promotion. Life Science Journa. 81, 519-533
13. Liviu Alexandru Margitas., Daniel Severus Dezmarian., Cristina Bianca Pocol., Marioara Ilea., Otilia Bobis., Iosif Gergen. (2010) The Development of a Biochemical Profile of Acacia Honey by Identifying Biochemical Determinants of its Quality. Bot. Hort. Agrobot. Cluj. 38(2), 84-90
14. Mbata, T. I., Debiao, L. U.and Saikia, A. (2008) Antibacterial activity of the crude extract of Chinese green tea (Camellia sinensis) on Listeria monocytogenes. African Journal of Biotechnology. 7(10), 1571-1573
15. Min-Jer Lu., Chinshuh Chen. (2008) Enzymatic modification by tannase increases the antioxidant activity of green tea. Food Research International. 4, 130-137
16. Paranthaman R., Vidyalakshmi R., Murugesh S., Singaravadiel K.(2008) Optimization of fermentation Conditions for production of Tannase enzyme by Aspergillus oryzae using sugarcane Baggasse and Rice Straw. Global Journal of Biotechnology & Biochemistry. 3(2), 105-110
17. Pereira C., Barros L., Vilas- BoasM., Ferreira C. (2012) Potentiating effects of honey on antioxidant properties of lemon- flavoured black tea. Int J Food Sci Nutr. 64(2), 230-234
18. Raghuwanshi S Misra S., Saxena R.K. (2012) Enzymatic treatment of black tea (ctc and kangra orthodox) using penicillium charlesii tannase to improve the quality of tea. Journal of food processing and preservation. 1745-4549
19. Sensory Evaluation Report for Consumer Acceptance Testing of Meat Products. Department of food science and technology. Oregon State University
20. Srivastava Anita., Kar Rita. (2009) Characterization and application of Tannase produced by Aspergillus Niger ITCC on pomegranate rind. Brazillian Journal of microbiology. 40, 782-789
21. Zobel H.F., Stephen A.M. (1995) starch structure, analysis and application. New York mackerel dekker. 34, 35-41

Fortification and nutritional analysis of bread using different varieties of seed flour

Puja Mukherjee and Jincy Abhraham

ABSTRACT

Fortification helps to combat deficiency diseases affecting majority of the population. The aim of the present study was to develop a fortified product using naturally available resources like nutritious seed in the dried form. Sensory evaluation method was used to standardise the product which was developed by incorporation with pumpkin seed, sesame seed and flaxseed flour. Standard analysis techniques were carried out for macronutrient estimation. Properties like moisture content, gluten content, anti-oxidant properties and shelf life were also evaluated. The developed product is a good source of calcium, zinc, fibre, desirable fat and people of all age, except celiac patients (it contains gluten) can consume this product. It is a product which on a regular consumption can help in prevention of many life style disorders.

Keywords: Bread, flaxseed, fortification, pumpkin seed, sesame seed

Introduction

Bread has been considered "staff of life" since the beginning of recorded times.[16] Bread is traditionally produced from wheat (Triticum aestivum) flour. Non-wheat growing regions import the wheat or bread to fulfil their demands.[21] Bread is essentially a baked mixture of flour and water. Additives such as yeast, sugar, egg, milk, and fats help enhance its flavour, appearance, and digestive quality.[4]

Flaxseed, or Linseed *(Linum usitatissimmum)*, popularly known as Alsi, Jawas, Aksebija in Indian languages, is a blue flowering rabi crop and a member of family Linaceae.[7] It is a readily available oilseed and a major dietary source of a-linolenic acid, dietary fibres, proteins, vitamins, minerals and amino acids. The flaxseed components have been reported to possess various physiological activities relevant to human health sustenance, especially in the prevention of cardiovascular disease, cancer and diabetes. Flaxseed consumption has been demonstrated to exhibit potential health benefits, including decreased tumor growth, reduced serum cholesterol levels and a decrease in the formation of breast, prostate, and colon cancers. (12,19) The beneficial effects of flaxseed on human health may result from its antioxidant capacity. The non-lignan phenolic compounds present in flaxseed may also contribute to its antioxidant potential.[2]

Pumpkins are gourd squashes of the genus Cucurbita and the family Cucurbitaceae. Pumpkins have long been used for traditional medicine in many countries. Pumpkin flesh and seeds are rich not only in proteins, antioxidant vitamins, such as carotenoids and tocopherols, and minerals, but also low in fat and calories. β-carotene reduces the skin damage from the sun and acts as an anti-inflammatory agent, while α-carotene is thought to slow the aging process, reduce the risk of developing cataracts, and prevent tumor growth. Vitamin E (tocopherols) protects the cell from oxidative damage by preventing the oxidation of unsaturated fatty acids in cell membrane.[14] Pumpkin seeds are a rich source of proteins, phytosterols, polyunsaturated fatty acids, antioxidant vitamins such as carotenoids and tocopherol and trace elements like zinc. It also contains linolenic acid, ω-6 fatty acid.[3]

Sesame is an oilseed plant in the genus Sesamum and the family Pedaliceae.[5] Sesame seeds contain a number of antioxidants, such as sesamin, sesamolin, sesaminol, and sesaminol glucosides.[15]

Methodology

Product Development: The raw materials were collected from the local market. The seeds were grounded in a kitchen grinder and sifted to obtain a smooth and fine powder. The seed powder was incorporated into the refined wheat flour in different amounts on the basis of the results of sensory evaluation, to produce nutritionally adequate flour. Bread samples were prepared using the Straight Dough Method.[11] The blend included 100 gm of flour with 20% of sesame seed flour; 40% of flax seed flour and 10% of pumpkin seed flour respectively. The acceptability of the prepared product was evaluated by sensory evaluation using a nine – point hedonic scale. The panel members involved where staff and students from the college.

Bread Analyses:

- The gluten content of bread samples were determined by AOAC standard method.(17) Moisture & Ash content of the bread samples was also determined by AOAC standard method.(17)
- Sugar content was determined by the DNSA (3, 5- Dinitrosalicylic Acid Method) method. O.D value was taken at 540 nm using a colorimeter. The calibration curve was performed using DSNA standard. (17)
- Protein content was determined by the Lowry's method. Absorbance was read at 750 nm using a spectrophotometer. The calibration curve was performed using Bovine Serum Albumin. (17)
- Amino acid content was determined by the Paper Chromatography. (17)
- Fat content was determined by the Soxhlet extractor method.(6)
- Crude fibre was determined after extraction of fat from the samples.(17)
- Calcium & zinc content were determined by the Complexometric Titration with EDTA.(22)
- Oxalic acid content of bread was determined by the titrating with standard potassium permanganate. (8)
- Antioxidant Analysis of Bread involved extraction of phenols, determination of total phenolic content using the Folin-Ciocalteau colorimetric method (10,19) and determination of free radical scavenging activity using DPPH free radical scavenging method

Shelf Life Estimation of Bread (9,18)- The different organoleptic properties like – odour, colour, appearance & texture changes occurred in the sample were monitored over time by sensory evaluation. The product (both standard & fortified) were kept at room temperature (28^0C to 30^0C) in a Ziploc packet & in a silver foil.

Statistical Analysis: All the studies were replicated two times and the mean values were reported. The t test was used to evaluate the difference between fortified samples and controls.

Result and Discussion

To evaluate the importance of fortification, the developed product was subjected to standard analysis techniques, the results of which are indicated below.

Type of Yeast used in Product Development: From the sensory evaluation result it can be concluded that the bread made from dry yeast is more accepted than the one made with fresh yeast, since it scored well on texture, flavour & taste. The average score of the fresh yeast bread is 6.35, whereas the dry yeast bread is 8.58. Dry yeast is more economical also since it can be stored and used latter, which is not possible with fresh yeast, which requires immediate usage due to more moisture content.

Product Development - The acceptability of bread based on sensory attributes described by appearance, aroma, texture, taste and overall rating had shown the average score for bread incorporated with 20 gm sesame seed powder to be higher i.e; 7.29 when compared with the other amounts of sesame powder. Likewise, the average score on incorporation of 40 gm flax seed powder to the already prepared sesame bread is higher i.e; 7.39 when compared with the other variations. And finally, the score of bread incorporated with 10 gm pumpkin seed flour to the already standardised bread with sesame & flax seed powder was higher i.e; 7.46.

Gluten Content -The average gluten content of flour used in making the non - fortified and fortified bread is 32%, since the seed flour added in case of fortified bread does not contain any amount of gluten in it. Gluten strength is an important factor in bread baking performance. It contributes to the ability of dough to rise and maintain its shape as it is baked.[23]

Moisture Content - The results showed that the moisture content of fortified bread was 19.7% which is lower than that of the non-fortified bread i.e; 27%. The moisture content of fortified bread is lower due to the incorporation of flax seed, pumpkin seed & sesame seed powder. Hence it can be assumed that due to less moisture a better shelf life can be observed for the fortified bread when compared with the non-fortified bread.

Statistically the value of t–test is higher than the tabulated t_2 value. The value of the t is 26.55, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average moisture content in fortified bread is lower than non-fortified bread. And null hypothesis (H_0) is rejected.

The commonly accepted level of moisture content in bread is known to be within a range of 35-40%.[11]

Sugar Content- The results showed that the sugar content of fortified bread is higher i.e; 0.068% than that of the non-fortified bread i.e; 0.038%.

The sugar content of fortified bread is increased due

to addition of flax seed, pumpkin seed and sesame seed flour. But when compared with the non-fortified bread the amount of increase of sugar content is less, since all the three seed flour are not a good source of carbohydrate content.

Statistically the value of t-test is higher than the tabulated t_2 value. The value of the t is 19.48, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average carbohydrate content in fortified bread is higher than the non-fortified bread. And null hypothesis (H_0) is rejected.

Protein Content - The results showed that the protein content of fortified bread is higher i.e; 0.482% than that of non-fortified bread i.e; 0.360%, due to the addition of flax seed, pumpkin seed and sesame seed flour to the bread. These three seeds are a good source of protein.

Statistically the value of t – test is higher than the tabulated t_2 value. The value of the t is 6.25, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average protein content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Amino Acid Content - The results of paper chromatography showed that, arginine content of fortified bread is higher, whereas no arginine was found in non-fortified bread. Studies have reported that arginine content of pumpkin seeds are much higher.[14] Arginine helps to increase serum HDL concentration in atherogenic rats.[1]

Fat Content- The fat content of the fortified product was analysed and the results showed that the fat content of fortified bread was higher i.e; 20.8% than that of the non-fortified bread i.e; 17.3%.

The fat content of fortified bread increases because flax seed and pumpkin seeds are good sources of fat. But the fat content involved here is of benefit to an individual.

Studies in 2011 & 2008, reported that, incorporation of flax and pumpkin seeds mixture is a good source of omega-3 & omega-6 fatty acids, has anti atherogenic hypolipidemic & immunomodulator effect.[13]

Statistically the value of t – test is higher than the tabulated t_2 value. The value of the t is 23.33, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average fat content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Crude Fibre Content- The fibre content of bread prepared with different levels of seed flour mixed with refined wheat flour was analysed after the fat was removed from the sample. The results showed that the crude fibre content of fortified bread is higher i.e; 2.3% than that of non-fortified bread. Since flax seed is a good source of fibre, addition of this to the bread helps in increasing the fibre content of bread.

The crude fibre is a measure of the quantity of indigestible cellulose, pentosans, lignins and other components present in food. The crude fibre has little food value but it gives bulk to the food and also helps to regulate certain physiological functions.[20]

Mineral Estimation

Ash Content- The results showed that the ash content of fortified bread is higher i.e; 24.06% than that of non-fortified bread. The addition of seeds to the non-fortified bread increases the ash content.

Statistically the value of t-test is higher than the tabulated t_2 value. The value of the t is 6.9, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average ash content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Calcium Content- The results showed that the calcium content of fortified bread is higher i.e; 90% than that of non-fortified bread i.e; 17%. The calcium content of fortified bread increases mainly because of the sesame seeds incorporation to the bread. Sesame seeds contain a high amount of calcium.

Calcium plays a crucial role in the growth, development and repair of our bones and teeth. It helps maintain the electrochemical balance that allows muscles to contract and neurons to transmit impulses. Eating plenty of calcium-rich foods may decrease risk of high blood pressure, osteoporosis, high blood cholesterol and obesity.

Statistically the value of t-test is higher than the tabulated t_2 value. The value of the t is 74, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average calcium content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Zinc Content- The results showed that the zinc content of fortified bread is higher i.e; 6% than that of non-fortified bread i.e; 0.11%. The zinc content of fortified bread increases mainly because of the sesame seeds & pumpkin seeds which contain good amounts of zinc.

Statistically the value of t-test is higher than the tabulated t_2 value. The value of the t is 15.26, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average zinc content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Zinc is vital to maintain the health of cardiovascular cells and the endothelium. Low zinc can cause a deficiency in the endothelial barrier, which leads to high cholesterol buildup and inflammation. Cholesterol and inflammation increases risk of heart disease. Zinc is a critical mineral for robust testosterone levels, and the cells of the male prostate require a very high concentration of zinc to work optimally. Low zinc in men impairs testosterone production, puts them at risk for developing prostate cancer, and causes infertility.

Antioxidant Estimation

Total Phenolic Content- Total phenolic content is an important parameter as it determines the polyphenolic content of the sample which is interrelated to the antioxidant activity of the sample. The total phenolic content of the bread prepared with different levels of seed flour was analysed using gallic acid standard. The results showed that the total phenolic content of fortified bread was higher i.e; 2% than that of non-fortified bread i.e; 0.9%.

The fortified bread contain high amount of phenolic compound, because flax seeds and pumpkin seeds contain lots of phenolic compound.

Statistically the value of t-test is higher than the tabulated t_2 value. The value of the t is 8.01, which is greater than the tabulated value of t2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average phenolic content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Free Radical Scavenging Activity- The antioxidant activities of bread prepared with different levels of seed flour mix were analysed using the DPPH radical scavenging activity method. The results showed that the antioxidant activity of fortified bread is higher i.e; 32.69 % DPPH inhibition than that of non-fortified bread i.e; 19.81 % DPPH inhibition. R. Meral had previously reported that the antioxidant activities of the bread increased significantly as the level of flaxseed substitution increased.[7] Another study had reported that flax seeds lignans have antioxidant activity.[7] Statistically the value of t-test is higher than the tabulated t_2 value. The value of the t is 21.11, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average antioxidant content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Oxalic Acid Content- The results showed that the oxalic acid content of fortified bread is higher than that of non-fortified bread. Bread containing seeds showed 0.31% oxalic acid, whereas bread without seed flour showed 0.11% oxalic acid.

If the fortified bread has excess oxalic acid then it will react with calcium and zinc, and produce calcium and zinc oxalate leading to the unavailability of these minerals. According to oxalic acid content of food by Helen O'Connor 0 to 2 mg per serving considered as a low oxalate. So the fortified bread considered as a low oxalate product.

Statistically the value of t-test is higher than the tabulated t2 value. The value of the t is 27.53, which is greater than the tabulated value of t_2 i.e. 4.303. So, H_1 hypothesis is accepted, i.e. average oxalic acid content in fortified bread is higher than non-fortified bread. And null hypothesis (H_0) is rejected.

Shelf Life Estimation of Bread- The breads were stored in plastic & aluminium foil containers at room temperature. There was no significant contribution of the packaging materials towards the stability of the product. The non-fortified bread was spoilt at room temperature much faster than the fortified bread and showed microbial spoilage within 48 hours of storage, whereas the fortified bread showed microbial spoilage after 72 hours of storage at room temperature. The spoilage was because of the mould formation since room temperature provided the optimal conditions for the growth of microbes. There was a particular off odour from the food product which denoted spoilage.

The average sensory scores for smell, appearance and texture have been reported to decrease as the time increases, the lowest being at 72 hours. At 48 hours, the average scores of the non-fortified bread is decreased but the average score of the fortified

bread is higher. The average scores of the non-fortified bread & the fortified bread is the same at 24 hours. Hence it can be concluded that the product developed has a better shelf life when compared with the non-fortified product.

Conclusion

Fortification can be used as an effective device to fight against micronutrient deficiencies and to achieve this; a staple food was incorporated with nutrient dense naturally available seeds like pumpkin, flaxseed and sesame in the powdered form.

Thus it was concluded from the present study that inclusion of the seed flour to the bread significantly improved the sugar, protein, amino acid, fat, fibre, calcium, zinc, phenolic compounds and antioxidant contents. Results indicated that flaxseed, pumpkin seed and sesame seed flour can be incorporated to the wheat flour of bread without negatively affecting the physical and sensory attributes of the bread.

The developed product can be consumed by people of all age groups & is mainly useful to prevent the lifestyle diseases.

Reference

1. Abuelgassim O Abuelgassim, and Showayman I A AL-showayman; "The effect of pumpkin (Cucurbita pepo L) seeds and L-arginine supplementation on serum lipid concentrations in atherogenic rats", Afr J Tradit Complement Altern Med, 9(31), (2012), 131 - 137.
2. Agnieszka Kosińska, Kamila Penkacik, Wiesław Wiczkowski, and Ryszard Amarowicz; "Presence of caffeic acid in flaxseed lignan macromolecule", Springer, 66(3), (2011), 270–274.
3. Barakat LA, Mahmoud RH; "The antiatherogenic, renal protective and immunomodulatory effects of purslane, pumpkin and flax seeds on hypercholesterolemic rats", North American Journal Of Medical Science, 3(9), (2011), 411–417.
4. Brigid M.K; "Nutritional Aspects Of Cereals"; British Nutrition Foundation Nutrition Bulletin, 29, (2004), 111-142.
5. Ekasit Onsaard; "Sesame Proteins", International Food Research Journal, 19(4), (2012), 1287 - 1295.
6. Food Science Astralia, "Meat Technology Information Sheet – Crude Fat Determination – Soxhlet Method", (2006).
7. Ganorkar, P. M. and Jain, R. K.; "Flaxseed – a nutritional punch", International Food Research,Gujrat, 20(2), (2013), 519 – 525.
8. H. Ilarslan, R. G. Palmer, J. Imsande, And H. T. Horner, "Quantitative Determination Of Calcium Oxalate And Oxalate In Developing Seeds Of Soyabean (Leguminosae)", American Journal of Botany, 84(9), (2013), 1042–1046.
9. Information Booklet for the Food Industry, "A Guide to Calculating the Shelf Life of Foods", (2005)
10. Lenzi Almeida KC, Teles Boaventura G, Guzmán Silva MA; "Influence of omega-3 fatty acids from the flaxseed (Linum usitatissimum) on the brain development of newborn rats", Nutricion Hospitaloria, (2011), 26(5), 991 - 996 .
11. Lipi Das, Utpal Raychaudhuri, Runu Chakraborty; "Herbal Fortification Of Common Bread By Fennel Seeds", Department of Food Technology and Biochemical Engineering, Jadavpur University, 3161, (2013),.
12. Lubna Masoodi, Dr Vidhu Aeri –Khalid Bashir; "Fortification of Biscuit with Flaxseed: Biscuit Production and Quality Evaluation", IOSR Journal Of Environmental Science, Toxicology And Food Technology (IOSR-JESTFT), 1(5), (2012), 06 – 09.
13. Makni M, Fetoui H, Gargouri NK, Garoui el M, Jaber H, Makni J, Boudawara T, Zeghal N, "Hypolipidemic and hepatoprotective effects of flax and pumpkin seed mixture rich in omega-3 and omega-6 fatty acids in hypercholesterolemic rats", Food & Chemical Toxicology, 46(12), (2008), 3714-20.
14. Mi Young Kim, Eun Jin Kim, "Young Nam Kim, Changsun Choi And Bog Hieu Lee; "Comparison Of The Chemical Compositions & Nutritive Values Of Various of Pumpkin Species & Parts", Nutrition Research & Practice, 6(1), (2012), 21 - 27
15. Mochizuki M, Tsuchie Y, Nakamura Y, Osawa T; "Identification and characterization of sesaminol metabolites in the liver", Agriculture & Food Chemistry, 57(21), (2009), 10429 - 34.
16. Mohammad Talaei; Noushin Mohammadifard; Mohammad-Reza Khaje; Nizal Sarrafzadegan; Firoozeh Sajjadi; Hasan Alikhasi; Maryam Maghroun; Farhad Iraji; and Shahram Ehteshami; "Healthy Bread Initiative : Methods, Findings, and Theories – Isfahan Healthy Heart Programs", Health Population & Nutrition, 31(1), (2013), 49 – 57.
17. N. Raghuramulu, K.Madhavan Nair, S.Kalyanasundaram; "A manual of laboratory techniques", NIN, Hyderabad, (2003).
18. Omer Mukhtar TARAR1, Salim-ur-REHMAN, Ghulam MUEEN-UD-DIN, Mian Anjum MURTAZA; "Studies on the shelf life of bread using acidulants and their salts", Turk J Biol,34, (2010), 13 - 138.
19. R. Meral and I. Sait Dogan; "Quality and antioxidant activity of bread fortified with flaxseed", Italian Journal of Food Science, 25(1), (2013), p51.
20. Shahzad Hussain, Faqir Muhammad Anjum, Mohammad Saleh Alamri; "Fortification Of Pan Bread With Healthy Flax Seed" ; Australian Journal of Basic and Applied Sciences, 5(11), (2011), 978-983.
21. Sweta Rai; Amarjeet Kaur; Baljit Singh; and K. S. Minhas; "Quality Characteristics Of Bread Produced From Wheat, Rice, And Maize Flours ", Food Science & Technology, 49(6), (2012), 786 – 789.
22. www.chem.uky.edu/courses/che226/labs/030-Zn_EDTA.pdf
23. www.healthyonraw.com/should-we-eat-greens-high-in-oxalic-acid/

Comparative Study on Nutritional Composition of Homemade Indian Curd using mixed Culture & Single Strain Culture

Ipsita Dutta and Adrija Sarkar

ABSTRACT

This study focuses on a comparison between two curd samples, one made from mixed culture and the other from single strain culture, where we discuss about the nutritional status of each and how inoculating starter culture effects the nutritional composition.

The study was subdivided into three sections of tests, which includes physical test, nutritional composition analysis and microbial analysis for shelf life study. Physical tests include pH level, ash content, solid & moisture content. Organoleptic evaluation was also carried out. Carbohydrate, protein, fat and calcium estimation was performed as a part of the nutritional composition analysis and standard plate count, total fungal count; total coliform count has been carried out for shelf life study.

The sensory evaluation Panel members liked pure culture more as it was less acidic than mixed culture. It was found from the study that mixed culture curd contain more ash, solid and calcium than pure culture curd whereas pure culture curd or single strain culture curd is better in containing moisture, carbohydrate , protein and fat. In case of microbial analysis mixed culture contain more microorganisms than pure culture. Again the mixed culture curd contains coliforms, an indication of fecal contamination, which is a major problem as coliform creates health hazards. But pure culture has less coliform count, and hence this is a significant point of difference between them.

Keywords: curd, Lactobacillus, probiotics

Introduction

Milk and dairy products continue to play an important role in the nutrition of people in many parts of the world. Milk has been shown to be ideal for the growth of microorganisms due to its water, protein, carbohydrate, mineral and vitamin contents (Seo et al., 2007; Shan-na Liu et al., 2011). Storage of milk for long periods under refrigeration temperatures has resulted in new quality problems for the dairy industry arising out of the growth and metabolic activities of psychrophiles (Jonghe De Valerie et al., 2011).

In addition, curd due to the probiotic activities of the beneficial bacteria contains good proportion of minerals like phosphorous and calcium as also proteins and vitamins further highlighting their efficacy as potential good probiotic products.

Therefore, curd contains all its needs to become a good probiotic product but loses out in the technical front to be claimed a true probiotic food. So, is there a possibility to overcome these drawbacks and make curd a probiotic product according to the set of WHO guidelines? Indeed, it can be a reality. The fermentation of curd can be regulated with the application of specific known probiotic strains in the batter culture. There is also a good possibility to enumerate the viable counts of the bacterial cells. Once these aspects are controlled, curd can be called a probiotic product (Kavitha C and Predeepa R J)

Methodology

Preparation of sample: Fermented dairy products are made from milk of almost all domesticated milk animals since ages. Several new types of fermented milk products are becoming popular all over the world. The traditional fermentations are taking place as a result of the activities of natural flora present in the food or added from the surroundings. Over the period, scientists have tried to isolate and study the characters of such desirable organisms. Among the bacteria, the most important dominant group bringing fermentation is lactic acid bacteria. The lactic acid bacteria are naturally accepted as GRAS (Generally regarded as safe) for human consumption.

Here curdling was prepared by using two types of culture, mixed culture, obtained from a local market and single strain culture obtained as capsule from a local medicine shop.

Quality Assessment by Organoleptic Test (Hedonic Scale):

The most common method is a questionnaire of generated foods or food categories in which a hedonic scale is used to rate the degree of liking. A 5 to 9 point balanced scale is used, usually a 9 point scale. The essential features of the hedonic scale are its assumption of a continuum of preferences and the direct way it defines the categories of response in terms of like and dislikes.

Physical Test: Physical tests include the comparison of ash content (a part of mineral estimation), total solid & moisture content and pH test that is relevant to acidity analysis.

Nutritional Analysis: Under nutritional analysis the comparison has been drawn on the basis of carbohydrate (DNSA method), protein (Folin Lowry Method), fat (Gerber method) and calcium (complexometric Tritration method) estimation.

Microbial Analysis: Microbiological quality assessment of the collected curd samples were done by determining the Standard Plate Count, Total Coliform Count and total Fungal count. Different types of media were used for microbiological quality assessment, i.e., Nutrient agar (NA), MacConkey Agar (MAC) and Potato Dextrose Agar (PDA). All microbial analysis was done from Analytical Methods For Microbiologicalw. Köster,

Result and Discussion

Organoleptic Evaluation

The result was withdrawn on the basis of score given by 20 panel members where 1= dislike extremely & 9= like extremely.

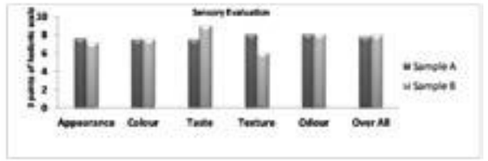


Figure 1 - Sensory Evaluation of Sample A and B

Appearance & Texture- The nice consistency of mixed culture curd was more attractive to 20 panel members as the there was less moisture than pure culture. As a result the watery part was also less and a fine texture appeared. On the other hand pure culture curd also appeared well but as there was more moisture than mixed culture so it became less attractive to panel members as the texture was hampered.

Taste- t is the major part of the total study. Taste differs much between them. People like pure culture curd most as it was less sour than mixed culture. This is due to the less pH of mixed culture curd than pure culture curd.

Color & Odor- A fine aroma was coming out from both kinds of curds with a suitable color also. This may be due to slow heating of milk for long time.

Physical Tests- Physical tests include ash content, total solid & moisture content and pH.

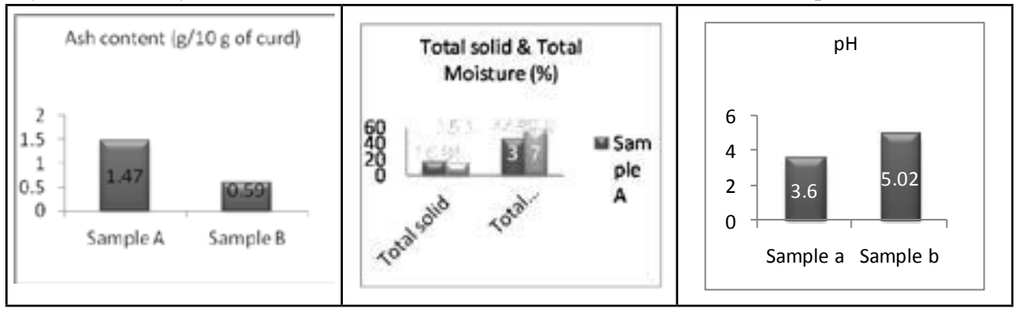


Figure 2: Physical test result

Ash content denotes the volume of solid part present in cud. The more solid is there the more ash will be there. Evaluation of ash amount is important because we can evaluate the minerals from the ash volume. As the moisture part is high in sample B (pure culture) so, it gave less amount of mineral where as more presence of ash in sample a(mixed culture) represents the more present of solid part in sample A than sample B.

The volume of solid part is very much important because only from the solid part we get the macronutrients like carbohydrate, protein & fat. So it is the actual part of curd that is responsible for nutritional benefit.

On the other hand moisture increases the volume of entire curd as well as bulk of it. It is also responsible for the fungal growth. The more moisture is there the more fungal growth will be there. Thus shelf life will be reduced. Again moisture helps in the growth of microorganisms. More moisture will be responsible for rapid growth of more lactobacillus resulting in increased sourness of curd which affects the taste consequently.

This part of the study compares the solid and moisture part between the two kinds of curd where the result says sample A contain more solid than sample B. Again sample B is high in moisture

content than sample A. So we can interpret that sample B has high chance of being contaminated therefore reduced shelf life, where as sample A is comparatively good nutritionally.

pH level denotes the acidity of curd. As we know that reducing the level of pH (less than 7), increases the acidity. Here, sample A is more acidic or sour than sample B which again affects the sensory evaluation. More sourness was not acceptable to panel members and thus sample B scored better than sample A in "taste parameter" of sensory evaluation.

Nutritional Analysis-

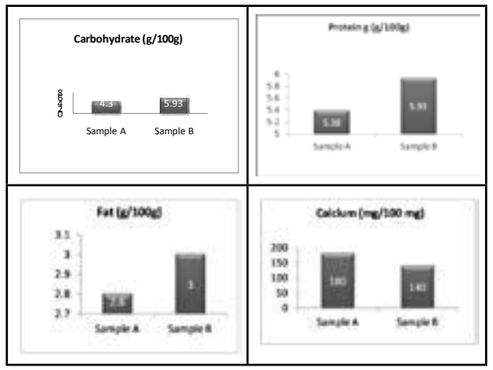


Figure 3 - Result of Nutritional Analysis

Carbohydrates, fat and protein are naturally occurring bulk nutrient present in almost all foods in differing quantities. Carbohydrates are sugar or polymers of sugar as starch, that can be hydrolyzed to simple sugar by the action of digestive enzymes or by heating with dilute acids. Generally, but not always, the hydrogen and oxygen in them are in the proportions to form water; hence the term carbohydrate. (B. Srilakshmi, Nutrition Science 3rd Ed, 2008).

Carbohydrate is the major macronutrient that takes part to provide maximum energy. 1 g of carbohydrate provide 4 kcal of energy. Not only for energy purpose carbohydrate is important for fetal brain development also. Here we can see Sample A contain less carbohydrate than sample B. So sample B is better in provide energy.

1 g of protein provides 4 kcal of energy. Curd is composed of two kinds of protein – Casein & whey protein. Here also sample A contain less protein than sample B. So here also we can see pure culture is more acceptable than mixed culture curd as it is nutritionally more adequate.

Fats are carbohydrates compounds of the three elements carbon, hydrogen & oxygen. The lower amount of oxygen in relation to the other two elements results in fat being a more concentrated source of energy than carbohydrate (B. Srilakshmi, Nutrition Science 3rd Ed, 2008).

This result indicates the increased availability of fat in sample B than sample A which signs for it developed quality. 9 Kcal is obtained from 1 g of fat. Sample A provides (3.1x9) 27.9 kcal whereas sample B provide (4.06x9) 36.54 kcal. As fat increases palatably of food so it may be a reason to be accepted as more tasty to the panel members. But it is good to take sample A for obese people as it contain less amount of fat.

The minerals present at levels more than 0.05 % in the human body are defined as macro minerals. Calcium, phosphorus, magnesium, sodium and potassium belong to this category.

Here only the value of calcium has been evaluated as milk and milk products is a good source of calcium. Here the result obtained that sample B contains less calcium than sample A which says that sample A is good for bone and muscle health. We can say sample B is also good as there is less difference between them but still sample A contains more calcium.

Microbial Analysis (Shelf Life)- For conducting the shelf life of the study the samples were kept or stored at 4^0C as it is well known that curd can't be stored as a table top food material i.e. room temperature. The Standard Plate Count (SPC) is used as an indicator of the level of bacteria in dairy products. The SPC does not measure the entire bacterial population, but rather the number of bacteria that grow in the presence of oxygen (aerobically) and at medium range of temperature (mesophilic) temperatures. The SPC is the reference method specified in the Grade A Pasteurized Milk.

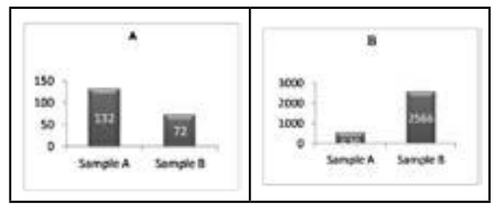


Figure 4 - Analysis of Shelf Life. A) Standard Plate Count B) Total Fungal Count. The results are typically expressed as colony forming units (C.F.U)/gm.

Ordinance for the examination of raw and pasteurized milk and milk products.

This result indicates the presence of more microorganisms in sample A than sample B which means sample A has high chance to be contaminated and create health hazard. This may be due to the fact that mix-culture was obtained from a local shop (non-reliable source). The pure culture sample had lesser count and hence safe as it was obtained from a reliable marketed product (Vizylac; B.No. DVC-13115, MFG- Sep 2013; Exp- Feb 2015)

Again Sample B contains more fungal contamination as both of the samples were kept for prolonged time or more than its shelf life (7 days).

Total Coliform Count

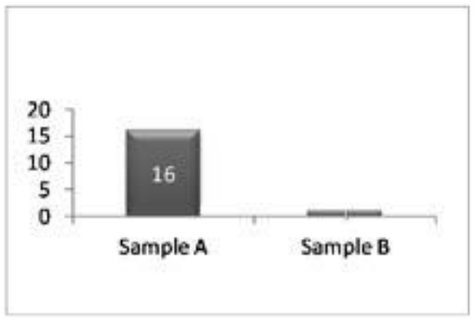


Figure 5: Number of coliform present in two curd samples. The results are typically expressed as colony forming units (C.F.U)/gm

Coliform bacteria are a commonly used bacterial indicator of sanitary quality of foods and water. They are defined as rod-shaped Gram-negative non-spore forming bacteria which can ferment lactose with the production of acid and gas when incubated at 35–37°C. Coliforms can be found in the aquatic environment, in soil and on vegetation; they are universally present in large numbers in the feces of warm-blooded animals. This result indicates the presence of more coliform in sample A than sample B which means sample A has high chance to be contaminated and create health hazard which further can be a cause of fecal contamination. This may be due to mixed culture was inoculated from an outsider source that is unreliable whereas pure culture was reliable.

Conclusion

The present study involved the comparison of nutritional analysis of two samples of curd made from mixed culture starter and pure culture starter. The usual way of preparing curd in our homes on a daily basis is the use of some leftover curd as started culture for inoculation. This study would point out if changing the way of inoculation would make an effect in its nutritional value and taste.

For assessing the nutritional quality, biochemical parameters like the calcium content, total solid content, total moisture content, and total ash content was measured. The amount of carbohydrate, protein and fat was measured as well. These estimations would reflect the nutritional implication of the food material. As a part of the shelf life study, the total standard plate count, total fungal count and total coliform count was estimated.

Finally from the study it can be concluded that Sample A mixed culture curd is good from the aspects of appearance, texture, total solid content but comparatively poor in nutritional value whereas Sample B pure culture curd is good from all nutritional aspects except calcium and it was also easily preferred by panel members.

From nutritional view though the solid part is less still pure culture gives 5.93 g of carbohydrate per 100 g where mixed culture gives 4.93g of carbohydrate per 100 g. In case of protein 5.98g is obtained from pure culture curd per 100 g where 5.38 g is obtained from mixed cultured curd per 100 g. 100g of pure culture gives 3 g of fat but 100g of mixed culture gives 2.8 g of fat. But calcium is more in mixed cultured curd (180 mg/100g) than pure culture curd (140mg/100g).

The amount of nutrients varies not significantly between them. Even in spite of having similar shelf life we say that pure culture is better than mixed culture as mixed culture curd carry coliforms by fecal contamination which again can create health hazard like diarrhea, vomiting, abdominal pain etc. We regard curd as a probiotic but it is not good that a probiotic itself become a health hazard.

References

1. Jonghe De V., Coorevits A., Van Hoorde K., Messens W., Van Landschoot A., De Vos P. Heyndrickx M. Influence of storage conditions on the growth of Pseudomonas species in refrigerated raw milk, Applied and Environmental Microbiology, 2011, vol 77, 460-470.
2. Kavitha C and Predeepa R J. Antibiosis of bacteriocins with domestic lactobacilli isolated from prepared curd, Journal of the Food & Agriculture 2010, vol 22 , 398-405.
3. Seo., Dong Mi., So-Young K., Hyun-Ju E., Nam Soo Han. Symbiotic Synthesis of Oligosaccharides during milk fermentation by addition of Leuconostoc starter and Sugars, Journal of Microbiology and Biotechnology, 2007, vol 17, 1758-1764.
4. Shan-na Liu., Ye Han., Zhi-jiang Zhou. Lactic acid bacteria in traditional fermented Chinese foods. Food Research International, 2011, vol 44, 643– 651.

Assessment of Knowledge and Practice of Mothers Towards Nutrition and Physical Health Among Children Aged between 6 Months to 6 Years in Bankura District, West Bengal, India

Sutapa Pal and Tanima Bhattacharya

ABSTRACT

Childhood is the period when the child is expected to learn the rudiments of knowledge that are essential for successful adjustments to adult life. These early formative years, during which foundations are laid for sound physical, cognitive and social development, are the most crucial period in one's life. Poor dietary habits during childhood may affect day-to-day well being and performance, growth and development, dental health and increase likelihood of iron-deficiency anemia. The key care practices that could impact on child nutrition include care of pregnant and lactating mothers, breast feeding and feeding young children, care of children during illness, psychosocial care of children, food preparation and storage, and hygiene. A study was therefore carried out to evaluate the knowledge and practice of mothers towards nutrition and physical health among children aged between 6 months to 6 years in Bankura district, West Bengal, India. The results revealed that almost 71% of the children had normal height, weight, MUAC for age where as about 29% children were suffering from malnutrition. Poor health status was also observed in adolescent mothers with low literacy rate, poor family planning and high number of family members. Clinical deficiency signs and symptoms like thin hair which is easily plucked; brittle nail and rough skin were observed in almost every child. The statistical analysis revealed that the mean protein, fat, carbohydrate and energy intake were deficient in children as compared to their RDA requirement thus resulting in undernourishment.

Keywords: Maternal & child health

Introduction

Proper feeding practices during infancy are essential for attaining and maintaining proper nutrition, health, and development of infants and children [1–4]. Results of studies on infant and child feeding have indicated that inappropriate feeding practices can have profound consequences on the growth, development, and survival of infants and children, particularly in developing countries.[5]

Childhood is the period of rapid growth and development. Children are the invaluable assets of the nation, and their physical and educational development determines the extent to which they contribute to the national growth and prosperity. Poor dietary habits during childhood may affect day-to-day well-being and performance, growth and development, dental health and increase likelihood of chronic diet-related diseases risk such as ischemic heart disease and cancer.[6]

The World Health Organization (WHO) recommends exclusive breast-feeding for the first 6 months of life and continuation of breast-feeding for 2 years.[7] The WHO and the United Nations International Children's Emergency Fund (UNICEF) has articulated a global strategy for infant and young child feeding.[6]

Breast-feeding and complementary feeding practices are fundamental to children's survival and development.[8] In many developing countries, nutritional problems in infants and young children are closely linked to these practices. Among other things, feeding practices have an impact on physical growth, which is regarded as one of the best indicators of children's well-being.[9]

If children do not get adequate diet or do not follow healthy eating norms, they may become malnourished. Out of 167 million children in the World who are under eight years of age, almost one-third of the children in developing countries are malnourished.[10] In India, around 46 per cent of all children below the age of three are too small for their age, 47 per cent are underweight and at least 16 per cent are wasted. Many of these children are severely malnourished.[11] If these children survive childhood, many of these children will suffer from poorer cognitive development and lower productivity. As adults, their ability to assure good nutrition for their children could be compromised, perpetuating a vicious cycle. It thus becomes mandatory to understand the causes of malnutrition and delineate which are the most important before we can identify and act upon those areas of intervention that will be most successful in reducing malnutrition.[10]

Thus, the aim of the study was to assess the nutrient intake of the children belonging to the age group of 1-6 years; and to assess the knowledge and practice of mothers towards nutrition and physical health of the children.

Methodology

Hypothesis: It is an exploratory research statistically testing of formal hypothesis is not referred, we are just to examine the "Assessment of knowledge and practice of mothers towards nutrition and physical health among children aged between 6 months to 6 years in Bankura District, West Bengal, India."

Selection of place: The pregnant women were selected from three sub-division in Bankura namely Bankura Sadar ICDS centre, Bishnupur ICDS centre and Khatra ICDS centre comprising of 22 blocks in total.

Selection of sample: 220 children were selected from 22different blocks by Simple Random Sampling. The age group of the children ranged from 6-12 months and 1-6 years. In order to collect samples (children) from 22 blocks in Bankura town, 10 subjects are collected from each blocks. Out of 220 children, 128 children (1 to 6 years), and 92 infants (6 months to 12 months). In 128 children, 72 children are under 1-3 years and 56 are under 4-6 years.

Standardization of the recipes: To carry out the dietary assessment, the most common recipes (in different proportions) like rice, dal, vegetables, chapattis, puris and parathas of different sizes and volumes were standardized in terms of measuring cups, glasses and spoons. This was done to make an accurate appraisal of the quantity of food consumed by the children.

Construction of Questionnaire: The questionnaire was divided into 6 parts.

Part I: Personal Details:

This part dealt with detailed general information about the mothers as well as children such as their name, age, and date of birth, sex, address, phone number, income, community and the preference for vegetarian and non-vegetarian food.

Part II: Anthropometric measurements:

This part of the questionnaire was constructed to find out the anthropometric measurements as they are reliable indicators of growth and development. It is an index from borderline to severe malnutrition. The anthropometric measurements commonly used as indices of growth and development for children include length and weight.

Height was measured with the help of a stadiometer. Weight of the child was taken by a portable human weighing machine with an accuracy of 0.5 kg. Mid upper arm circumference was also calculated.

The National Centre for Health Statistics (NCHS) in the United States has collected data on the anthropometric measurements of the child population. This reference data has been recommended to be used throughout the world. Well- nourished children in developing countries like India grow in the same way as their counterparts in the developed countries like USA, therefore the data here, is used for Indian children too.

Table 1: Classification to determine the degree and type of malnutrition among children [12]

Name of the classification	Indicator	Reference population	Classification of Malnutrition
Waterloo	Height for age	NCHS	<85% Severe malnutrition
			85-90% Moderate malnutrition
			90-95% Marginal malnutrition
			> 95% Normal

Height for Age: Height is a very reliable measure that reflects the total increase in the size of the individual up to the moment it is determined. Recording the height helps us to know whether the child is growing normally and is in good health or not. Height for age is a measure for long duration malnutrition and low height for age indicates "stunting" or chronic malnutrition in a child. The girls belonging to both the income schools were classified according to the degree and type of malnutrition. To determine the height for age, the following formula was used:

$$= \frac{\text{Height of the child x 100}}{\text{Reference height acc to (NCHS)}}$$

Table No. 2: Classification to determine the degree and type of malnutrition among children[12]

Name of the classification	Indicator	Reference population	Classification of Malnutrition
Indian Academy of Pediatrics (IAP)	Weight for age	NCHS	<50% Grade 4 malnutrition
			50-60% Grade 3 malnutrition
			60-70% Grade 2 malnutrition
			70-80% Grade 1 malnutrition
			> 80% Normal

Weight for age: this is a commonly used indicator of body size and it reflects the level of food intake. The relative change in body weight with age is more rapid than that of height and is much more sensitive to changes in the growth pattern of the child. Therefore, weight for age is a very sensitive measure of short duration malnutrition. To determine the weight for age, the following formula was used:

$$= \frac{\text{Weight of the child x 100}}{\text{Reference Weight acc to (NCHS)}}$$

Part III: Clinical Information- This section included questions to analyze the child's physical well being. Different body parts of the children like hair, eyes, tongue, nails and their skin were assessed as well as micronutrients like Vitamin A deficiency, Iron deficiency and calcium deficiency and clinical manifestations were assessed.

Part IV: Eating pattern of the child- Eating patterns of the children were probed to find out the kind and the quality of food consumed. For infants breast feeding, formula feeding related questions were there.

Part V: Hygiene and Sanitation: There are some hygiene related questions are included to know mother's knowledge and practices.

Part VI: Three day dietary recall- This was done using the standardized measurements of common recipes to estimate the average nutrient intake, calculated using ICMR tables.

Analysis of Data: The data obtained was tabulated and organized according to the objectives of the study for appropriate analysis. Percentages were calculated for analysis of the mother marital status, education, knowledge and practices, family income, clinical assessment, eating pattern, miscellaneous and results were represented in the form of tables, and bar graphs and pie graphs. Macronutrients like protein, fat, carbohydrate and energy were calculated using the ICMR table, to check whether they met the daily recommended allowances. The degree and type of malnutrition among children was determined on the basis of height for age, weight for age, Mid Upper Arm Circumference and Body Mass Index calculations. Statistical analysis was done by using t-tests to accept or reject the formed hypothesis.

Results and discussions

The infancy and childhood period has been called the latent time of growth. The rate of growth is high and body changes occur gradually. A child who goes to school is very active. Majority of our school children consume inadequate diet and so they are malnourished. As mother's physical health, marital status, literacy rate, family planning, and also nutrition related knowledge and practice all are much important factors which may produce malnutrition of the children.

It is observed that, most of children's mother age of more than 18 years, but they already have more than one or two children. So the pregnancy period is much early compare to age. About 31.81% belonging below sixteen years and 22.72% are sixteen to eighteen years as they got married in adolescent period.

Again it is observed that, most of the children belong to low income group families. Their standard of living was poor and the monthly income was not enough to support the family. They did not have knowledge of healthy eating habits and good nutrition. Due to these reasons children were neglected very much.

Result determines that there are poor family planning and high number of family members. About 45.45 per cent have more than seven numbers of family members.

Only 11.36% have under nuclear family planning.

The result shows that, 43.18% mothers are primarily educated, only 8.18% and 7.27% are under college and university level and 21.81% of child's mothers are illiterate. So the literacy rate is low.

It can be determined that 54.55% of the children had normal weight for their age. But 34.45% of children were suffering from Grade I malnutrition and 10% were suffering from Grade II malnutrition which is due to very low intake of nutrients. Children consumed meals maximum 3 times a day. But sometimes they skipped meals especially breakfast. Children were also suffering from frequent infection due to improper hygienic maintenance.

On the other hand it is showing that 64.54% of the children have normal height for their age. But 21.81% of children were suffering from marginal malnutrition and 11.36% were suffering from moderate malnutrition and 8% children were suffering from severe malnutrition which is due to very low intake of nutrients and also due to lack of knowledge about good nutrition.

Data represents that 94.55% children were normal. But 5.45% children were at risk of malnutrition which can be again due to low intake of nutrients for a longer period of time and also lack of knowledge, ignorance which affects child's health.

A similar study has been done on 3747 school children in Haryana to determine the nutritional status of those children. Age, weight, length/ height and MUAC of children were recorded. And it has been seen that 48.8% children were stunted, 49.6% were underweight and 9.1% were wasted whereas 47.6% children had neither wasting nor stunting. Prevalence of severe stunting, underweight, and wasting was 18.1%, 11.5% and 0.6%, respectively. The MUAC of most of the children was <13cm.[13]

Result exhibits that every child and infant has a common problem which is nail problem (white spot in the nail, brittle, soft nails). It can be due to calcium deficiency as the children did not consume milk or milk products in adequate amount. Most of the children consume milk or milk products once in a fortnight and rest of the children do not consume milk or milk products.

These children had skin and hair problems. Skin problems could be attributed to deficiency of micronutrients like vitamin A and C where as hair problems could be due to deficiency of iron in the diet. This could be due to low intake of fruits and vegetables in their diet. Apart from this consumption of green leafy vegetables as wells milk and milk products was also low.

A study conducted has showed that iron (involved in many critical physiological processes within the hair follicle) deficiency in school children leads to hair problem (easy to pluck).[14]

Among other problems most of the children were suffering from diarrhea and then cold and cough. Diarrhea can be a major cause of malnutrition. Even if the children consume right amount of food their digestion and absorption will be hampered due to diarrhea. Clinical problems can happen due to the improper maintenance of personal hygiene and sanitation, which can result in diarrhea.

Data represent that, the child feeding practices, how formula feed to be given the baby, how it is kept and how many time it may be kept, this type of knowledge and practices. Due to lack of feeding knowledge and practices baby may be at the risk of irritability, food allergy etc.

The study also revealed poor hygiene practices followed by mothers as well as children. 48% mothers are do not washing their hand before cooking at regularly, and also before feeding their child. It also shows that about 7% child often never wash their hand before eating. And about 52% some time wash their hand but not regularly. It is also represent poor hygiene and sanitation may increase the chances of diarrhea, vomiting, infection and various other types of diseases.

Table 3: The mean of nutrient intake of the three day dietary recall

Nutrients	RDA Requirement		1-3 years	4-6 years
	1-3 years	4-6 years		
Protein (gm)	16.7	20.1	29.55	36.43
Fat (gm)	27.91	36.06	24.5	23.95
Carbohydrate (gm)	185.5	236.2	162.67	163.29
Energy (kcal)	1060	1350	990.35	1018.26

Table 4: The statistical analysis of nutrient intake of the three day dietary recall

Nutrients	RDA specification		Actual consumption		t-test value compute	t-test value tabulated	Null hypothesis accepted or rejected
	1-3 yrs	4-6 yrs	1-3 yrs	4-6 yrs			
Protein (gm)	16.7	20.1	29.55±6.4	36.43±3.77	7.22	1.96	Null hypothesis rejected
Fat (gm)	27.91	3 6.06	24.5±2.71	23.95±2.67	1.158	1.96	Null hypothesis accepted
Carbohydrate (gm)	185.5	2 36.2	162.67±11.8	163.29±14.26	0.271	1.96	Null hypothesis accepted
Energy (Kcal)	1060	1 350	9 90.35±31.3	1018.2±67.246	2.477	1.96	Null hypothesis rejected

Computed t value for testing for protein, fat, carbohydrate and energy supplied by three days, the tabulated value at 5% level of significance. This indicated that in the collected sample the average intake of protein provided by the three days the null hypothesis is rejected and there are significant difference in mean (average) intake of protein between 1-3 years and 4-6 years. The value for fat indicated that in the collected sample the average intake of fat provided by the three days the null hypothesis is accepted and there are no significant difference in mean (average) intake of fat between 1-3 years and 4-6 years.

The result for carbohydrate indicated that in the collected sample the average intake of carbohydrate provided by the three days the null hypothesis is accepted and there is no significant difference in mean (average) intake of carbohydrate.

The energy value indicated that in the collected sample the average intake of energy provided by the three days the null hypothesis is rejected and there are significant difference in mean (average) intake of energy between 1-3 years and 4-6 years.

Conclusion

Malnutrition during childhood retards growth and delays development in children. A diet inadequate in quantity and quality is a relevant factor affecting growth and development. While malnutrition affects the people of all ages, it is agreed that children are the worst sufferers.

There could be many reasons for the malnourishment in children. The result obtained from the study show that, most of mothers age above 18 years, but they already have more than one or two children. So the pregnancy period is much early compare to age. About 31.81% belonging below sixteen years and 22.72% are sixteen to eighteen years. The literacy rate is also low. Due to more number of family members and family income being low, many children in the district are suffering from malnutrition because of low intake of nutrients. Micronutrient deficiencies were also common.

Children were also suffering from frequent infection due to improper hygiene maintenance and practices.

It was also observed that, knowledge of most of the mothers about breast feeding and breast feeding practices was poor. So lack of knowledge, late initiation of breast feeding, lack of exclusive breast feeding, and inappropriate complementary feeding may have led to malnutrition. Mother's knowledge of their child's health was also poor. Overall, awareness can be spread to improve the knowledge of mothers towards nutrition and physical health of their children to combat and treat malnutrition.

Reference

1. Abd El-Aty N.A., Moftah F.M., 'Assessment of Knowledge and Practice of Mothers toward Home Accidents among Children under Six Years in Rural Areas in Assiut Governorate , 8 (2), 11-29,(2005).
2. Agrawal P.K., Agrawal S., Ahmed S., Darmstadt G.L., Willuams E.K., Rosen H.E., Kumar V., Kiran U., Ahuja R.C., and Srivastava V., 'Effect of Knowledge of Community Health Workers on Essential Newborn Health Care :A Study from Rural India,' Health Policy and Planning ,27(1),115–26,(2012).
3. Amosu A .M., Atulomah N. O. S., Thomas M.A., Olanrewaju M.F., and Degun M., 'Child Care Practices and the Nutritional Status of Infants of Working Mothers in Day Care Centres in Oshun State, Nigeria, Scholars Research Library, Annals of Biological Research, 2 (5), 140-48,(2011).
4. Apurba S., Dipta K., and Asit B., 'Infant- and Young Child-feeding Practices in Bankura District, West Bengal, India,' Journal of Health Population and Nutrition, 28(3), 294-99, (2008).
5. Bandyopadhyay D., Ahemed T., ' A Study of Knowledge, Attitude and Practice Among Mothers Towards Acute Respiratory Infection in Urban and Rural Communities of Burdwan District, West Bengal, India,' 1(8), 28-51,(2013).
6. Freedman DS, Dietz WH, Srinivasaan SR, 'The relation of Overweight To Cardiovascular Risk Factors Among Children and Adolescents', The Bogalusa Heart Study, Pediatrics, 103(6), 1175-118,(1999).
7. Development & Planning Department Government of West Bengal. District Human Development Report : Bankura45-6, (2006).
8. Data Bank, Infant, Child and Teen Mortality, 5-37,(2013).
9. Marshall, S.J. (2004). 'Developing countries face double burden of disease', Bulletin of the World Health Organization, 82 (7), 45-49, (2001).
10. Sifferlin, A., 'Understanding How Breastfeeding Affects Obesity.'(2013).
11. Mehrotra M., Arora S., Nagar V., 'Physical Health Status of Primary School Children', S.I.T.U Council of Educational Research, 37 (1), 568- 578, (2010).
12. Mitchell S.A., Miles L.C., Brennan L., Matthews J., 'Reliability of the school Food Checklist for in school audits and photograph analysis of children's packed lunches', Journal of Human Nutrition and Dietetics, 23, 48-53,(2009).
13. Kumar R., Aggarwal A., Iyenger S., 'Nutritional status of children: validity of mid-upper arm circumference for screening under nutrition' Community Medicine, Postgraduate Institute of Medical Education and Research, Chandigarh 160 012.(2007).
14. Dirk M., Elston., 'Iron Deficiency and Hair Loss Problems with Measurement of Iron', Journal of the American Academy of Dermatology, 63(6), 1077- 1082,(2010).

Study on the Food Pattern changes and Nutritional status of college students in Kolkata, coming from North-eastern region

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ABSTRACT

Health is a "state of complete physical, mental, and social well being and not merely the absence of the infirmity. Food contributes to maintain proper health, while lack of food deteriorates the health. "A good Food Habit" not only contributes to the good physical health but also a healthy mind. Today there is good health is taken over by pressure of work, studies, which contributes to wrong dietary pattern which is further deteriorates by unhealthy food choices. In this study attempt was taken to evaluate Dietary habit of the College students of North-eastern students coming to Kolkata, and staying in 'paying guests', 'hostels'. 'mess', etc. The significance of the study lies in the comparison between the nutrient intake of the subjects in their hometown and in

Kolkata. In this study it was found that nutrient intake is deteriorated in Kolkata while consumption of fat is more, students are inclined to the foods high in saturated fats and trans-fats, high-sugar containing foods, ready to eat foods etc, all of which contributes to the high fat consumption. There are changes in meal pattern seen among students like consumption of less meal, skipping breakfast and consumption of more fast foods, the reasons behind these changes were also found, which were the method of food preparation where it was found that food prepared in Kolkata are more oily than the food prepared in their own home, the foods are also spicy which make them neglect their meal. It was found that 55.55% of the students feel tired most of the time, due to increased pressure and decreased nutrient intake. Thus this significant study which focus on the nutritional status of the students, the wrong practice of the dietary habit of the students can be checked if attention given on the food choices and diet pattern. Moreover this study gives a further scope to research on dietary habit changes in other community students, and comparison between the food habits in different communities.

Keywords: college students, Food pattern, nutritional status

Introduction

Nutrition is the sum total of the processes involved in the taking in and the utilization of food substances by which growth, repair and maintenance of the body are accomplished. It involves ingestion, digestion, absorption, and assimilation. Sometimes the term nutrition is used to refer to the nutritional status or bodily condition of an individual .the condition of body resulting from utilization of essential nutrients available to the body is termed the nutritional status. It may be good, fair, or poor, depending on the intake of dietary essentials on the relative need for them, and on the body's ability to utilize them.

The north east India is a true frontier region. It has over 2000 km border with Bhutan, China, Myanmar and Bangladesh. North-east India comprises of seven states known as 'Seven Sisters" they are Arunachal Pradesh, Tripura, Manipur, Mizoram, Nagaland, Assam and Meghalaya. Each state has its distinct cultures and traditions. Each state is a traveler's paradise, with picturesque hills and green meadows.

College life is the transitional period of a student's life. This is the time when most of the changes occur be it social or psychological. Most of the students of for the higher studies generally go outstations away their home.

Students staying outside their home for the first time are independent and no more staying with family makes them lead their life the way they need. During this period they face number of problems from living arrangements to the dietary pattern. Students are generally observed neglecting healthy diet pattern, generally in the habit of skipping breakfast, consuming street foods, eating outside more frequently, consuming carbonated beverages, high fat foods etc.[1]

While the global epidemic of nutrient deficiency, imbalance dietary intake, and unhealthy food intake is well described in adult population, data regarding obesity, consumption of more fat, skipping meals and important nutrients, less consumption of fruits and vegetables are also well documented in several studies. In India several studies are done on dietary pattern and physical activity and related problems on college students. Very few studies are done on prevalence of obesity among the college students in India. There are no such studies on North-eastern students coming to Kolkata or different states for study and staying there. So keeping this in mind this study was undertaken to determine the Food Pattern changes and Nutritional Status of College Students coming from North- Eastern Region staying in Kolkata away their home.

Methodology

Selection of sample:

The required respondents were selected from different North-eastern houses of different areas in Kolkata. The study was conducted on a total of 180 subjects from four houses. While selecting the subjects the following criteria were kept in mind:

A) Age group: All the subjects belonged to the age group of 23 -27 years.

B) Community: only North-eastern community students were selected.

No special emphasis was given in the gender .All the students falling under concerned age group and community were considered for data collection. Answers to the questions regarding personal information of the subjects were collected by personal interview. A questionnaire was prepared previously keeping in mind their socio-economic status, their eating patterns, and physical activity. Each subject filled in the questionnaire in the presence of the present investigator.

• Procedure

To carry out the dietary assessment, the most common recipes (in different proportions)like rice, dal, vegetables, fruits, meats of different volumes and chapatis, puris, parathas, of different sizes were standardized in terms of measuring cups, glasses and spoons in the food laboratory of J.D. Birla Institute (Appendix-II). These standardised cups, glasses, spoons, and the different shapes and sizes of chapatis, puris, and parathas cut out on paper and were shown to the college students from which they could select the one similar to the amount they have consumed. This was done to make the accurate appraisal of the quantity of food consumed by the young college students.

• Statistical Analysis:

Table 1: Statistical Analysis of the Nutrient intake of the students

Nutrients	Nutrients Null-Hypothesis (H_0)	Alternative Hypothesis (H_1)
Energy	There is no significant difference between the mean energy intake of North-eastern students in their hometown and in Kolkata.	There is significant difference between the mean energy intake of North-eastern students in their hometown and in Kolkata.
Carbohydrate	There is no significant difference between the mean carbohydrate intake of North-eastern students in their hometown and in Kolkata.	There is significant difference between the mean carbohydrate intake of North-eastern students in their hometown and in Kolkata.
Protein	There is no significant difference between the mean protein intake of North-eastern students in their hometown and in Kolkata.	There is significant difference between the mean protein intake of North-eastern students in their hometown and in Kolkata.
Fat	There is no significant difference between the mean fat intake of North-eastern students in their hometown and in Kolkata.	There is significant difference between the mean fat intake of Northeastern students in their hometown and in Kolkata.

- Standard deviation: Standard deviation is a widely used measurement of variability or diversity used in statistic and probability theory. It shows how much variation or "dispersion" there is from the average (mean or expected value).
- t-test: A t-test is any statistical hypothesis test in which the test statistic follows a student's distribution ,if the null hypothesis is supported. It is most commonly applied when the test statistic would follow a normal distribution if the value of the scaling term in the test statistic were known. When the scaling term is unknown and is replaced by an estimate based on the data, the test statistic (under certain conditions) follows a student's distribution.
- Standard error: When the population standard deviation is unknown it has to be replaced by its estimated value obtained from the sample values. This is called the standard error.
- Construction of the questionnaire: A questionnaire is a list of questions pertaining to the enquiry. Under this method, a questionnaire is sent to various informants with a request to answer the questions and return the questionnaire. The questionnaire was given to the respondents who were expected to read the questions. Their respective answers were recorded in the space meant for the purpose of questionnaire itself. In this study a multiple choice of questionnaire were formulated for the respondents to answer and were noted down. This questionnaire aimed at attaining relevant information on the

dietary pattern and health implication of college students. Keeping in mind the objective of the study, a questionnaire was prepared. In order to attain relevant information regarding the subject, the questionnaire was divided into 5 parts:

Part 1- Socio-economic background:
This part dealt with detailed general information about the subject and his family, such as name, age, address, sex, educational qualification, and religion and food habit.

Part 2- Anthropometric measurements:
The second part of the questionnaire dealt with the anthropometrical measurements of the subjects such as height, weight, and BMI. To measure the subject's height a non stretchable tape was used, for weight weighing machine was used and for the waist and hip circumference non stretchable tape was used.

Part 3- Physical activity
This part dealt with the amount of physical activity that the college students actually do. Options included Gym, Yoga, walking, swimming and dancing.

Part 4- Clinical information
This part dealt with hair, skin and nail problem that the girls might have.

Part 5 - General and dietary questions
This part dealt with the sleeping pattern, the amount of water consumption, their meal pattern, consumption of junk foods, soft drinks, and its frequency of eating, and whether they like eating in hostel or they eat out most of the time etc.

• Food frequency questionnaire:
Food frequency Questionnaire (FFQ) was designed to assess habitual diet for the respondents by asking about the frequency of the consumption of the food items, or specific food groups over a reference period of 1 month. The frequency of foods consumption was assessed by multiple response grid in which respondents were asked to estimate how often a particular food item or beverage was consumed. Categories such as how often a particular food item or beverage was consumed. Categories such as "daily (specifying number of times)".

Selection of cut-offs for overweight and obese:
WHO has recommended classification of body weight that include degrees of underweight and gradation of excess weight or overweight that are associated with increased risk of obesity.

Table 2: Classification of BMI according to WHO

Category	BMI Range (Kg/msq.)
Underweight	Less than 18
Normal	18.1-22.9
Overweight	23-24.9
Obesity I	25-30
Obesity II	30-35
Extremely obese	More than 35

Selection of technique for dietary survey: For the present study, the diet survey technique based on questionnaire method was used to be answered by subject. The ICMR has recommended several methods of conducting diet survey.

Among the different methods the food frequency method and the interview questionnaire method is the most accurate.

Collection of data: After the respondents were identified, the investigator personally interviews the respondent to collect the data. The questionnaire was written in English so that the subject could fill it with ease.

Analysis of data: The data was tabulated and organized according to requirements of appropriate analysis. The data was further analyzed and the results were represented in tabular form and graphical representation.

Results and Discussion

Anthropometric measurement and the prevalence of overweight and obesity.

Table 3: BMI classification of the respondents (according to WHO)

Category	Status of the subjects
<18	6.66%
18.1-22.9	82.22%
23-24.9	11.11%
25-30	—
30-35	—
>35	—

The BMI of the subjects were analyzed and the data shows the prevalence of normal weight is seen Some people were underweight i.e. 6.67%.There are subjects who are underweight by their own choice, especially girls who were reducing the food

intake intentionally to reduce weight .percentage of overweight is about 11.11%.Therefore it is found in this study that majority of the subjects are coming under normal weight, followed by overweight and underweight. In this study the result differ from the studies done by previous researchers where obesity, overweight was found predominantly, it may be due to the physical feature of the subjects coming from Northeastern states.

Comparison of dietary habit between hometown of the students and Kolkata:
Meal pattern of the respondents- In this study irregular meal consumption by is reported. Most of the students were found to consume only two meals per day, the percentage being 61% leads the category, followed by 33% students consuming three meals in a day. Four meals which are advisable are consumed by only 5.55% of the subjects, which is very less. The youngsters who are staying outside their home for study are in a habit of having less numbers of meals that lead to the unhealthy food choice, overeating later during the day, frequent snack and carbonated drink consumption etc. In the present study it was found that respondents practiced majorly three meals in their hometown, followed by four meals a day. This is may be because when they stay at home they follow a particular dietary habit; moreover they have their near ones to take care of themselves. Austin et al, Hegarty state that lack of favorite meals means non-enjoyment of meals which may compromise nutrient intake. Those who got their favorite meals were those who favored traditional meals, which they could get easily and these meals were cheaper but lacked variety and most of the dishes do not supply adequate nutrients.

It is seen from the above graph that consumption of soft drink is higher in ratio than that of milk and fruit juice. Daily consumption of soft drink is higher i.e. around 50% students consume soft drink on daily basis, followed by the consumption of milk and juice i.e. 11.11% on daily basis.

Students staying outside their home practice unhealthy lifestyle some have also reported to develop daily alcohol consumption and smoking. Though majority of the North-eastern students already possess these unhealthy behaviors when they are not away from home, those who don't possess any sort of bad habits they adapt them after migration. There are many bad dietary practices performed by the students, staying outside home. Some of these are skipping breakfast, eating out frequently, consuming non-nutritious food in between meals more frequently, all of which replace healthy food habit of the students. This means majority of them were in the habit of skipping meals. When asked that which of the meal was most often skipped by them it was seen that 44.44% of them were skipping breakfast followed by lunch and dinner. The common reasons cited by the students for skipping breakfast include lack of time, lack of hunger, taste of food, or dieting to lose weight.

Nutritional awareness among college students
Nutritional awareness among college students is mandatory in a right way. Choosing right kind of food and healthy dietary pattern makes a young college student healthier. Health behaviors formed during adulthood may have a sustaining impact on health across life .Entering college life can be exiting, yet stressful event for many adults as they face trying to adapt to changes in academic workloads ,support networks and their new environment. In the present study it was found that 66.6% students prefer ready to eat foods. Since they don't like the taste of the foods Ready to eat foods are consumed more, reasons cited by the respondents were majorly the taste of the food and saving time.

Table 4: Mean nutrient intake of students in their hometown and Kolkata

Nutrients	Recommended Dietary Allowance of the students	Mean intake in hometown	Mean intake in Kolkata	Difference	Std error	t statistics
Energy (Kcal)	2,230	1894	2013.1	119.1	69.44	-17
Carbohydrate (gm)	350	234.460	206.477	27.983	22.67	12.34
Protein	60	52.89	44.029	8.861	1.6248	51.82
Fat	30	63.14	62.82	0.32	9.744	0.27

Fat- Since the tabulated value of t-statistic at 5% level of significance is >1.96, therefore H_1 is rejected and H_0 is accepted. Hence it is said that, there is no 68 significant difference between the mean fat intake of North-eastern students in hometown and in Kolkata.

Carbohydrate- Since the tabulated value of t-statistic at 5% level of significance is >1.96, therefore H_0 is rejected and H_1 is accepted. Hence it is said that, there is significant difference between the mean carbohydrate intake of North-eastern students in hometown and in Kolkata.

Protein- Since the tabulated value of t-statistic at 5% level of significance is >1.96, therefore H_0 is rejected and H_1 is accepted. Hence it is said that, there is significant difference between the mean protein intake of North-eastern students in hometown and in Kolkata.

Energy- Based on the t-test computed value on the basis of sample observation it was revealed that in case of Energy there is a significant difference seen in the dietary intake of the respondents in their hometown and in Kolkata. The computed t-statistic at 5%value being 17.16 is > 1.96, hence H_0 is rejected and H_1 is accepted. Hence it is said that there is significant difference between the mean Energy intake of North-eastern students in hometown and in Kolkata.

Conclusion

Education being the foremost important aspect of life, in today's world people is seeking better opportunity for higher Education. Young adults are found to move in different cities for higher education, not only within the country but also in different countries all over the world. In the process of migration they get away from their family and near ones. They become independent not only socially, but also they have loads of responsibility to rear themselves for the purpose of living. They are bound to take care of their health along with the study. In this process students become stressful, which affect their health. Majority of the students fail to take proper diet. While doing this study it was found that no. of meals in the respondents are comparatively low than their hometown. Majority of the students practice having only two meals a day while very few were found to say that they consume four or five meals a day. Due to which nutrient intake is also low, make them suffer from some of the health related problems like headache, stress, feeling hungry all the time, lack of concentration etc. Healthy foods are replaced by ready to eat foods, carbonated beverages and fattening foods. Hence, Nutritional education should be given to the students, through awareness programmes and workshops, one should maintain the healthy food pattern, avoid skipping meals, inclusion of healthy foods more to maintain a healthy body.

Reference

1. Horikawa C, Kodama S, Yachi Y , Heianza Y, Harawa R, (2011) ' Skipping Breakfast and Prevalence of overweight and obesity in Asian and Pacific regions-A meta analysis .Prev Med,53 (4-5), 260-267
2. Eaton.DK, Kann. L, Kinchen. S. (2012) 'Youth risk behavior surveillance-United States, 2011. Morbidity and Mortality Weekly Report Surveillance Summary,12, 61-71
3. Mozaffari H., Nabei B., (2007) 'Obesity and related Risk factors' , Indian Journal of Pediatrics, 74(3), 265-267
4. Sjajewaska et al, (2010) 'Systematic review demonstrating that breakfast consumption influence body weight in outcomes in children and adolescents in Europe' Critical reviews in Food Science and Nutrition; 50 (2), 113-119
5. Wizterl S. (2008), 'Socio economic and Nutritional Status and Health', The Indian Journal Of Nutrition and dietetics,45 (1), 1-10.
6. Dietary habits and stomach cancer in Mizoram,India. Phukan RK,Narain K, Mahanta J (2006)
7. Sinha DN, Gupta PC, Pednekar M. (2004) 'Tobacco water: a special form of tobacco use in the Mizoram and Manipur states of India' National Medical Journal; 17(5), 245-247
8. Phukan RK, Chetia CK, Ali MS, Mahanta J. (2004) 'Role of dietary habits in the development of esophageal cancer in Assam,the north-eastern region of India' National Medical Journal; 28(14), 178-204
9. Ainworth B.E , Haksell W.L.,Leon A.S., (1993) 'Compendium of physical Activities', Medicine and Science and Sports in Sports and Exercise:25 , (71-80).
10. Colic Baric I., Satalic Z., Lukesic Z: 'Nutritive value of meals, dietary habits and nutritive status in Croatian university students according to gender', Int. Journal of Food Sci Nut; (54) , (473-484)
11. Philogure J .Lawrence, G., (2004) 'Prevalence of Multiple Chronic Disease and Risk factors', American journal of Preventive Medicine,272(2), 18-24.
12. Krinke U: Adult nutrition. (2002) 'In Nutrition through the Life Cycle', Edited by Brown J. Wadsworth / Thomson Learning, Belmont, CA; (383-407).
13. B. Srilakshmi., (2011), 'Dietetics' Nutritional Requirements of Adults,, 6th Edition; 96-107.
14. Kolodinsky J., Harvey- Berino JR.,Berlin L., Johnson RK.,Reynolds TW., (2007) 'Knowledge of current dietary guidelines and food choice by college students,better eaters have higher knowledge of dietary guidance' J Am Diet Association, (107), (1409-1413)
15. Kelly Ann Mcgrath, (2007) 'Dietary pattern Associated with Weight Change in College Students' Virginia Polytechnic Institute, and State University, 31-39.
16. Johnson MF , Nichols JF., Sallis JF.,Calfas KJ., Hovell MF.,(1998), 'Interrelationships between physical activity and other health behaviors among University Women and Men.' Prev Med. (27); 536-544.
17. Racette SB, Deusinger SS., Highstein GR., (1998) 'Weight Changes ,Exercise ,and Dietary Patterns during Freshman and Sophomore Years Of College' J Am Coll Health. (46) ; (189-191)
18. Priya D, Prasanna KS, Sucharitha S, Vaz NC.(2010) 'Body Image Perception and Attempts to Change Weight among Female Medical Students at Mangalore.' Indian Journal Community Med. (35) (2): 316-320.
19. Kremaida L.S., Papadaki A., (2003), 'Differentiating between the effect of rapid dietary acculturation and the effect of living away from home for the first time, on the diets of Greek students studying in Glasgow'(50) (2-3), (455-463).

Assessment of nutrient adequacy of pregnant women who are ICDS beneficiaries in rural West Bengal

Arpita Das and Tanima Bhattacharya

ABSTRACT

With a view to improving the health and nutritional status of pregnant women and lactating mothers, the Special Nutrition Programme has been included as one of the most important components of the ICDS Programme. Malnutrition, endemic poverty and low household incomes over the years have resulted in poor nutritional status of the population in these households resulting in food distress and food insecurity. Growing infants and children, pregnant women and nursing mothers face far greater risk from the nutritional depletion than others due to their greater vulnerability and higher biological needs. This nutritional insecurity in the formative years is addressed through the Special Nutrition Programmes under the ICDS scheme and has proved to be one of the most important food—based interventions in the State. The study conducted deals with the assessment of diet quality and nutritional status of ICDS beneficiaries of Santhal Pregnant mother and lactating mother scheme, a national programme targeted towards their nutrition/health needs. Out of 200 pregnant mother (aged 15-26 years) from Bankura rural blocks as well as Non ICDS beneficiaries of the seam blocks assessed, it was found that the subjects who followed a two meal pattern though monotonous and cereal-based have energy intake less than that of RDA while a substantial proportion of them had inadequate nutrient intake with respect to most of the micronutrients especially iron , folic acid and vitamin A . Therefore, sustained efforts are needed to strengthen the scheme for improving its field-level implementation.

Keywords:

Introduction

Health means not the mere absence of disease but it is the "complete state of the physical, mental and social wellbeing". Health of an individual can be affected by general health condition of the society and vice-versa. Therefore, health of the community needs higher attention while considering the development of a region or a country.(1)

An adequate availability of nutrients during gestation period is probably the single most important environmental factor influencing pregnancy outcome. Although psychological adjustments in nutrient utilization and metabolism are geared to improve the utilization of dietary nutrients during pregnancy, these adjustments maybe insufficient to meet the demands for pregnancy and lactation if the women are in poor nutrient status at conception. An adequate supply of nutrients is required to maintain the delicate balance between the needs of the mother and those of the foetus. An inadequate supply will cause a state of biological competition between the mother and the fetus, in which the well-being of both organisms is at serious risk. The consequences of this undesirable situation on the foetus are well known; but the consequences of under nutrition on the mother are less well documented.(2)

Women are more likely to suffer from nutritional deficiency than men for several reasons, including their reproductive biology, low social status, poverty and lack of education.(3) Women in low-income settings often consume inadequate amount of micronutrients because of resource limitation. Severely malnourished mothers have reduced lactation performance contributing to the increased risk of child mortality.(4) Many programmes have been implemented in order to reduce the intensity of the diseases which has been severely affecting the people in India especially the poor livelihood. ICDS is one of them.(17)

The Integrated Child Development Services (ICDS) was launched in 1975 to improve the health and nutrition status of children in the 0-6 age group by providing supplementary food and coordinating with state health departments to ensure the delivery of the required health inputs, to provide conditions necessary for pre-school children's psychological and social development through early stimulation and education; to provide pregnant and lactating women with food supplements, to enhance the mother's ability to provide proper child care through health and nutrition education and to achieve effective coordination of policy and implementation among the various departments to promote child development.

The present study was undertaken to assess the nutritional status of pregnant women in Santhal

groups with ICDS Beneficiary and non ICDS Beneficiary pregnant women, their dietary habits and effect on health in pregnant condition.

Methodology

Selection of place: The pregnant women were selected from three sub-division in Bankura namely Bankura Sadar ICDS centre, Bishnupur ICDS centre and Khatra ICDS centre comprising of 22 blocks in total.

Selection of Respondents

200 pregnant women from Santhal tribe were selected by purposive sampling method. Pregnant women from ICDS centre who are beneficiaries were taken from Bankura's ICDS centres and non ICDS beneficiary pregnant women from Santhal tribe area in Bankura.

Of the total respondent (n=200), 80 respondents were from ICDS centre who are ICDS beneficiaries and 120 respondents were non ICDS beneficiaries pregnant women who are not connected with ICDS centre.

Data Collection Method

1. Construction of Questionnaire- Keeping in mind the objectives of the present study and the limitations imposed by time and available facilities, the questionnaire was framed in a manner, which was more accurate and less laborious.
   The questionnaire was divided into 4 parts:
   General information comprising ofname, age, occupation, income, and pregnancy associated information such as height, weight before conception, the weight gain, due date and birthweight; and biochemical parameters like hemoglobin level, blood pressure, medical history to note accurately the pregnancy related complications, if any.
   Food frequency questionnaire: to know about the intake of food products, food habit of Santhal pregnant women.
   General awareness questionnaire: About the awareness in respondent.
   Dietary pattern: The last part of questionnaire dealt with the collection of information regarding the food habits, food consumed and avoided during pregnancy.
2. Standardization of the common recipes: The standardized measures were used for caring out the diet survey among Santhal pregnant women. This was done to make accurate recording of quantity of food consumed by the respondent.
3. Collection of Data: Answer to questions regarding personal information of patients was collected by personal interviews. Medical history and other relevant information were collected from medical test reports and case history. Post- delivery information was collected from the hospital records well as through personal communication via personalinterview. Diet survey was conducted using 3-day diet recall method.

Data Processing & Analysis: Percentage was tabulated for the background characteristic of the respondent which included the respondent age occupation, family income, and month of pregnancy, weight gain during pregnancy complication, hemoglobin status, blood pressure.

An average of the three consecutive day's meal consumption was calculated by finding out the macronutrient values consumed by the respondents using the food composition tables given in "the nutritive value of Indian foods. NIN 1994" and the nutrient adequacy was assessed comparing to the RDA given by ICMR.

The result was adjusted using the "reference pregnant women" method [1992 National Nutrition Survey] to permit comparisons with the dietary intake reported in the 2002NNHS.After obtaining the mean value, the standard deviation was calculated for all the macronutrient in the two groups. Percentages were calculated for analysis of the clinical sign and symptom of the pregnant women. Statistical analysis was done by using T- tests to accept the formed hypothesis.

Result and Discussion

Table 1: Economic Status of Santali Pregnant Women

Sl. No.	Economic Status	Percentage (%)
1.	< 5,000 Rupees	39
2.	< 10,000 Rupees	28
3.	10,000- 20,000 Rupees	24
4.	21,000- 30,000 Rupees	12
5.	> 30,000 Rupees	3

From the table it can be concluded that 39% of the total population earns below Rs. 5,000, about 28% of the total population earns below Rs. 10000, 24% and 6% earns Rs. 10,000 to Rs. 20,000 respectively and only 3% of the total population earns more than Rs. 30,000.

Table 2: Mother's Literacy Rate

Sl. No.	Education Level	Percentage (%)
1.	Uneducated	30
2.	Up to 5-8 class	20
3.	10th Class Pass	20
4.	HS Pass	10
5.	Graduate Pass	7
	Postgraduate Pass	3

From Table 2, we can conclude that 30% of the population is uneducated, about 20% of the population is literate up to 5-8 class, 20% of the population are literate up to 10th class, 10%, 8% and 3% are literate up to HS, Graduate and Post Graduate pass respectively. It depicts the fact that only 20% of the total population which is very less, are only educated where ICDS should improve their literacy rate in order to improve their conditions.

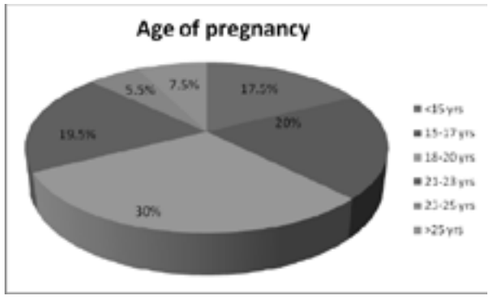

Figure 6: Pie graph represents age of pregnancy in the survey

From the fig 5.3, it can be concluded that, the Santhal women tend to get married at very early age, about 17.5% of tribal women in West Bengal's Bankura district, get pregnant at the age below 15 years, about 20% of women get pregnant between the age of 15-17 years, while most of the women about 30% of the total population are found pregnant between the age of 18-20 years. About 19.5% of the women are pregnant between 21-23 years, 5.5% women get pregnant between 23-25 years, whereas rest of the women is found pregnant after 25 years i.e., 7.5% respectively. From result it is seen that lack of awareness and poor access to the existing Government schemes for women's welfare worsen their situation.

Table 3: Represent the number of pregnancies

Times of Pregnancy	Percentage (%)
1st time	30
2nd time	22.5
3rd time	28.5
4th time	14
More than 4 times	18

Different survey reports suggest that average age of marriage for women in Bankura lies between 15-18 years and it is lower than national average. From the table it is observed that about 30% of the total Santali women are found pregnant for the first time, 22.5% for the 2nd time, while 28.5% and 14% for the 3rd and 4th time respectively whereas, still they are giving birth to their child after having 4 children which is found to be about 18% of the total population. One of the major reasons for increasing number of repetitive pregnancies is due to miscarriages, as most of the women are involved in heavy work duties. As there is a high level of dropouts of tribal females from school as they are habitual to keep themselves isolated from the developed areas, they remain unaware of the population control techniques like using contraceptive pills, condoms, etc. There is a need for ICDS to study the Santhal community exclusively to understand the underlying causes for the particular community to remain so poor and so backward.

5.1 Demography Details of the Respondents:

The demographic details of the respondents taken from Bankura sub divisional ICDS centers and tribal areas are given in table.

Table 4: Demographic Details of the Respondents

		Number of Pregnancy				Type of family	
	Pregnancy Age ±SD	1st child	2nd child	3rd child	More than 3 time	Joint family	Nuclear family
ICDS Beneficiary n=80	21.47±2.39	12.5%	68.75%	18.75%	0	20%	60%
Non ICDS Beneficiary n=120	16.53±2.32	41.6%	29.16%	12.5%	16.6%	30%	90%

The mean pregnancy age of the respondents was found to be WWWWW years. The respondents differed in their number of pregnancy. It was seen that all ICDS beneficiary group respondents were 1st time pregnant 12.5%, 2nd time pregnant 68.75% 3rd time pregnant 18.75%.

Both the groups studied, (ICDS beneficiary and Non ICDS beneficiary) showed a higher trend towards a

nuclear family. Which is the cause of malnutrition of the pregnant women because of high amount of household work, undergo stress, and not receives proper attention.

We also conclude that 29% of the total population are regularly connected to the ICDS centre, 11% are irregularly connected while more than half of the population i.e., 60% are not connected to the ICDS. The reason for this is the regular morning duty that they perform to earn for their living whereas the other reason is that the walking distance of the Santali tribal women is very far from the ICDS centre and therefore they are unable to connect to the ICDS centre regularly.

Table 5: Preferable food of pregnant women given by ICDS centre

Preferred food	Percentage (%)
Khichdi	68
Rice	32

From the table, we conclude that Khichri is more preferred by the Santali pregnant women about 68% and Rice is provided with egg curry which is not very preferable by them i.e.,32%. Thus, we can observe due to low budget of ICDS rice and egg curry is not tastier as compared to Khichri, thus people prefer khichri and egg curry more than Rice and Egg Curry.

5.2 Mean Macro nutrition intake of the respondents
Macro nutrition deficiency, whether clinical or sub-clinical, may affect growth cognition, and reproductive performance. However, though the negative effect of diets low in energy on pregnancy outcome are well documented; less clear are the effect of diets that are low in one or more essential macro nutrients.[5] The mean intake of macronutrients among the respondent of non ICDS pregnant group and ICDS pregnant group are given in table.

Table 6: Mean Intakes of Macronutrients among the Respondents

Nutrients	RDA requirement for pregnant women	ICDS benefi-ciary	Non ICDS benefi-ciary
Protein (gm)	82.5	52.31	41.72
Fat (gm)	70.83	52.87	41.98
Carbohydrate (gm)	564.3	422.44	311.81
Energy(kcal)	3225	2864.3	2430.8

From the above table it evident that there is significant difference between the actual consumption of nutrients and the RDA requirements. It can be due to inadequate intake of nutrients, as they did not include fruits and vegetables in enough amounts in their diet, metabolism and absorption of the nutrients was hampered. They did not include dairy products in the diet which increases value of each nutrient as it is an almost complete food. Consumption of outside food and then skipping the proper dinner also decreases the total nutrient intake. Lack of daily dietary, carbohydrate, protein, fat and energy for a longer period of time leads to malnutrition and the pregnant mother gives birth to baby with low birth weight .The mothers also suffer from anemia and other malnutrition diseases. Other than that improper maintenance of hygiene, lack of knowledge about health and good nutrition also hamper the digestion, absorption in the body and leads to health problems which again affect the overall health.

A study showed that children from lower socio economic classes and larger families had lower daily nutrient intake. They were more likely to have less than the recommended daily intake of calcium and riboflavin.[7]

Another study has been done on school children to assess their eating pattern during lunch time. It has been seen that majority of children did not meet the recommended targets for lunch time's nutrient intake, especially for micronutrients.[8

Table 7: The statistical analysis of nutrient intake of the three day dietary recall)

Nutrients	RDA specifi-cation for pregnant women	Actual consumption		S.D		t-test value compu-ted	t-test value tabu-lated	Null hypothesis rejectected / accepted
		ICDS pregnant women (n=80)	Non ICDS pregnant women	ICDS pregnant women	Non ICDS pregnant women			
Protein (gm)	82.5	52.31	41.72	±3.81	4.3	17.65	1.96	Rejected
Fat (gm)	70.8	52.87	41.98	±4.8	5.56	15.29	1.96	Rejected
Carbohydrate (gm)	564.3	422.44	311.81	±336.9	82.3	3.477	1.96	Rejected
Energy	3225	2864.3	2430.8	±255.3	374.94	9.14	1.96	Rejected

It was seen that respondents of ICDS group having a higher macro nutrition intake had higher maternal weight gain and higher birth weight as compared to respondents of non ICDS group. Studies have also indicated maternal intake of fat [9] and carbohydrate [10] to be positively associated with birth weight of the infant.

5.3 General problem during pregnancy
Table 8: Table showing general problems during pregnancy in to ICDS and Non ICDS Pregnant Women.

Dietary problem	ICDS pregnant women (n=80)	Non ICDS pregnant women (n=120)	Total (n=200)
Nausea	12.5% (n=10)	21.6%(n=26)	18% (n=36)
Vomiting	21.25% (n=17)	45.8%(n=55)	36% (n=72)
Heartburn	6.5% (n=13)	33.3%(n=40)	26.5% (n=53)
Morning sickness	0	50%(n=50)	25% (n=50)
Fatigue	7% (n=14)	25%(n=30)	22% (n=44)
Constipation	7%(n=14)	8.3%(n=10)	12% (n=24)
Anorexia	2.5% (n=5)	16.6%(n=20)	12.5% (n=25)
Edema	12.5 (n=10)	12.5%(n=15)	12.5% (n=25)
Leg cramp's	7% (n=14)	8.3%(n=10)	12% (n=24)
Allergy	6% (n=12)	15%(n=18)	15% (n=30)
Anemia	2.5% (n=5)	33.3%(n=40)	22.5% (n=45)
Diarrhea	2.5% (n=5)	20.8%(n=25)	15% (n=30)

From the above table 4, we can observe that the women who are regular connected to the ICDS scheme are less affected by the anemia 2.5%, morning sickness 0%, nausea 12.5%, vomiting 21.25% heartburn 6.5%, fatigue 7%, constipation 7%, anorexia 2.5%, eadema 12.5%, allergy 6% and diarrhea 2.5% as compared to the non ICDS or irregularly connected women with ICDS nausea 21.6%, vomiting 45.8%, heartburn 60.5%, morning sickness 50%, fatigue 2%, constipation 8.3%, anorexia 16.6%, edema 12.5%, leg cramp's 9.3%, allergy 15%, anemia 33.3%, diarrhea 20.8% ICDS provides the regular checkup ,providing hygienic food and education about hygiene, medicines and free medical checkups.

5.4 Weight gain (kg) during pregnancy and Birth weight (kg) of body
Several reports have indicated that maternal weight gain is an important determinant of newborn size of pregnancy[8,11] and is positively associated with birth weight further birth weight is an important correlated of neonatal and health and has been recently with adult diseases including Anaemia Hypertension Heart burn Constipation disease. To correlate the association between maternal nutrition, maternal weight gain and the birth weight of the infant.

Table 9: Table showing Weight gain (Kg) during pregnancy

Weight category based on BMI	ICDS beneficiary (n=80)	Non ICDS beneficiary (n=120)	Total (n=200)
Under weight (BMI<19.8)	18.75% (n=15)	66.6% (n=80)	47.5% (n=95)
Normal weight (19.8- 26)	62.5% (n=40)	25% (n=30)	30% (n=60)
Over weight (>26)	6.2% (25)	12.5% (n=10)	17.5% (3n=35)

From the table 5 it can be seen that respondents of Non ICDS beneficiary pregnant women had high underweight tendency (66.6%) as compare to respondents of ICDS beneficiary pregnant women (18.75%) Similar results of maternal weight gain.

5.5 Birth Weight of the Infants
Any abnormality in intra- uterine environment can detrimental to foetal growth. Adequate maternal nutrition is essential to prevent of other conditions. Good nutrition-that is, nutrition containing adequate amounts of arrange of nutrients is essential for foetal growth and resultant birth weight of infant.

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Table 10: Table showing Birth weight (kg) of infants

Birth weight 0	ICDS beneficiary (n=80)	Non ICDS beneficiary (n=120)	Total (n=200)
Under Weight (<2.5)	----	79.16% (n=95)	47.5% (n=95)
Normal Weight (>2.5)	100% (n=80)	20.8% (n=25)	52.5% (n=105)

The data from the table 6 shows that the 47.5% babies have under weight and 52.5% babies having normal birth weight. But in ICDS beneficiaries are low in underweight right than the non ICDS beneficiaries group.

Conclusion

An adequate availability of nutrition during gestation is probably the most important environmental factor influencing pregnancy outcome.[12,13] Poor maternal nutrition during pregnancy, is a major cause of low birth weight (L.B.W) in developing countries [14,15].Low birth weight is associated with an increased risk of morbidity and mortality.[16]

From the study conducted it was observed that Non ICDS Santhal pregnant women suffered from macronutrient deficiency as well as malnutrition disease like anemia during pregnancy though deficiency of all the macronutrient was seen in both the groups statically. The mean protein and fat intake was found to be lower amongst the respondent of non ICDS beneficiary Santhal pregnant women. as compared to the respondents of ICDS beneficiary Santhal pregnant women.

Some data revealed that the nutritional and health status of ICDS beneficiary pregnant women are better compared to the non ICDS beneficiaries, and they can even give birth to babies with normal weight. So motivation is required for the non ICDS beneficiaries so that they attend the health checkup camps, knowledge and skill upgradation and even the supplementary program and mid day meal program, which can influence their health status thus betterment may be observed in them.[17]

References

1. Houdegbe. A Discussion on health problem among rural women in Africa, Child Trop,15(6),57- 61,(1995)
2. Aneja B, Singh P, Tandon M, Pathak P, Singh C, Kapil U. Etiological Factors of Malnutrition among Infants in Two Urban Slums of Delhi. Indian Pediatrics; 38: 160-165.2001
3. Dietary guidelines for Indians- a manual. Hyderabad: National Institute of Nutrition, Indian Council of Medical Research, ,73(1),255-260,1998.
4. Deshpande PV, Anupama V, Wasu and Mujumdar S. Knowledge level about health practices among rural women, Mah. J. Exten. Edu., 18(5), 311-312, 1994.
5. Geervani P, Jayshree G. A study of nutritional status of adolescent and adult pregnant and lactating women and growth of their infants. Journal of Tropical Pediatrics 34(5),58-59,1988.
6. Jelliffe D. The nutritional status of the community, Geneva, WHO14(7), 10 - 96,1966.
7. Nutritive value of Indian foods. Hyderabad: National Institute of Nutrition, Indian Council of Medical Research,:8(1) 47-67,1989.
8. Chandra, K. Singh. Health problems in rural Haryana. Rural India, Dec,9(1) 234-356,2006.
9. Bhalani KD, Kotecha PV. Nutritional status and gender differences in the children of less than 5 years of age attending ICDS Anganwadis in Vadodra city. IJCM 2002; 27: 124-129.2009
10. Spear B. Adolescent growth and development. In: Rickert VI, ed. Adolescent nutrition - assessment and management. New York: Chapman and Hall, Thompson Publishing, 7(2),13-24,1996.
11. Islam et al. Nutritional status of women in Bangladesh: comparision of energy intake and nutritional status of a low income rural group with a high income urban group, J Health Popul Nutr,2004, 28(1):86–94, (1995).
12. Kumari R. Rural female adolescence: Indian scenario, Social Change: Issues and Perspectives; 25(2,)55-56,1995.
13. LadipoOA,Nutrition in pregnancy, mineral and vitamin supplement, American Journal of clinical nutrition,72(1),280-290,2000.
14. Lee K.S Ferguson RM ,Corpuz M and Gartner L.M ,Maternal age and incidence of low birth weight at term, a population study .American Journal of Observation and gynecology,158(2),38-40,1991.
15. Satyanarayan K, Naidu AN, Swaminathan MC, Rao BSN. Effect of nutritional deprivation in early childhood on later growth - a community study without intervention. Am J Clin Nutr; 34(4), 136-147,1981.
16. Ene-Obon. Determinants of Health and nutritional status of rural nigerian women, J Health PopulNutr Dec;19(4), 320-330,2001.
17. Yankanchi GM, Naik Rama K, Gaonkar V, Nutritional status of rural pre- schoolchildrenbyanthropometry.IndianJ. Nutr.Diet.;39(2),402-404,2002

Dyeing of Cotton with Red Sandalwood

Rakhi Agarwal and Deepali Singhee

ABSTRACT

In this study, cotton has been dyed with aqueous extract of red sandalwood. The effect of varying conditions of extraction and dyeing process variables on surface colour strength as well as fastness (light, wash, and rub) has been studied and optimum values established. Temperature, dye concentration and MLR have been identified the predominating dyeing parameters for red sandalwood as indicated by the widely dispersed CDI values of dyed cotton fabric.

Keywords: Colour fastness, cotton, red sandalwood, natural dyes

Introduction

Natural dyes have been a part of human life since time immemorial. Egyptians mummies and documents from the Mughal period bear testimony to the use of these natural dyes in the ancient times. India was known as the colour box of the ancient world. According to statistics, about 500 plant sources were available in India which could produce colouring matter.

However, with the invention of synthetic dyes in 1856, the use of natural dyes took a back seat. The main reason behind this was the simplified process of application, ease of the natural ones. Synthetic dyes possess moderate to excellent colour fastness, while natural dyes have inadequate degree of fixation and fastness properties [Samanta & Agarwal 2009]. Since the synthetic dyes are produced in high quantities and that too at a low cost, they replaced the natural dyes almost completely and the use of natural dyes declined [Vankar et al. 2007].

It was found that during the manufacture of synthetic dyes, many carcinogenic chemicals are required and some synthetic dyes contain non eco-friendly carcinogenic amines/ toxic substances [Vankar & Shanker 2009]. These are not only harmful to the environment, but also to the wearer wearing apparel dyed with synthetic dyes.

With the present national and international awareness on environment, ecology and pollution control, there is a growing demand for eco friendly textiles. Consumers are demanding natural products dyed with natural dyes. They not only want a quality product at a right time and at reasonable prices, but also with no harm to the ecology during manufacture as well as during use. In this context natural dyes that exhibit better biodegradability and have a higher compatibility with the environment that their synthetic counter parts looks more attractive.

Literature survey has shown that dyeing with natural dyes is associated with uncertainty of results. Other eminent problems encountered in the use of natural dyes are lack of availability due to difficulty in collection of the source material as well as standardization of the methods of application that would give reproducible shade [Bhargava & Shahnaz 2013]. The cost of dyeing with these dyes is also very high [Islam & Leyla 2012], hence using agro, forest or industrial waste that are available free of cost. Also in the absence of any precise technical of dyeing with these dyes is also very high, hence, in the knowledge and written scientific records, there is a need to study and standardize the method of application of these dyes to promote its use and prevent shade variation from one batch to the other. Hence the present study on dyeing cotton with red sandalwood has been undertaken.

Methodology

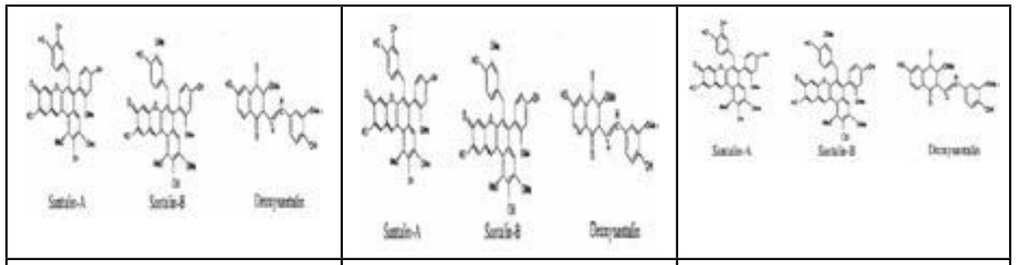
Materials

Bleached, undyed and plain woven 100% cotton fabric with fabric thickness of 26mm, total crease recovery angle of 1690 and with an area density of 63g/m2 was used.

Laboratory reagent (LR) grade aluminium sulphate 16-hydrate of E-Merk brand obtained from Sova Chemicals Pvt Ltd. was used as the metal mordant. Dried red sandalwood powder was obtained from Bada bazaar in Kolkata.

The red colorants of the insoluble class of red sandalwood are mostly compounds presenting associated with isoflavanoid – flavanoid. The heartwood of red sandalwood contains up to 17% of such flavanoids, santalins A and B and a small amount of Santalin C (dioxy-santalin), grouped respectively as CI Natural Red 22 and CI Natural Red 23. Santalin, the main colouring component has a quinonoid structure. Red sandalwood has

been classified as a mordant dye. However, has also been classified as a disperse dye because it has an electron withdrawing group (OCH_3) [Gulrajani et al. 2002].



Santalin-A	Santalin-B	Santalin-C (Deoxy-santalin)

Figure 1: Colouring components in red sandalwood

Methods

Desizing of cotton fabric

The bleached cotton fabric was desized using 20 ml/l (3.6% HCl) at 100^{0}C for 60 min using MLR 1:10 [Samanta et al. 2007b].

Scouring of cotton fabric

The desized cotton fabric was further scoured using 2-3 gpl NaOH and 2-3gpl of non- ionic soap at 100^{0}C for 120 min using MLR 1:20 and pH at 12 [Kumar et al. 2012].

Aqueous extraction of colour from red sandalwood

The coloring matter from the red sandalwood was extracted by soaking 5gm of the powdered wood in 100ml of water for 90 minutes. Extraction of the dye was carried out at variable process conditions of temperature, time, MLR and pH as mentioned in Table 1. The extracted liquor was finally filtered using a muslin cloth. The extraction conditions were optimized on the basis of optical densities of the solution i.e. the one with the highest optical density was considered optimum.

Table 1: Variation in process conditions of temperature, time, pH and dye concentration - MLR for optimizing conditions for extraction of colour from red sandalwood

Parameter	**Varying Conditions**
Temperature (^{0}C)	RT, 40, 60, 80, 100
Time (in minutes)	15, 30, 45, 60, 75, 90
MLR	1:10, 1:20, 1:30, 1:40, 1:50
pH	2, 4, 7, 9, 11

Pre-treatment of scoured cotton with myrobolan extract

The scoured cotton fabric was treated with myrobolan (harda) before pre-mordanting it with aluminium sulphate. For this 10% (owf) harda was soaked overnight using MLR of 1:10 and was then extracted at 80^{0}C for 30 min. The solution was strained thoroughly using a muslin cloth and the cotton fabric treated with this solution at 80^{0}C for 30 min and dried in air without washing [Samanta et al 2007a].

Pre-mordanting of myrobolan treated cotton fabric with mordant (aluminium sulphate)

Myrobolan treated cotton was treated with varying concentrations (10% to 50%) of aluminium sulphate calculated on the weight of the fabric (owf) at 60^{0}C for 30 mins using MLR 1:20. After mordanting, the samples were rinsed thoroughly in running water and finally air dried. Mordant concentration was optimized on the basis of minimum strength loss, maximum color yield and good fastness of pre-mordanted cotton.

Dyeing of aluminium sulphate pre-mordanted cotton with aqueous extract of red sandalwood

The aluminium sulphate pre-mordanted cotton samples were dyed on a water bath using the aqueous extract of red sandalwood extracted under optimized conditions established earlier in the present study under variable conditions of time, temperature, pH and MLR. While varying one parameter the other were kept constant at time- 90 min, temperature - 80oC, MLR - 1:20 and pH- 11. After dyeing the samples were rinsed thoroughly in running water and air dried in shade.

Testing and Evaluation

All the dyed samples were conditioned for 48 hr at 65% (± 2%) RH and 27^{0}C (± 2^{0}C) as per standard IS: 6359-1917 [Mahagande et al. 2009] for each method of testing and evaluation described below:

Measurement of breaking tenacity and breaking extension

Wrap-way and weft-way breaking tenacity (cN/tex) of pre-mordanted fabric were measured after conditioning following the raveled strip method

(sample size of 10 cm × 2.5 cm) as per IS:1969:1968 [ASTM 1979] using Instron (model-1445) CRT-Universal tensile tester with a traverse speed of 100mm/min and a pretension of 0.5 N. The final gauze length (sample size) of the fabric sample was 50 mm × 20 mm after raveling.

Estimation of surface colour stength and related colour interaction parameters

The K/S value is considered as an index for the surface dye uptake, i.e higher the K/S value, higher is the surface dye uptake of the sample

Surface colour strength of cotton fabric samples was estimated in terms of K/S values (Kubelka Munk function) by measuring surface reflectance of each of the dyed samples at their respective λmax using a Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software for converting the reflectance values to K/S using the following relationship. [Bhattacharya & Shah 2000]

$$K/S = \frac{(1-R_{\lambda max})^2}{2R\,\lambda_{\lambda max}} \alpha \quad C_D$$

where, K = coefficient of absorption, S = coefficient of scattering, Rλmax = surface reflectance value of sample at wavelength of where maximum absorption occurs for a particular constant, λmax = maximum absorbance wavelength and CD = concentration of dye.

Total colour difference (ΔE), lightness/darkness (ΔL*), redness/greenness (Δa*), blueness/yellowness (Δb*), change in chroma (ΔC*), and change in hue ((ΔHab), values were measured before and after dyeing to compare the shade depth and color differences of each dyed sample against particular undyed (bleached / mordanted) standard sample using a Premier Color Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software using the following CIE-lab equations [Eom et al. 2001]:

General metamerism index (MI) was calculated employing the Nimeroff and Yurow's equation [Fairman, 1987].

A newer colour interaction parameter called Colour Difference Index (CDI) which indicates the combined effects of different known individual colour difference parameters between any two samples when dyed with varying shade under different conditions of dyeing has also been used in the present work as postulated earlier [Samanta et al. 2011] to understand the combined effects of different dyeing variables by a single parameter.

$$\text{Colour difference index (CDI)} = \frac{\Delta E \text{ X } \Delta H}{\Delta C \text{ X MI}}$$

Evaluation of colour fastness

Colour fastness to light was determined as per IS: 2454-1984 [ISI 1989] method using Xenon Arc lamp. Colour fastness to washing of the dyed cotton samples was determined as per IS: 764-1984 method [ISI 1982] following IS-2 (equivalent to ISO-II) and IS-3 (equivalent to ISO-III) fastness methods using a Sasmira launder-o-meter and relevant standard grey scales (ISO-105-AO2 and ISO-105-AO3. Colour fastness to rubbing (dry and wet) was assessed as per IS: 766-1984 [ISI 1982] method using a motorized semi-automatic digital crockmeter from MAG Solvics Pvt. Ltd., India.

Results & Discussion

Determination of the wavelength of maximum absorbance for aqueous extract of red sandalwood

The absorbance/optical density of any specific coloured solution (aqueous extract of a natural dye) vary widely at different wavelengths, and there is always a specific wavelength of maximum absorbance for a particular dye solution depending on its predominating hue, chroma and value. For measurement of any colour parameter when the difference between two coloured samples is required, the difference can be maximized/made predominant at the position of maximum absorbance points. Hence, the difference in absorbance peaks between an undyed and dyed sample is always proportionately higher at the maximum absorbance wavelength (λ_{max}) of the concerned aqueous dye solution. This the reason why maximum absorbance wavelength of 1% aqueous extracted solution of red sandalwood was identified by evaluating the relative optical densities of the aqueous extracted solutions of the natural dye (extracted at 80^{0}C for 60 min using 1 gm of dry source material of the said natural dye in 100 ml of water) at different wavelengths in the visible range (400 to 700 nm) using Hitachi-U-2000 UV-VIS absorbance spectrophotometer. The aqueous solution of red sandalwood shows maximum optical density (2.9) at 440 nm. This maximum absorbance wavelength (λ_{max}) was used for all further measurement of all colour parameters.

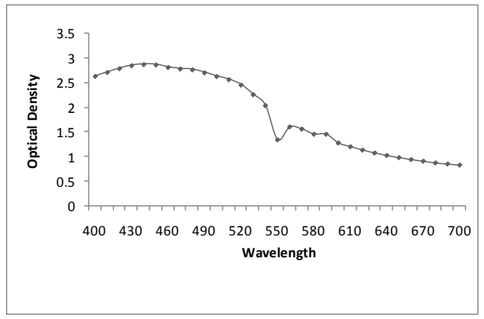


Figure 1: Optical density of red sandalwood extract at different wavelengths in the visible range (400-700nm)

Optimization of extraction conditions

Before starting the dyeing of cotton with aqueous coloured extract of red sandalwood, the conditions for extracting the natural colour (dye) from its source (wood) were standardized / optimized. Table-2 indicates the relative optical densities of the extracted solution of red sandalwood for different time periods and temperatures. It can be seen from the table that when extracted at 100^{0}C at pH 11 using MLR 1:10 when extracted for 45 minutes aqueous extract of red sandalwood showed highest optical density at the maximum wavelength (440nm).

Table 2. Optical density at λ_{max} of aqueous extract of red sandalwood extracted under different process conditions (440 nm)

Process Variables	**Optical density at λ_{max} (440 nm for Red Sandalwood)**
Variation in Time (in minutes)	
15	2.7
30	3.1
45	3.4
60	3.3
75	2.9
Variation in Time Temperature (in ^{0}C)	
RT	1.3
40	1.2
60	1.9
80	2.2
100	3.1
Variation in MLR	
1:10	3.5
1:20	2.7
1:30	3.2
1:40	2.2
1:50	1.7
Variation in pH	
1	2.2
4	2.1
7	2.7
9	3.1
11	3.4

Highest optical density at the λ_{max} (440nm) is obtained when extracted is carried out for time - 45 min, temperature - 100^{0}C, MLR - 1:10 and pH - 11.

Optimization of the mordant concentration

Treatment with different concentrations of the mordant (aluminium sulphate) shows some loss in the warp-way and the weft-way tenacity of the cotton fabric after the mordanting. For the loss in tensile strength in both the warp and weft directions increase with increase in the mordant concentration upto 25% (owf). The loss in tenacity is always higher in the warp direction as compared to the weft direction; expect when 50% (owf) or more of aluminium sulphate is used as mordant. The higher loss of strength in the warp direction compared to that in the weft direction may be due to more shrinkage that arises in the warp direction as a result of exposure of the warp yarns to more tension during weaving rendering it more vulnerable to strength loss than the relatively more relaxed weft yarns. Least loss in strength in both directions (respectively 1.2% in the warp and 1.0% in the weft) is obtained when 35% (owf) of mordant is used.

Table 3. Tenacity (cN/Tex) of scoured cotton pre-mordanted with varying concentration of the aluminum sulpahte

Mordant Concentration	**Tenacity (cN/tex)**	
	Warp	**Weft**
NIL (scoured cotton sample)	9.9	6.0
10%	9.57 (2.9%)	5.8 (2.2%)
15%	9.6 (3.1%)	5.8 (2.9%)
20%	9.5 (3.8%)	5.8 (2.9%)
25%	9.4 (4.3%)	5.8 (2.9%)
35%	9.7 (1.2%)	5.9 (1.0%)
50%	9.6 (2.3%)	5.6 (6.2)

*data in the parenthesis are the corresponding strength loss in percentage.

K/S increases with increase in the mordant concentration and mordanting with 35% (owf) aluminium sulphate gives highest surface colour strength (K/S- 2.75), on pre-mordanted cotton fabric dyed with aqueous extract of red sandalwood.

Table 4. Surface colour strength (K/S) evaluated at λmax (400 nm) and wash fastness (ISO-II) of silk fabric pre-mordanted with varying concentration of the mordant and then dyed with red sandalwood extract

Mordant Concentration	**K/S at λ_{max} (440 nm)**	**Wash Fastness (ISO-II)**		
		LOD	**ST**	
			C	**CW**
NIL	1.5	1	3-4	3-4
10%	2.2	1-2	4	3-4
15%	2.5	1-2	4	3
20%	2.4	1	4	3-4
25%	2.3	1	4	3
35%	2.8	1	4	3
50%	1.8	1	4	3-4

LOD – Change in depth of shade, ST – Extent of staining, C – Cotton, CW-Cotswool

The highlighted data correspond to the optimum values

Variation in mordant concentration does not have much impact on the fastness properties of cotton pre-mordanted with aluminium sulphate (as mordant).

Optimization of dyeing process conditions

With respect to surface colour strength and other colour interaction parameters

Dyeing process variables have been optimized on the basis of maximum and uniform colour yield and good fastness properties, cotton aluminium sulphate pre-mordanted cotton dyed with red sandalwood extract (Table 5).

Keeping all other variables fixed, an increase in the time of dyeing (15–90 min) increases the K/S values till 30 min after which it starts decreasing. This may possibly be due to the achievement of dyeing equilibrium at 30 min for this dye. Also there maybe some desorption / breaking of dye-fibre-mordant complex beyond 30 min which leads to a decrease in the K/S value for dyeing time above 30 min.

Increase in the dyeing temperature from room temperature to 100^{0}C increases in the surface colour strength (K/S) till 40^{0}C after which it decreases.

With an increase in the pH from 2 to 11, there is a noticeable decrease in the K/S values.

Keeping other variables constant, the K/S value of the dyed samples increases till 1:30 MLR beyond which it decreases.

There is a slow increase in K/S value with the increase in dye concentration from 25-100% (calculated on the basis of the weight of dry red sandalwood powder). This increase is noticeably highest at 100% concentration. Hence, 100% dye concentration is taken as the optimum value.

Table 5 also shows the effects of the different process variables other colour interaction parameters, like total colour difference (ΔE), changes in chroma (ΔC), general metamerism index (MI) and the colour different index (CDI) values. It is interesting to note that among the dyeing conditions (time, temperature, pH, MLR and dye conc.) varied, the most important and predominating variable has been identified as dyeing temperature, pH and dye concentration of the dye-bath as indicated by the wide dispersion of CDI values. Dispersion of CDI for variation in dyeing temperature from RT to 100^{0}C is 0.9; while for variation in pH it is 2.3 and that for variation in dye concentration is 1.0. For variation in time, it ranges form 1.9 to 3.0 and for variation in MLR, it is 1.9 to 3.1. The order of increasing CDI values therefore appears to be as follows:

Temperature < Dye Concentration < MLR < Time < pH

Therefore, for uniform dyeing of cotton and other cellulosic fibres using red sandalwood extract, special care should be taken to control of MLR and pH of the dye-bath for uniform shades.

The higher range of ΔE value (>50) is observed for the variation in time, temperature, pH and dye concentration, indicating that these three are the major controlling parameters responsible for uniform dyeing. ΔL* value in case of all process variables are found to be negative indicating darker shade/tones of dyed cotton. Δa* is always greater than the corresponding Δb* indicating a more redder/greener tone.

Changes in hue ΔHap for all the cases are found to be negative, indicating that there is no major change in predominating hue, except showing some hypsochromic shift in the colour / tone. However, the maximum negative ΔHap value is again observed in case of the variation in temperature (40^0-80^{0}C), pH (2) and dye concentration (100%), which further indicates the colour yield for this natural dye

obtained from red sandalwood, is sensitive to these three dyeing process variables.

The general metamerism index indicates the metameric effect on red sandalwood dyed cotton fabric for different conditions of dyeing. In all the cases, the MI varies from 2.5 to 5.5 and the data are widely dispersed within a particular condition being varied, and also varies to a noticeable degree from one condition to other, indicating potent metameric effect from one varying condition to the other.

Table 5: Colour strength and related parameters of aluminium sulphate pre-mordanted cotton fabric dyed with aqueous extract of red sandalwood under variable conditions of dyeing

Varying Parameters	**K/S at λ_{max}**	**ΔE**	**ΔL***	**Δa***	**Δb***	**ΔC***	**ΔHab***	**MI (LABD)**	**CDI**	**RCR (CDImax - CDImin)**
Scoured and Alum pre-mordanted cotton (CONTROL)	1.1	8.6	-1.4	5.0	5.6	3.9	-2.1	2.6	0.6	---
Variation in Time (in min)										
15	2.2	15.1	-5.4	10.6	9.3	12.3	-7.0	3.9	2.6	1.5
30	3.0	17.7	-10.6	11.0	9.0	12.2	-7.2	4.00	3.4	
45	2.2	14.2	-6.3	10.0	7.6	10.5	-6.9	3.7	2.5	
60	2.0	14.3	-4.2	10.1	9.2	11.9	-6.8	3.8	2.4	
75	1.4	12.2	0.4	9.6	7.6	10.2	-6.7	3.6	1.9	
90	1.2	13.6	-5.2	9.3	8.5	10.8	-6.4	3.5	2.3	
Variation in Temperature (^{0}C)										
RT	2.2	11.6	-4.1	5.6	9.3	10.0	-4.2	2.5	1.9	0.9
40	2.7	14.8	-8.8	8.6	8.2	10.2	-6.0	3.2	2.7	
60	2.0	12.5	4.6	8.2	8.3	10.1	-5.8	3.1	2.1	
80	1.7	12.0	2.5	9.1	7.5	9.9	-6.4	3.4	1.9	
100	1.9	12.3	4.7	8.8	7.2	9.5	-6.3	3.2	2.1	
Variation in pH										
2	3.1	21.9	-11.7	16.3	8.8	15.7	-9.9	5.5	4.2	2.6
4	2.8	18.7	-9.9	13.5	8.5	13.4	-8.6	4.7	3.6	
7	1.7	10.9	-0.7	8.0	7.3	9.2	-5.8	3.1	1.7	
9	1.6	11.6	-1.0	8.4	7.9	9.9	-5.9	3.2	1.8	
11	1.4	11.9	0.9	8.2	8.5	10.3	-5.8	3.2	1.9	
Variation in MLR										
1:10	2.0	11.3	-4.4	7.2	7.4	8.9	-5.3	2.8	1.9	1.2
1:20	2.1	13.2	-5.4	8.9	8.6	10.3	-6.2	3.3	2.5	
1:30	2.2	14.0	-5.1	9.4	9.0	11.3	-6.4	3.5	2.4	
1:40	2.0	14.3	-5.5	11.1	7.1	10.8	-7.6	4.0	2.5	
1:50	1.3	16.3	-8.0	12.4	6.8	11.5	-8.3	4.4	3.1	
Variation in Dye Concentration (5)										
25	1.2	9.5	-7.5	6.7	6.1	7.4	-5.1	2.5	1.5	1.0
50	1.6	10.1	-1.6	7.5	6.5	8.2	-5.6	2.8	1.5	
100	2.2	14.5	-5.3	9.6	9.4	11.8	-6.5	3.6	2.4	

dE – total colour difference, dL – lightness/darkness, da – greenness/redness difference, db – blueness/yellowness, dH – change in hue, dC – change in chroma, MI – metamerism index, CDI – colour difference index

The highlighted data correspond to the optimum values

Colour fastness (washing, rubbing and light)

Light fastness of cotton pre-mordanted with aluminium sulphate and then dyed with aqueous extract of red sandalwood ranges from 1-2 to 3 and is highest for the process conditions optimized in terms of surface colour strength. Wash fastness with respect to loss in depth of colour for aluminium sulphate pre-mordanted cotton dyed with red sandalwood extract ranges from good to poor (3 to 1). Wash fastness with respect to extent of straining ranges from good to very good. Again in most cases, the corresponding fastness for each variable is slightly better for ISO-III as compared to ISO-II. Also there is not much variation in fastness with the dye process variable or from one variable to another. There is not much variation in the wet and dry rub fastness of dyed cotton samples with respect to the variation in the conditions of the dyeing parameters and it ranges from good to very good. The wet rubbing fastness is either same or marginally lower in most cases than the corresponding dry rubbing fastness.

Table 6: Colour fastness properties of aluminium sulphate pre-mordanted cotton fabric dyed with aqueous extract of red sandalwood (RS) under variable conditions of dyeing

Variables	**LF**	**Wash Fastness**						**Crocking Fastness**	
		ISO-II			**ISO-III**			**Dry**	**Wet**
		LOD	**ST**		**LOD**	**ST**			
			Cot	**CW**		**Cot**	**CW**		
Variation in Time (in min)									
15	2	1	3-4	3	1	3-4	3	3	3
30	2	1	4	3	1	4	2-3	4	3
45	1-2	1	4	2-3	1	4	2-3	4	2-3
60	1-2	1	4	3	1	3-4	2-3	4	3
75	1-2	1	4	3	1	3-4	2-3	4	3
90	2	1	4	3	1	4	2-3	4	3
Variation in Temperature (^{0}C)									
RT	3	1-2	4	3	1	3-4	2-3	3-4	2-3
40	3	1-2	4	3	1	3-4	2-3	4	3
60	2	1-2	3-4	3	1	3-4	2-3	4	3
80	2	1-2	4	3	1	3	2-3	3	2-3
100	3	1-2	4	3	1	3-4	2-3	4	2-3
Variation in pH									
2	3	1	4	2-3	1	3-4	3	4-5	3
4	3	1	4	2-3	1	3-4	3	3-4	3
7	3	1	4	3-4	1	3	3	4-5	3
9	2	1-2	4	3-4	1	3-4	3	4	3-4
11	2	1-2	4	3-4	1	3-4	3	4	3
Variation in MLR									
1:10	2	1	4	3	1-2	3-4	3	4	3
1:20	2	1	4	3	1-2	3-4	2-3	4	3
1:30	2	1	4	3-4	1	3-4	2-3	4-5	3
1:40	2	1-2	4	3	1-2	3-4	2-3	4	2
1:50	2	2	4	3-4	1	3-4	2	4	3
Variation in Dye Concentration									
25	2	1	3-4	4	1	3-4	2-3	4	3-4
50	2	1	3-4	4	1	4	2-3	4	4
100	2	2	3-4	3	1	3-4	2-3	3	3-4

LT – Light fastness, LOD – Loss in dept of shade, ST – Extent of staining, Cot – Cotton, CW- Cotswool

Conclusion

Shades of red brown to rust have been obtained on cotton pre-mordanted with aluminium sulphate and dyed with aqueous extract of red sandalwood. Darker shades result which dyeing was carried out under acidic pH and increase in MLR and dye concentration intensifies the colour tone. The optimized extraction condition for red sandalwood with respect to the highest optical density is time - 45 min, temperature - 100^{0}C, MLR - 1:10 and pH - 11. 35% (owf) mordant concentration is found to be optimum with respect to both minimum strength loss and maximum surface colour strength for the treated and dyed cotton fabric. The conditions of

dyeing aluminium sulphate pre-mordanted cotton with aqueous extract of red sandalwood has been optimized as follows: time- 30 mins, temperature- 40^{0}C, pH-2, MLR-1:20 and dye concentration- 100% (owf). Uniform dyeing of cotton and other cellulosic fibres with red sandalwood extract, special care should be taken to control of pH and MLR of the dye-bath apart from the dye concentration as indicated by a wide dispersion of CDI.

References

1. Annual Book of ASTM Standards (American Society for Testing & Materials Philadelphia, Pennsylvania, USA), 1979, E23-02: 219.
2. Bhargava D, Shahnaz J. Microbial dyes: A new dimension to natural dyes. Colourage, 2013; 60(7):42-43.
3. Bhattacharya SD, Shah AK. Metal ion effect on dyeing of wool fabric with catechu. J Soc Dyes Color, 2000; 116(1):10.
4. Eom Seong-il, Shin Dong-yoon, Kee-jong Yoon. Improving the dyeability of natural colorants on cotton by cationization. Indian J Fibre Text. Res., 2001; 26(4):425-431.
5. Fairman HS. Metameric correction using parameric decomposition. Colour Research Application, 1987; 12:261-265.
6. Gulrajani ML, Bhaumik S, Oppermann W, Hardtmann G. Kinetic thermodynamic studies on red sandalwood. Indian Journal of Textile Fibre Research, 2002; 27:91-94.
7. ISI (BIS) Handbook of Textile Testing, Bureau of Indian Standards, New Delhi, 1982: 24, 221, 255, 536, 553, 571, 573.
8. ISI (BIS) Handbook of Textile Testing, SP: 15 (Part-4-1988),Bureau of Indian Standards, New Delhi, 1989:115,157-158.
9. Ismal ÖE, Leyla Y. Almond shell as a natural colorant. Indian Journal of Fibre Textile Research 2012; 37(4):358-363.
10. Kumar D V, Boopathi N, Karthick N, Ramesh P. Aesthetic Finishes for Home Textile Materials. International J. Text Science, 2012; 1(3):5-9.
11. Mahangade RR, Varadarajan PV, Verma J K, Bosco H. New dyeing technique for enhancing colour strength and fastness properties of cotton fabric dyed with natural dyes. Indian J Fibre Text. Res., 2009; 34(3): 279-282.
12. Samanta AK, Agarwal P and Datta S. Dyeing of jute and cotton fabrics using Jackfruit wood extract: PartI- Effects of mordanting and dyeing process variables on colour yield and colour fastness properties. Indian J Fibre Text. Res., 2007a; 32(4): 466-476.
13. Samanta, A.K., Singhee D, Basu G, Mahalanabis KK. Hydrogen peroxide and potassium per-oxo-disulphate combined room temperature bleaching of jute, cotton and jute-cotton union fabrics – An energy-efficient ecofriendly process. Indian Journal of Fiber & Research, 2007b; 32: 221-231.
14. Samanta AK, Konar A, Chakraborti S, Datta S. Dyeing of jute fabric with tesu extract: Part 1- Effects of different mordants and dyeing process variables. Indian J Fibre Text. Res., 2011; 36(1): 63-73
15. Samanta AK & Agarwal P. Application of natural dyes on textiles. Indian J Fibre Text Res, 2009; 34(4): 384-399.
16. Vankar P, Shanker R. Eco-friendly pretreatment of silk fabric for dyeing with Delonix regia extract. Coloration Technology, 2009; 125(3): 155-160.
17. Vankar P, Shanker R, Verma. Enzymatic natural dyeing of cotton and silk fabrics without metal mordants. Journal of Cleaner Production, 2007; 15: 1441-1450.

Eco-friendly dyeing of silk with Ashoka bark (Saraca asoca)

Nidhi Gupta, Yamini Dhanania and Deepali Singhee

ABSTRACT

The present study explores the use of easily and abundantly available natural resources like Ashoka bark that have so far remained unexplored for the purpose of dyeing textiles. In the study, silk has been dyed with aqueous extract of Ashoka bark (Saraca asoca) using aluminium sulphate as a mordant. The effect of variation in the process conditions of extraction (time, temperature, MLR and pH), mordanting (mordant concentration) and dyeing (time, temperature, pH, MLR, and dye concentration) on the surface colour strength and colour fastness of the dyed silk fabric has been assessed and optimum values established for each.

Keywords: Ashoka bark, natural dyes, mordant, pre-mordanting, Saraca asoca, silk

Introduction

Natural dyes are mainly based on plant/animal origin and are renewable [Alam et al., 2020], biodegradable [Bechtold, 2009] and eco-friendly [Saxena and Raja, 2014]. They can produce uncommon and soothing shades [Samanta etc al., 2003]. However, the common drawbacks of natural dyes include poor reproducibility of shades [Gupta, 2019], poor to moderate colour fastness [Deepali et al, 2021] and non-availability of standard application methods [Gupta, 2019].

Derived from the Sanskrit word, and literally meaning 'sorrow less / without sorrow', Ashoka is the most ancient and legendary tree in India commonly also known as Indian Fir or Mast tree. The tree is considered sacred throughout the Indian subcontinent, especially in India, Nepal and Sri Lanka and plays an important role in the cultural traditions of India and the adjacent areas [Smitha and Thondaiman, 2016]. It is believed Shakyamuni Buddha was born under an Ashoka tree in the Lumbini gardens. Reference to the Ashoka tree can also be found in the Indian epic, Ramayana, which refers to 'Ashoka Vatika' or garden of Ashoka trees, where Hanuman first met Sita in Lanka. The tree is also associated with Kamadeva, the Hindu god of love, who included an Ashoka blossom, representing seductive hypnosis among the five flowers in his quiver. With the passing of the centuries, the yakshi under the Ashoka tree became a standard decorative element in Hindu sculpture and gradually got integrated as salabhanjika into the Indian temple architecture. Today also, Ashoka is highly valued for its evergreen handsome appearance, beautiful foliage, abundance of fragrant and attractive flowers, and is often found in royal palace compounds and temples surroundings throughout India. It is also cultivated in tropical gardens as an ornamental tree, is a popular park and garden plant and is much used in landscaping. The flower of Ashoka tree is the state flower of Indian state of Odisha [Smitha and Thondaiman, 2016].

The Ashoka tree belongs to Caesalpiniaceae, under legume family and is botanically known as Saraca asoca (Roxb.) [Common Trees]. It has at least 16 different names in Sanskrit, some such names being Karkeli, Anganapriya and Apashaka. The tree is sometimes incorrectly known as Saraca indica and is often confused with 'false Ashoka tree' or Polyalthia longofolia that is cultivated to resemble the growth pattern of erect pillar-like Mediterranean cypress trees. S. asoca can easily be distinguished by its smaller height, simple alternately arranged leaves deep green leaves, red flowers (initially orange in color) and fruits in the form of broad beans containing multiple seeds; while P. longifolia is taller having lance-shaped coppery red leaves with wavy border that turn into pale/deep green with time, apple green flowers and small spherical fruits that contain only one seed.

Saraca asoca is a small, erect evergreen tree, with deep green foliage (simple leaves) growing in dense clusters with fragrant, bright orange-yellow to red flowers. The tree is indigenous and is distributed in the evergreen rain forests of India. It is found throughout India especially in the Himalayas, Kerala, Bengal and the whole of Southern India [Divya et al., 2017]. Its original distribution was in the central areas of the Deccan plateau, as well as the middle section of the Western Ghats in the western coastal zone of the Indian subcontinent. It is also found in Burma, Ceylon (Sri Lanka) and Malaya [Common Trees and Smitha and Thondaiman, 2016].

Tree and foliage, Saraca asoca (Roxb.)

Tree and foliage, False Ashoka, Polyalthia lobgofolia

Apart from this, the tree also has many health benefits and has long been used in traditional Indian medicine as a key ingredient in various therapies and cures. Bark, flower, leaves, roots and seeds of the Ashoka are used as medicine. In Ayurvedic system of medicine, the bark of the tree is used for several medicinal purposes. The Ashoka bark is mainly used for treatment of menstrual disorders associated with excessive bleeding, other gynecological disorders, congestion, pain, dysmenorrhea, abdominal pain, and uterine spasms. It is used to cure fever, dyspepsia, dysentery, bleeding haemorrhoids, piles, sores, bronchitis, leucorrhea, menorrhagia, diabetes, biliousness and ulcers. It has estrogenic effects and can also be used as astringent, analgesic, uterine stimulant, anti-oxidant, and abortifacient. It is also known to have properties like uterine stimulant, anti-ulcer, anti-tumor, anti-cancer, anti-depressant, antibacterial, antioxidant and larvicidal activity [Divya et al., 2017, Baliarsingh et al., 2012, Smitha and Thondaiman, 2016 and Teunis and Labadie, 1986]. Dried flowers are used for treatment of syphilis, hemorrhagic, diabetes and dysentery [Smitha and Thondaiman, 2016]. It also helps to get rid of the toxins from the body and is effective in purifying the blood naturally and in preventing skin allergies [Smitha and Thondaiman, 2016]. Seeds are used to treat bone fracture and vesicle calculi [Smitha and Thondaiman, 2016].

Though very little is known about its use in colouring textiles, Saraca asoca plant is known to contains glyocoside, flavonoids, tannins and saponins [Pradhan et al., 2009], which are known to be a good source of colourants [Deveoglu and Karadag 2019]. Baliarsingh et al. [Baliarsingh et al., 2012] dyed silk with solvent extract of leaves and bark of Saraca asoca containing flavonoids and tannin moieties and showed the dyed fabric to exhibit excellent antimicrobial activity against the fungal strain, Aspergillus niger, while Gill and Singh [Gill and Singh., 2005] dyed wool with bark-extract of false Ashoka, Polyalthia longofolia using different natural mordants. Such studies are however are limited.

India is a storehouse of more than 500 species of plant resources, the leaves, fruits, seeds, flowers, barks and roots of which produce natural colours [Aggarwal 2020]. The art of natural dyeing is very old in India and is deeply embedded in its cultural traditions. However, with the passage of time, the art of natural dyeing lost its glory. This was mainly due to the introduction of the more cost effective and easier to produce and apply synthetic dyes on one hand and due to the unavailability of proper records owing to poor documentation of the natural dyeing process and methods by the Indian craftsman on the other. As a result, many of the known sources of colorants are long forgotten now and also many abundantly occurring plant species that could be a potential source of natural dyes/colourants remain unexplored. But now with the revival of natural dyeing and demand for natural dyed products on the rise, the market impact of natural dyes in a developing country like India is expected to increase in the coming years in both the industrial and small scale segments. Thus, with the objective of exploring an easily and abundantly available natural resource that has so far remain unexplored, a study on the use of the extract of lesser known natural dye like Ashoka bark (Saraca asoca) to colour silk has been undertaken and the process conditions for extraction, mordanting and dyeing have been optimized . So far, literature review has showed that studies in this direction are sporadic and extremely limited and it is expected that the present study will be useful and will provide another alternative and easily available dye source of natural origin to dye textiles.

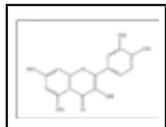
Methodology

Materials

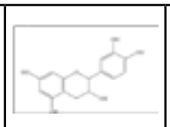
Undyed and plain weave 100% crepe silk fabric having 27 ends/dm and 43 picks/dm, with area density 62 g/m2 and fabric thickness 0.19 mm, obtained from Heritage, Kolkata was used.

Laboratory grade aluminium sulphate 16-hydrate [$(Al_2(SO_4)_3.16H_2O$] was used as a chemical mordant since its environmental toxicity is low compared to other metallic mordants. Sodium carbonate, glacial acetic acid and non-iconic surfactant obtained from Bharati Chemicals Pvt. Ltd. of E. Merck, India make were used.

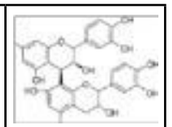
Dried barks of the Ashoka tree were obtained from a local seller at Maullick Bazaar, Kolkata. They were further sun-dried, crushed to a powdered form using a mechanical grinder. Ashoka bark contains querecetin [Baliarsingh et al., 2012], catechin [Baliarsingh et al., 2012] and epicatechin procyanidin B2 [Pradhan et al, 2009] as the colouring components. Three flavonoids, (−)-epicatechin, epiafzelechin-(4β→8)-epicatechin and procyanidin B2, together with β-sitosterol glucoside, have been isolated from dried bark [Jain and Sharma, 1967].



(a) Querecetin



(b) Catechin



(c) Epicatechin procyanidin B2

Methods

Degumming of silk fabric

Crepe silk fabric was degummed using non-iconic surfactant (5 gpl of) and sodium carbonate (2 gpl) 50^0C for 30 min using MLR 1:20 [Ammayappan et al, 2004]. The fabric was rinsed in running water and air dried in shade.

Optimization of aqueous extraction conditions

The coloring matter from the powdered Ashoka bark was extracted under varying process conditions of pH (2-11), MLR (1:10-1:50), temperature (ambient to boil) and time (15-90 min). While varying one process condition, the others were kept constant at pH - 7, MLR - 1:20, temperature - 90^0C and time - 30 min. The conditions of extraction were optimized on the basis of highest optical density of the extracted solution of the bark at wavelength of maximum absorption (λ_{max}).

Optimization of mordant concentration

Prior to dyeing, the degummed crepe silk fabric samples were pre-mordanted with various concentrations (10% to 50%) of the mordant (aluminium sulphate) at 60^0C for 30 mins using MLR 1:20. The concentration of the mordant was optimized on the basis of minimum strength loss in mordanted silk fabric and high colour yield (K/S) and good wash fastness of the mordanted silk subsequently dyed with Ashoka bark aqueous extract. The fabric samples were washed in running water after the treatment with the mordant and carried forward for dyeing.

Optimization of dyeing process conditions

The pre-mordanted crepe silk samples were dyed with aqueous extract of the bark (extracted at optimized conditions established earlier) under varying process conditions of varying pH (2-11), MLR (1:10-1:50), temperatures (ambient to 100°C) and time (15-90 min) min. While varying one process condition, the others were kept constant at pH - 5, MLR - 1:50, temperature - 90^0C and time - 75 min. The dyeing conditions was optimized on the basis of comparatively better surface colour strength and colour fastness properties.

Testing and Evaluation

Physical properties

Warp-way and weft-way breaking tenacity (cN/tex) of aluminium sulphate pre-mordanted crepe silk fabric samples were measured after conditioning at 65% ± 2% RH, at 27^0C ± 2^0C) temperature for 48 h by the IS: 6359–1917 method [ISI, BIS 1982] following raveled strip method with sample size 10 cm × 2.5 cm as per IS:1969:1968 procedure [ISI, BIS 1982] using an Instron (model-1445) CRT-Universal tensile tester with a traverse speed of 100mm/min and a pretension of 0.5 N. The final gauze length of the samples after raveling was 50 mm × 20 mm.

Colour related properties

Determination of wavelength of maximum absorbance for aqueous extract of Ashoka bark

The wavelength of maximum absorbance of the aqueous extract of Ashoka bark was identified by measuring the optical density of diluted 1% aqueous solution of Ashoka bark (1 gm of powdered Ashoka bark extracted in 100 ml of water at 90^0C for 30 min) at different wavelengths in the visible range (400-700 nm).

Estimation of surface colour strength & colour interaction parameters

Colour related parameters were evaluated using a Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software. Surface colour strength of dyed samples was measured by employing Kubelka Munk function or K/S values at λ_{max} [Tomer 2004]; the total colour difference (ΔE), lightness/darkness (ΔL*), redness/greenness (Δa*), blueness/yellowness (Δb*), change in chroma (ΔC*), and change in hue (ΔHab) of the dyed

samples were measured against undyed (mordanted) taken as standard sample following CIE-lab equations [Samanta et al., 2008], Colour Difference Index (CDI) as postulated earlier [Samanta et al., 2011] and Metamerism index (MI) as per Nimeroff and Yurow's equation [Shah and Gandhi 1990] were assessed.

Evaluation of colour fastness

Colour fastness to washing of the dyed samples was assessed as per IS: 764:1984 method [BIS 1982] following IS-2 (equivalent to ISO-II) wash fastness method (2 g/L non ionic detergent, 50 ±2 temperature, 45 min time and MRL 1:50) using Sasmira Launder-o-Meter and relevant standard grey scales (ISO-105-A02). Both dye & wet crocking fastness were assessed using a semi-automatic digital crockometer manufactured by MAG Solvics Pvt. Ltd., India following the IS: 766-1984 method [BIS 1982].

Results & Discussions

Determination of wavelength of maximum absorbance of the aqueous extract of Ashoka bark

Aqueous extract of Ashoka bark shows maximum optical density (4.30) at 400 nm. Thus, all further measurement of colour related parameters (K/S values, ΔE, ΔL*, Δa*, Δb*, ΔC*, ΔHab, MI, etc.) were determined at 400 nm.

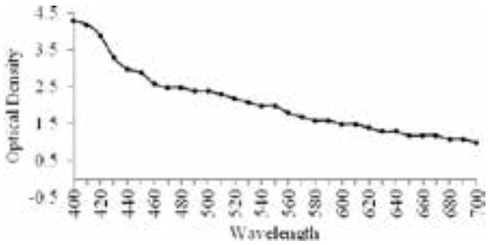
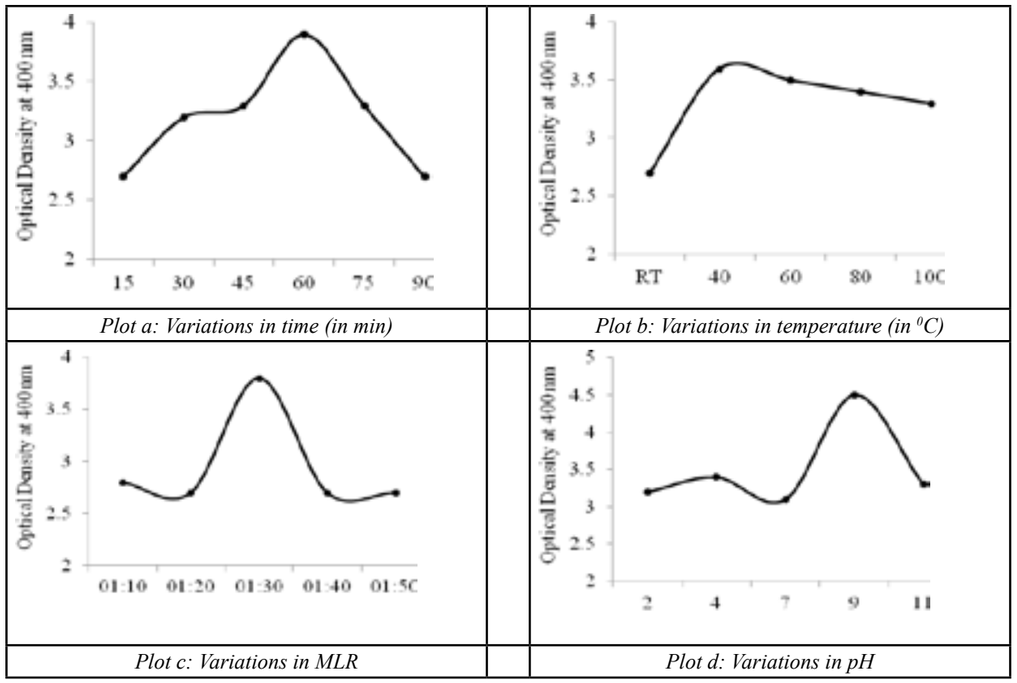


Figure 1: Colour yield (optical density) of 1% aqueous solution of Ashoka bark (extracted at 80^{0}C for 60 min using 1 gm of dye source in 100 ml of water) at different wavelengths in the visible zone (400-700 nm)

Optimization of the conditions of extraction

The extracted solution of Ashoka bark shows the highest optical density of 3.87 (at maximum absorbance wavelength of 400 nm) when extracted for 60 min (Plot a), of 3.63 when extracted at 40^{0}C (Plot b), of 3.39 when extracted using MLR - 1:30 (Plot c) and of 4.48 when extracted at pH - 9 (Plot d) and hence these values were considered optimum for the extraction process of colour from Ashoka bark.



Plot a: Variations in time (in min)

Plot b: Variations in temperature (in ^{0}C)

Plot c: Variations in MLR

Plot d: Variations in pH

Figure 2. Effect of variation in time, temperature, pH & MLR on aqueous extraction of Ashoka bark on the colour yield (optical density) of the extracted solution

Optimization of the mordant concentration

Degummed silk fabric samples were pre-mordanted using aluminium sulphate and the resultant changes in tenacity of the treated fabric assessed. The loss in tenacity is always higher in the warp direction as compared to the weft direction for the degummed silk fabric pre-mordanted with varying (10% to 50%) mordant concentrations calculated on the basis of the weight of the fabric (Table 1) due to more shrinkage in the warp direction as a result of exposure of the warp yarns to the more tension during weaving rendering it more vulnerable to strength loss than the relatively more relaxed weft yarns.

20% (owf) mordant concentration shows least loss in strength (5.09% loss) in the warp direction for the treated (mordanted) silk; while the loss in the weft direction is least (5.48% loss) for 10% mordant concentration.

Table 1. Tenacity (cN/Tex) of degummed silk pre-mordanted with varying concentration of the aluminum sulpahte

Mordant Concentration in % (calculated on the weight of fabric)	Tenacity (cN/tex)	
	Warp	Weft
NIL (degummed silk fabric)	13.36	11.31
10	11.67 (12.65)	10.69 (5.48)
15	12.28 (8.08)	10.36 (8.40)
20	12.68 (5.09)	9.98 (11.76)
25	12.39 (7.26)	8.51 (24.76)
35	12.23 (8.46)	7.98 (29.44)
50	12.06 (9.73)	6.84 (39.52)

*data in the parenthesis are the corresponding strength loss in percentage. The highlighted data correspond to the optimum values.

The effect of mordant (aluminium sulphate) concentration on the surface colour strength and wash fastness properties of pre-mordanted silk fabric dyed with aqueous extract of Ashoka bark was assessed and the data (Table 2) indicates that 20% (owf) of the mordant gives maximum surface colour depth (K/S of 12.2) when used to pre-mordant silk fabric which is then dyed with Ashoka bark extract. The wash fastness is poor and ranges from grade 1 to grade 2-3. With respect to the staining of the adjacent fabric (cotton and silk), the fastness ratings are better on cotton and higher for 20% (owf) mordant concentration.

Table 2: Surface colour strength (K/S) evaluated at λ_{max} (400 nm) and wash fastness (ISO-II) of silk fabric pre-mordanted with varying concentration of the mordant and then dyed with Ashoka bark extract

Mordant Concentration	K/S at 400 nm (λ_{max})	Wash Fastness (ISO-II)		
		LoD	ST	
			Cot	Sil
Degummed silk	10.1	1-2	3	3
10%	11.1	1-2	4	2
15%	11.2	2	3-4	3
20%	12.2	2	4	3
25%	12.0	2	3-4	2
45%	11.9	2-3	3-4	2
50%	11.1	2-3	4	2
LoD, Change in depth of shade; ST, Extent of staining; Cot, Cotton; Sil, Silk				

Though the strength loss for the mordanted and dyed silk fabric in the weft direction is comparatively much less for 10% (owf) mordant concentration, but the strength retention in the warp direction, the K/S values and wash fastness is better for 20% (owf) mordant. Thus, taking an overall view of test parameters of minimum strength loss, good surface colour strength and good wash fastness, 20% (owf) mordant is considered optimum.

Optimization of dyeing process variables with respect to surface colour strength and colour interaction parameters

The dyeing process variables have been optimized on the basis of uniform and good colour yield and maximum fastness properties of crepe silk fabric pre-mordanted with 20% (owf) aluminum sulphate (optimized concentration) and subsequently dyed aqueous extract (Table 3).

The K/S value of aluminum sulphate pre-mordanted silk fabric dyed with Ashoka bark extract increases with the increase in dye concentration from 25-50% (owf) and is noticeably highest at 100% owf concentration (K/S - 14.9). In general, increase in dye concentration increases the number of dye molecules available per surface unit area of the fibre surface and hence, higher dye concentration renders better dye adsorption and subsequent diffusion into the fibre.

K/S value increases with increase in the pH of the dye-bath till pH of 4 after which it decreases with further increase in pH. Maximum K/S is observed for pH 4 (K/S - 15.1) when maximum ionization and consequent dye adsorption takes place, this is

also conducive for dyeing silk which required an acidic medium.

K/S value of silk samples pre-mordanted with 20% (owf) aluminium sulphate and dyed with aqueous extract of Ashoka bark increases with increase in material to liquor ratio (MLR) till MLR 1:30 thereafter it decreases and gives maximum colour yield (K/S value of 14.6).

With other process variables fixed, K/S values increase with an increase in the dyeing time from 15 to 90 min and reaches equilibrium at 45 min after which it starts to decrease. Dyeing for 45 min gives highest surface colour yield on silk. This may possibly be due to the achievement of dyeing equilibrium at 45 min for this dye from Ashoka bark.

Increase in the dyeing temperature inevitably supplies more energy for the transportation of the dye molecule, thereby facilitating higher rate of dye sorption and diffusion. Higher temperature thus favors better dye penetration and hence there is maximum dye absorption (K/S - 13.1) of Ashoka bark extract by pre-mordanted silk at higher (100^0C) compared to lower temperatures.

Table 3. Surface colour strength, colour difference and related colour interaction parameters assessed at λ_{max} (400 nm) of silk fabric premordanted with 20% (owf) aluminum sulphate and then dyed with aqueous extract of Ashoka bark under varaible dyeing process conditions

Varying Parameters	**K/S at 400 nm (λmax)**	**ΔE**	**L***	**a***	**b***	**C***	**ΔHab**	**MI (LABD)**	**CDI**	**RCR (CDImax - CDImin)**
Degummed and alum pre-mordanted silk (Control)	0.3	2.4	-2.2	-0.0	1.8	1.6	-0.8	0.5	1.0	----
Variation in Dye Concentration										
25%	9.2	52.3	-44.4	19.6	22.6	26.4	-8.2	7.5	2.2	0.9
50%	12.8	58.8	-49.1	22.9	20.1	31.1	-8.1	6.4	2.4	
100%	14.9	60.9	-56.7	27.9	13.8	20.7	-8.3	5.3	3.1	
Variation in pH										
2	15.0	60.6	-51.5	21.1	24.1	30.9	-8.3	7.3	2.2	1.4
4	15.1	60.1	-53.3	19.9	19.7	26.7	-8.5	6.6	2.3	
7	11.7	53.8	-46.3	20.1	18.6	26.0	-8.5	6.3	2.8	
9	2.5	30.1	-25.4	10.4	13.5	16.2	-5.2	4.0	2.4	
11	2.0	23.9	-19.6	1.3	13.8	13.7	-1.0	2.8	1.4	
Variation in MLR										
1:10	8.7	83.7	-43.7	21.6	22.6	29.9	-8.6	7.2	2.1	0.5
1:20	10.9	55.6	-46.8	21.8	20.6	28.7	-8.9	7.1	2.4	
1:30	14.6	58.4	-48.3	22.9	23.6	31.6	-8.4	7.4	2.0	
1:40	12.1	55.5	-47.2	20.6	20.7	28.0	-8.6	6.7	2.5	
1:50	10.6	59.6	-49.8	22.2	24.1	31.7	-8.9	7.3	2.3	
Variation in Time										
15 min	9.8	59.2	-49.1	22.7	23.1	31.1	-8.9	7.4	2.3	0.8
30 min	13.9	58.3	-53.8	16.7	14.9	21.1	-7.7	7.7	2.7	
45 min	17.1	60.5	-50.7	23.2	24.6	32.6	-8.9	7.6	2.2	
60 min	16.3	60.7	-50.3	24.5	25.0	33.7	-9.2	7.8	2.1	
75 min	13.6	57.8	-50.5	20.1	19.8	26.9	-8.5	6.6	2.6	
90 min	12.2	55.9	-50.6	19.0	18.2	25.1	-8.2	6.2	2.9	
Variation in Temperature										
RT ^{0}C	5.9	44.4	-55.6	18.3	22.8	28.1	-7.6	6.4	1.9	0.5
40^0C	8.6	51.3	-51.8	22.5	23.9	31.1	-8.6	7.5	1.8	
60^0C	9.8	52.7	-52.0	23.0	24.6	32.5	-8.8	7.6	1.9	
80^0C	13.5	57.2	-50.7	21.2	21.1	28.7	-8.7	6.9	2.2	
100^0C	14.4	59.1	-49.1	16.3	14.1	20.1	-7.7	5.3	2.3	

Silk dyed under optimized conditions										
Dye concentration- 100% (owf), pH- 2, MLR- 1:50, temperature- 100^0C and dyeing time- 45 min	15.7	54.4	-45.2	20.8	24.1	30.7	-8.5	7.2	2.1	----

ΔE, total colour difference; L*, lightness/darkness; a*, greenness/redness difference; b*, blueness/yellowness; C*, change in chroma; ΔH, change in hue; MI, metamerism index; CDI, colour difference index

Higher range (> 50) of ΔE (total colour difference) is observed for the variation in nearly all process variables barring only three exceptions in case of variation in MLR and temperature indicating that all are major controlling parameters for achieving increased colour depth without much change in the predominating hue ((ΔHab) uniform dyeing of silk with Ashoka bark aqueous extract.

Changes in hue (ΔHab) for all the cases are found to be negative indicating that there is no major change in the predominating hue, except for some hypsochromic shift in the colour/tone. However, maximum negative ΔHab value is observed for variation in pH and time, indicating high sensitivity towards tonal variation imparted by the colour component in Ashoka bark towards these two dyeing process conditions.

Dispersion in CDI values (or RCR values i.e. CDImax – CDImin) are within the controllable limit of 2.0, but among the process variables it is relatively higher for variation in pH (CDI – 1.4). Variation in CDI values within each process variable is also insignificant. The order of increasing CDI values is therefore as follows – pH < dye concentration < time < MLR = temperature.

ΔL* values in case of all process variables are found to be negative indicating darker shades/tones of the dyed silk. Between the Δa* and Δb* for a particular process variable, the corresponding redness/greenness tone (Δa*) is higher for variation in dye concentration, in case of other process variables, the blueness/yellowness tone (Δb*) is generally predominant. This result also corroborates with the visual assessment of colours developed on aluminum sulphate pre-mordanted silk dyed with aqueous extract of Ashoka bark; most shades developed varying from ochre to dark rust.

The measured MI indicates that the metameric effect of silk fabric dyed with Ashoka bark under varying dyeing process conditions varies to a noticeable degree (MI - 7.7 to 2.8) from one condition to the other, indicating potent metameric effect in case of one varying parameter of dyeing against the other. The data are also widely dispersed within a particular dyeing process condition except in the case of variation in pH.

Thus, with respect to surface colour strength and colour related parameter, the optimized conditions of dyeing silk fabric dyed with aqueous extracts of Ashoka bark is identified as 100% (dye concentration), 4 (pH), 1:30 (MLR), 100^0C (temperature) and 45 min (dyeing time).

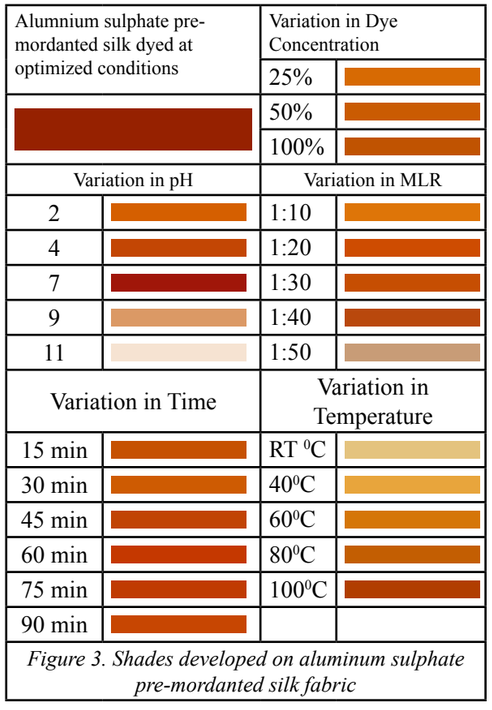


Figure 3. Shades developed on aluminum sulphate pre-mordanted silk fabric

Optimization of dyeing process variables with respect to colour fastness

Wash fastness with respect to loss in color depth (LoD) of the silk fabric dyed with aqueous extract of Ashoka bark is in general poor and ranges from 1 to 1-2 (Table 4). Wash fastness with respect to extent of staining of the adjacent non-mordanted silk or cotton fabric is very good (4 to 4-5) and in some sporadic

cases, fair. There is not much variation in the fastness values when a particular process parameter is varied. Also no variation is observed with respect to the wet and dry rub fastness of silk fabric dyed with Ashoka bark extract and, in general both the rubbing fastness (dry and wet) is poor (1-2 to 2). Variation within a particular process parameter also does not impact these values. Wet rubbing fastness is always higher than the corresponding dry rubbing fastness baring a few instances (when pH is varied).

Table 4: Colour fastness of silk dyed with Ashoka bark extract under different dyeing process conditions

Variables	Wash Fastness			Crocking Fastness	
	ISO-II			Dry	Wet
	LoD	ST			
		Cot	Sil		
Variation in Dye Concentration					
25	1-2	4	4-5	2	2
50	1-2	4-5	4-5	2-3	1-2
100	1-2	3	4	2	1-2
Variation in pH					
2	1	2-3	2-3	2-3	1-2
4	1-2	4	4-5	2	1-2
7	2	4	4	3	4
9	2-3	4-5	4-5	4	4
11	3	4-5	4	4	2
Variation in MLR					
1:10	1-2	4	4	2	2
1:20	1	4	4	2	2
1:30	1	4	4	2	1-2
1:40	1-2	4	4	2	1-2
1:50	1	3-4	4	2	1-2
Variation in Time					
15 min	1	4-5	4-5	2	1-2
30 min	1	4	4	1-2	2
45 min	1	4	4	2	1-2
60 min	1	4-5	4-5	2	1-2
75 min	1	4	4-5	2	1-2
Variation in Temperature					
RT	1	4-5	4-5	3	2-3
40^{0}C	1	4	4-5	3	2-3
60^{0}C	1	4-5	4	2	2
80^{0}C	1	4-5	4	2	1-2
100^{0}C	2	4	4	2	1-2
LoD, Loss in dept of shade; ST, Extent of staining; Cot, Cotton; Sil, silk					

Conclusion

The conditions for extraction of colour from Ashoka bark in the aqueous medium is optimized at 60 min (time), 40^{0}C (temperature), 1:30 (MLR) and 9 (pH). 20% (owf) mordant concentration gives optimum results both with respect to the minimum strength loss and maximum surface colour strength to the pre-mordanted silk that is subsequently dyed with Ashoka bark extract. MLR and pH have been identified as critical dyeing controlling parameters for Ashoka bark to be used as a natural dye for obtaining uniform shades as indicated by a wide dispersion within their respective CDI values. The optimized conditions of dyeing alum pre-mordanted silk fabric dyed with aqueous extracts of Ashoka bark with respect to surface colour strength and colour related parameter have been identified as 100% (dye concentration), 4 (pH), 1:30 (MLR), 100^{0}C (temperature) and 45 min (dyeing time).

Beautiful shades of ochre to rust have been obtained on silk dyed with aqueous extract of Ashoka bark thereby justifying its used as a potential source of natural colourant. Further studies can be undertaken on other fibres and with other mordants to provide enhanced data on dyeing of textiles with Ashoka bark. Attempts could be made to improve the fastness properties of this dye.

References

1. Alam SMM, Islam S and Akter S, Reviewing the Sustainability of Natural Dyes. Advance Research in Textile Engineering, 5(2), 1050 (2020)
2. Bechtold T and Mussak R, Natural colorants - Quinoid, naphthoquinoid and anthraquinoid dyes, Handbook of Natural Colorants, John Wiley & Sons Ltd., UK (2009)
3. Saxena S and Raja A, Natural Dyes: Sources, Chemistry, Application and Sustainability Issues. In: Muthu, S., Eds., Roadmap to Sustainable Textiles and Clothing. Textile Science and Clothing Technology, Springer, Singapore, 37-80 (2014)
4. Samanta A K, Singhee D and Sethia M, Application of single and mixture of selected natural dyes on cotton fabric: A scientific approach. Colourage, 50(10), 29-42 (2003)
5. Gupta V K, Fundamentals of natural dyes and its application on textile substrates in Chemistry and Technology of Natural and Synthetic Dyes and Pigments, ed. A. K. Samanta, N. S. Awwad and H. M. Algarni, InTech Open., UK (2020).
6. Singhee D, Sarawagi S and Dhanania Y, Standardization of dyeing of cotton with red sandalwood extract. Asian Textile Journal, 30(3), 40-48 (2021)
7. Smitha G R and Thondaiman V, Reproductive biology and breeding system of Saraca asoca (Roxb.) De Wilde: a vulnerable medicinal plant. Springerplus, 5(1), 2025 (2016)
8. Santapau H, Common Trees, National Book Trust, India, 1st ed. (1966), 8th reprint (2001)
9. Divya K R, Anjali A R and T Rajesh K, Phytochemical screening of Saraca asoca (Roxb.). De. Wild, Journal of Pharmacognosy and Phytochemistry, 6(3), 518-521 (2017)
10. Baliarsingh S, Panda A K, Jena J, Das T and Das N B, Exploring sustainable technique on natural dyes extraction from native plants for textile: Identification of colournats, colourimetric analysis of dyed yarns and their antimicrobial evaluation. Journal of Cleaner Production, 37(December), 257-264 (2012)
11. Teunis B and Labadie R P, An in vitro examination of bark extracts from Saraca asoca for oxytocic activity. Parmaceutical Biology, 24(1), 41-44 (1986)
12. Pradhan P, Joseph L, Gupta V, Chulet R, Arya H, Verma R and Bajpai A, Saraca asoca (Ashoka): A Review. Journal of Chemical and Pharmaceutical Research, 1(1), 62-71 ((2009)
13. Deveoglu O and Recep K, A Review on the Flavonoids – A Dye Source. International Journal of Advanced Engineering, Pure and Applied Sciences, 31(3), 188-200 (2019)
14. Gill Pankaj and Singh O P, Dyeing wool with vegetable dyes and natural mordant. Colourage, 52(3), 55-62 (2005)
15. Aggarwal S, Efforts and opportunities. Natural Resource Forum, 45(1) 63–86 (2021)
16. Jain S R and Sharma S N, Hypoglycaemic drugs of Indian indigenous origin. Planta Medica, 15(4), 439-442 (1967)
17. Ammayappan L, Ganesh K and Dwaraka K, A study on fastness properties of natural dye extracted from pseudo-stem of Musa paradisiacal on silk fabric. Man-made Textiles, 47(6), 218-220 (2004)
18. ISI (BIS), Handbook of Textile Testing. 1st ed., Bureau of Indian Standards, New Delhi, 24, 221, 255, 571, 536, 553 (1982)
19. ISI (BIS), Handbook of Textile Testing, SP:15 (Part-4 - 1988), Bureau of Indian Standards, New Delhi, 115, 157-158 (1989)
20. Praveen T, Singh J and Saroj, Utilization of banyan leaves for wool dyeing. Text Trends, 46(11), 25-27 (2004).
21. Samanta A K, Agarwal P and Datta S, Dyeing of jute with binary mixtures of jackfruit wood and other natural dyes - Study on colour performance and dye compatibility. Indian Journal of Fibre and Textile Research, 33(2), 171–80 (2008)
22. Samanta A K, Konar A, Chakraborti S and Datta S, Dyeing of jute fabric with tesu extract: Part-I: Effects of different mordants and dyeing process variables. Indian Journal of Fibre and Textile Research, 36 (1), 63–73 (2011).
23. Shah H S, and Gandhi R S, Instrumental colour measurements and computer aided colour matching for textiles, Mahajan Book Distributers, Ahmedabad, India, 76–116 (1990).
24. BIS (Bureau of Indian Standards), 1982. Handbook of Textile Testing. 1st ed, New Delhi: BIS (221, 255, 536, 553 & 573).

Dyeing and Antibacterial Finishing of Cotton with Azadirachta indica A. Juss (Neem Leaves)

Daksha Jalan, Yamini Dhanania and Deepali Singhee

ABSTRACT

In the present study harda-treated and aluminium sulphate pre-mordanted cotton fabric has been dyed with aqueous extract of neem leaves under different process conditions. Each process variable has been optimized with respect to the surface colour strength, other colour related parameters and fastness to light, wash and rubbing. The antimicrobial property of cotton dyed under optimized condition has been evaluated. pH and MLR of the dye-bath have been identified as the pre-dominating dyeing process variables and cotton dyed with neem leaves extract under optimized conditions shows better balance of properties with high surface colour strength (K/S), good light, wash and rubbing fastness. Harda-treated and aluminium sulphate pre-mordanted cotton shows significant antimicrobial property against gram positive (S. aureus) and gram negative (E. coli) bacteria till 72 hrs of inhibition.

Keywords: Anti-microbial property, Aluminium sulphate, Cotton, Colour fastness, Harda-treatment, Natural dye, Neem leaves

Introduction

Recent revival in the use of natural dyes for textile colouration has been mainly due to the stringent environmental standards imposed in response to the toxic and allergic reactions associated with synthetic dyes and their use. Natural dyes in comparison are non-allergic[1], non-toxic[2] and exhibits biodegradability[3] though they are associated with high cost[4] and poor reproducibility of shades[5]. The study has thus been undertaken to overcome the problem of standardization of shades through optimization of dyeing process variables, and to achieve reduction in the total cost of dyeing through use of forest by-products or waste like neem leaves.

Azadirachta indica known as neem or Indian Lilac and belonging to Meliaceae (mahogany family) is a fast-growing tree that is widely distributed throughout the Indian subcontinent, Pakistan, Sri Lanka, Nepal, Maldives, Bangladesh, the southern part of Iran. Although, the tree is evergreen and grows fast, it is also deciduous in nature and sheds its leaves at the end of the growing season though it is never completely bare. Thus, the leaves are available, every year, in abundance, as a forest waste.[6]

Neem is associated with a variety of medicinal and germicidal properties that is attributed to phytochemical compounds present in its leaves, bark, seeds and other parts of the plant.[7] Its seeds and leaves have been used not only to treat a number of human ailments due to its immunomodulatory, anti-inflammatory, anti-arthritic, anti-pyretic, hypoglycaemic, diuretic, anti-gastric ulcer, anti-inflammatory, anti-bacterial, anti-fungal, anti-malarial, anti-viral and anti-tumour properties[8], but also as a household pesticide.[9,10,11] At the same time its bark and leaves are a good source of tannins and are used in tanning and dyeing of a number of products.[12] Some studies have been conducted on the use of neem leaves extract as a adsorbent to remove colours and dyes from industrial effluents.[13,14] Neem extract is not completely water soluble and thus Bukhari et al[7] used methanol and acetone as co-solvents with water to increase solubility of the pigments from neem leaves extract and also identified the presence of quercetin (flavonoid) in the neem extract. Though neem is associated with such interesting dyeing related and functional properties, studies on its use for dyeing and functional finishing of textiles are limited and sporadic. Mohammad Zuber[15] isolated tannin from neem bark using microwave radiation and used it for dyeing chemical and bio mordanted silk fabric. Kumar et al studied the moth-efficacy of wool dyed with neem.[16] Nabawia et al highlighted the potential of neem leaves in the dyeing of wool in shades of yellow with good wash and fairly good light fastness that too without the use of any mordants.[17] Patel dyed polyurethane fibres with good fastness properties and antimicrobial resistivity using neem leaves extract.[18]

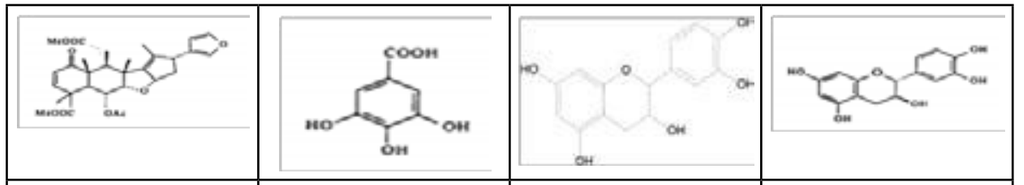
In the present study efforts have been made to optimize the process conditions for extraction and dyeing to dye cotton with aqueous extract of neem leaves. It has already been reported in literature that nimbolide present in the neem leaves exhibit antifungal and bactericidal activity against S. aureus and S. Coagulase[19] while mahmoodin possesses antibacterial activity against some human pathogenic bacteria.[20] Keeping this in mind, the antimicrobial properties of cotton dyed with neem leaves extract has also been explored to find suitable end use for neem dyed cotton fabric in the field of personal apparel, intimate and medical textiles.

Methodology

Bleached, undyed and plain weave 100% cotton fabric having 65 ends and 54 picks and 0.42 mm fabric thickness was used in the present work. Physical characteristics of the fabric used are mentioned in Table 1.

Table 1: Physical characteristics of the cotton fabric used

Breaking Tenacity (cN/tex) Warp-way	39.6 cN/tex
Weft-way	30.6 cN/tex
Breaking Elongation (%) Warp-way	12.4%
Weft-way	18.3%
Flexural Rigidity (mg.cm) Warp-way	4.3mg.cm
Weft-way	4.1mg.cm
Total Crease Recovery Angle (degree)	750
Whiteness Index (Hunter)	91.3
Whiteness Index (CIE)	110.6
Surface Reflectance (at 370nm)	1.11



Nimbin	Gallic acid	Catechin (* = S) Epicatechin (* = R)	Quercetin

Figure 1. Colouring components found in neem leaves

Commercial grade laboratory reagent (LR) grade aluminium sulphate 16-hydrate [(Al2(SO4)3.16H2O] as the chemical mordant; acetic acid and sodium carbonate for adjusting pH; hydrochloric acid (HCl) for desizing; sodium hydroxide and non-ionic surfactant for scouring obtained from E. Merck, India was used.

Methods

3.1 Desizing of cotton fabric

Bleached cotton fabric was desized using 25ml/l of 3.6% hydrochloric acid at 60^{0}C for 60 min using MLR 1:20. [24]

3.2 Scouring of cotton fabric

The desized cotton fabric was scoured using 3gpl NaOH and 3gpl of soap (non-ionic surfactant 100^{0}C for 2 hour using MLR 1:20 and maintaining pH at 12 using sodium carbonate.[25]

3.3 Treatment of scoured cotton fabric with myrobolan

10% owf of harda was soaked for overnight at room temperature using MLR 1:10. This paste was mixed with a known volume of water and heated for 30 min at 80^{0}C. The solution was filtered using a muslin cloth. The scoured cotton fabric was treated with this extracted myrobolan (harda) solution for 30 min at 80^{0}C and finally washed.

3.4 Optimization of mordant (aluminium sulphate) concentration

Scoured cotton fabric was mordanted with varying concentrations (10-50%) of aluminium sulphate at 60^{0}C for 30 min using MLR 1:20 and the

Neem leaves collected from ICAR-National Institute of Natural Fibre Engineering and Technology, Kolkata and harda obtained from a local supplier were dried in sun and powdered using a mechanical grinder.

The phytochemical constitutents present in neem are nimbidin, nimbin, nimbolide, Azadirachtin, gallic acid, epicatechin, catechin and margolonee.[21] The colouring component of neem leaves is mainly nimbin and polyphenols (gallic acid, catechin, epicatechin and quercetin).[7,22] Quercetin is a flavanol with a hydroxyl group in position 3 of the C ring while catechins or dihydroflavonols are 3 hydroxy derivatives of flavanones.[23]

mordant concentration was optimized on the basis of minimum strength loss, maximum colour yield and good colour fastness properties of the treated cotton fabric.

3.5 Pre-mordanting of myrobolan-treated cotton fabric with aluminium sulphate

The harda-treated cotton fabric was pre-mordanted with optimized concentration of aluminium sulphate at 60^{0}C for 30 min using MLR 1:20 followed by thorough rinsing in running water and air drying.

3.6 Aqueous extraction of dye from neem leaves

The colouring matter was extracted in water from the powdered sun-dried neem leaves at variable process conditions of pH (2-11), MLR (1:10-1:50), temperature (RT-100^{0}C) and time (15-90 min) followed by filtration of the extracted solution using a muslin cloth. Before extraction, the powdered leaves were soaked in water in the ratio of 5:100 for 30 minutes. The conditions of extraction was optimized with respect to the highest optical density of the extracted solution.

3.7 Dyeing of aluminium sulphate pre-mordanted cotton with neem leaves extract

The harda-treated and aluminium sulphate pre-mordanted cotton samples were dyed with the solution of neem leaves extracted at optimized conditions of pH, MLR, time and temperature. While varying a particular parameter, the other were kept constant at 7.5 (pH), 1:30 (MLR), 45 min (time) and 90^{0}C (temperature). After dyeing, the samples were rinsed thoroughly in running water and air dried in shade.

3.8 Testing and Evaluation

3.8.1 Measurement of breaking tenacity and breaking extension

Warp-way and weft-way breaking tenacities (cN/tex) and the breaking extension (%) of aluminium sulphate treated cotton fabric were measured following raveled strip method with sample size 10cm × 2.5cm as per IS:1969:1968 procedure [26] using an Instron (model-1445) CRT-Universal tensile tester with a traverse speed of 100mm/min and a pretension of 0.5 N. The final gauze length (sample size) of the fabric sample was 50mm × 20mm after raveling.

3.8.2 Measurements of crease recovery

The total crease recovery angle (CRA) of the cotton fabric (warp-way and weft-way) was assessed as per ASTM-D-1295-67 (1972)[27] method with 5 min loading and 5 min recovery time using Sasmira Crease Recovery Tester.

3.8.3 Measurement of fabric stiffness

The fabric stiffness of the cotton fabric as expressed by the bending length in both the warp and weft directions was measured as per IS: 6490-1972[26] standard test method (cantilever test) using Sasmira Fabric Stiffness Tester with specimen size of 200mm × 25mm.

3.8.4 Measurement of Whiteness Index

The degree of whiteness of the fabric sample was measured using a Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software and has been expressed on the basis of the Hunter equation of CIE[28] by:

Hunter whiteness: $100 - [(100-L^2) + a^2 + b^2]^{1/2}$

3.8.5 Maximum absorbance wavelength of the aqueous extract of the dye from neem leaves

The maximum absorbance wavelength of 1% aqueous extract (1gm of powdered neem leaves extracted in 100 ml of water at 90^{0}C for 30 min) of neem leaves (natural dye) was identified by evaluating the relative optical densities of the solution at different wavelengths (360-700 nm visible range) using Hitachi-U-2000 UV-VIS absorbance spectrophotometer.

3.8.6 Surface colour strength

K/S value is considered as an index for the surface dye uptake, i.e higher the K/S value, higher is the surface dye uptake of the sample.[29] Surface colour strength of the dyed cotton fabric was estimated in terms of K/S values (Kubelka Munk function)[30,31] by measuring surface reflectance of each of the dyed samples at the λ_{max} using a Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software. The surface reflectance values were converted to K/S using the following relationship:

$$K/S = \frac{(1-R_{\lambda max})^2}{2R\,\lambda_{max}}\ \alpha\ \ C_D$$

where, K = coefficient of absorption, S = coefficient of scattering, $R_{\lambda max}$ = surface reflectance value of sample at wavelength of where maximum absorption occurs for a particular constant, λ_{max} = maximum absorbance wavelength and CD = concentration of dye.

3.8.7 Colour interaction parameters

Total colour difference (ΔE), lightness/darkness (L*), redness/greenness (a*), blueness/yellowness (b*), change in chroma (ΔC), and change in hue ((ΔH), values were measured before and after dyeing to compare the shade depth and colour differences of each dyed sample against particular undyed (bleached / mordanted) standard sample using a Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software using the following CIE-lab equations.[29] General metamerism index (MI) was calculated employing the Nimeroff and Yurow's equation.[32]

3.8.8 Measurement of colour difference index

A newer colour interaction parameter called Colour Difference Index (CDI) postulated by Samanta et al[33] that indicates the combined effect of different known individual colour difference parameters between any two samples when dyed in varying shades under different conditions of dyeing has also been used in the present work to understand the combined effects of different dyeing variables on a single dyeing parameter and has been calculated as per the following equation.

$$\text{Colour Difference Index (CDI)} = \frac{\Delta E \times \Delta H}{\Delta C \times MI}$$

where ΔE is the total colour difference, ΔC is the change in chroma, ΔH is the change in hue and MI is the metamerism index.

3.8.9 Evaluation of colour fastness

Colour fastness on exposure to UV-light was determined as per IS: 2454-1984[34] method. Light fastness ratings from 1-8 were assigned to the dyed samples as per the details mentioned in Table 2.

Table 2: Inferences of the light fastness ratings

Blue Wool Standard No.	Description
1	Very poor fastness
2	Slight fastness
3	Moderate fastness
4	Fairly good fastness
5	Good fastness
6	Very good fastness
7	Excellent fastness
8	Outstanding fastness

Colour fastness to washing (ISO-II and ISO-III) of the dyed samples was determined as per the IS:764-1984 method[35] using a Launder o Meter and assessed in terms of loss of depth of colour and staining using Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software.

Colour fastness to rubbing (dry and wet) was assessed as per IS: 766-1984[26] method using a motorized semi-automatic digital crockmeter (MAG Solvics Pvt. Ltd., Coimbatore) and assessed in terms of loss of depth of colour and staining using Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software.

3.8.10 Assessment of antimicrobial properties

The qualitative test method AATCC-147-1993[36] was used to determine the antimicrobial activity of the cotton fabric dyed with neem leaves using agar diffusion test against both gram positive Staphylococcus aureus (S. aureus) and gram negative Escherichia coli (E. coli) bacterium. The bacterial culture was prepared by inoculating it at 37^{0}C for 24 hrs in an incubator. This was followed by sterilizing agar (nutrient) and the petriplates along with samples to be tested in an autoclave at 121^{0}C. The sterilize agar was then poured on the sterilized petriplates and allowed to solidify for 40 minutes in the laminar. After the solidification, the required amount (100 micro lt.) of bacterial culture, inoculated earlier for 24 hours, was poured on the agar base using micro pipette, spread evenly on the base using a sterilized spreader and left for 15 minutes for proper absorption by the agar base. Finally small pieces of the dyed cotton fabric were placed on the agar and the petriplates covered with a lid. The prepared plates were placed inside the incubator at 37^{0}C for about 24 hours. The zone of inhibition was calculated using the following equation:[37]

$$W = (T - D)/2$$

where: W = width of clear zone of inhibition in mm; T = total diameter of test specimen and clear zone in mm; D = diameter of the test specimen in mm.

Results and Discussions

4.1 Determination of the wavelength of maximum absorbance for aqueous extract of neem leaves

The optical densities of the aqueous extract of neem leaves at different wavelengths in the visible range (360 to 700 nm) is shown in Figure 2. Maximum optical density is observed at 370 nm and all further tests on colour parameters (K/S values, ΔE, L*, a*, b*, ΔC, ΔH, MI, etc.) were assessed at this

wavelength of maximum absorbance. The results also corroborates with the finding of an earlier study.[38]

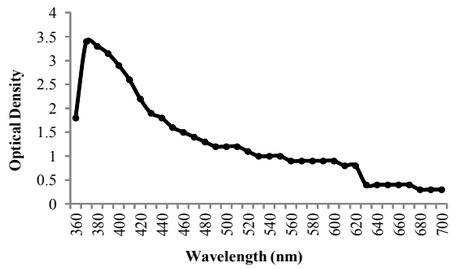


Figure 2: Colour yield (optical density) at different wavelengths in the visible zone (360 nm to 700 nm) of 1% aqueous solution of neem leaves extracted at 80^{0}C for 60 min using 1 gm of dye source in 100 ml of water.

4.2 Optimization of the mordant concentration

Scoured cotton fabric was pre-mordanted using varying concentrations (10-50%) of mordant (aluminium sulphate) at 60^{0}C for 30 min using MLR 1:20. The resultant changes in tenacity was assessed to find the optimum mordant concentration to be used for mordanting.

The corresponding data in Table 3 shows that treatment of cotton with aluminium sulphate results in some loss in the tensile strength of the treated cotton fabric in both the warp-way and weft-way directions. This loss in strength increases with increase in the mordant concentration till 20%. Mordant concentration of 10% gives maximum loss in strength. The percentage loss in all cases is much higher in the warp direction compared to the weft direction probably due to the higher shrinkage in the warp direction as a result of exposure of the warp yarns to more tension during weaving that makes it more vulnerable to strength loss as compared to the relatively more relaxed weft yarns. 25% mordant concentration does not have any impact on the tensile strength of the mordanted cotton fabric and the fabric retains 100% strength even after being treated with this mordant concentration. Beyond 25% of mordant concentration, the strength loss again starts increasing.

Table 3. Effect of mordant concentration on the mechanical property of scoured cotton pre-mordanted with varying concentration of aluminium sulphate

Mordant Concentration	Tenacity (cN/tex)	
	Warp-way	Weft-way
Scoured cotton fabric without any treatment (CONTROL)	6.8	6.3
10%*	7.6 (11.8)	6.7 (6.3)
15%*	7.3 (7.4)	6.7 (6.3)
20%*	7.0 (2.9)	6.4 (1.6)
25%*	6.8 (0)	6.3 (0)
35%*	6.9 (1.5)	6.4 (1.6)
50%*	7.4 (8.8)	6.8 (7.9)
*on the weight of fabric; data in the parenthesis are the corresponding strength loss values expressed in percentage		

The mordant concentration was also optimized on the basis of the highest surface colour strength and colour fastness properties of the cotton fabric pre-mordanted with varying concentrations of the mordant (10-50%) under fixed conditions of mordanting (at 60^{0}C for 30 min using MLR 1:20) and then dyed with aqueous extract of neem leaves under fixed conditions of dyeing (20% dye concentration, 1:20 MLR, 30 min time and 60^{0}C temperature).

Though use of mordant enhances the colour of the cotton fabric as can been seen from the data in Table 4. Scoured cotton dyed with neem extract without the use of mordant (control) gives a K/S of 3.4 compared to the much higher K/S values (ranging between 4.1 to 6.3) of dyed cotton that was mordanted with various concentrations of the mordant. As reflected by the data in Table 4, the surface colour strength in terms of the K/S values increases considerably on mordanting with 10% aluminium sulphate, but is decreases with further increase in the mordant concentration from 10-50% on the weight of the fabric. 10% mordant concentration though gives the highest K/S value, while the surface colour strength rendered by 15% & 20% mordant concentration is also appreciable. Complex formation between the dye and mordant generally depends on the particular stoichiometric ratio and in this case, 10-20% mordant concentration probably satisfies the required stoichiometric ratio and gives good results in terms of the surface colour strength (K/S) of the dyed sample.

Table 4. Effect of mordant concentration on surface colour strength and wash fastness of scoured cotton dyed with 20% (owf) of aqueous neem extract using MLR 1:20 for 30 min at 60^{0}C.

Mordant Concentration	K/S at 400 nm (λ_{max})	Wash Fastness (ISO-II)		
		LoD	ST	
			C	Cw
Harda treated and pre-mordanted cotton fabric dyed with aqueous neem extract (CONTROL)	10.1	1-2	3	3
10%*	6.3	4	4-5	4
15%*	6.0	4	4-5	4
20%*	6.0	4	4-5	4
25%*	5.2	4	4-5	4
35%*	5.0	4	4-5	4
50%*	4.1	4	4-5	4
*on the weight of fabric; LoD, loss in depth of shade; ST, extend of staining; C, cotton; Cw, cotswool				

Generally the use of aluminium sulphate as a mordant renders good wash fastness to cotton fabric dyed with neem leaves extract with respect to change in depth of colour and staining of the adjacent cotton or cotton-wool (cotswool) fabrics as indicated by the data in Table 4. There is no change in the fastness properties with variation in mordant concentration.

Comparing both the properties of mechanical strength and surface colour strength (K/S), though 10% mordant concentration gives highest surface colour strength, it reduces the strength of the treated cotton fabric by 11.8% in the warp-way and 6.3 in the weft-way direction. On the other hand, 25% mordant concentration though has no adverse effect on the strength of the treated fabric which remains same even after the treatment with the mordant, it renders comparatively much lower surface colour strength to cotton on dyeing. In this regard, 20% mordant concentration though gives slightly less surface colour strength, but is associated with much lower strength loss (only 2.9% in the warp-way and 1.6% in the weft-way directions). Thus, considering the high colour yield with negligible colour loss and reasonable/minimum cost due to use of lower concentration of the mordant, 20% aluminium sulphate is taken to the optimum mordant concentration.

4.3 Optimization of the conditions of extraction of colour from neem leaves

The conditions for extracting colour from dried and powdered neem leaves under variable process conditions of pH, MLR, time and temperature were optimized on the basis of comparative highest optical density of the extract for a particular process variable.

Table 5. Optical densities of the aqueous extract of neem leaves under variable process conditions evaluated at 370nm (λ_{max})

Parameters Varied		Optical Density (at 370nm)
pH	3	2.8
	4	2.8
	7	2.8
	9	2.8
	11	2.1
MLR	1:10	2.8
	1:20	2.8
	1:30	2.8
	1:40	2.8
	1:50	2.8
Temperature (°C)	RT	2.8
	40	2.7
	60	2.7
	80	2.8
	100	2.7
Time (min)	15	2.7
	30	2.7
	45	2.8
	60	2.7
	75	2.7
	90	2.7
	120	2.7
*highest values		

Effect of pH on extraction of colour from neem leaves as reflected by data in Table 5 indicates that the colour in neem leaves is not much sensitive to pH although higher alkaline pH of 11 reduces the colour yield. Since extraction under variable pH from 3-9 gives similar colour yield, pH 7 has been taken as optimum since it is well known that cotton being a cellulosic fibre is damaged under acidic conditions[39] and a neutral pH is easier to maintain during the extraction operation as it does not involve any additional chemicals unlike in case of the alkaline pH that needs a alkali. Further, extraction at pH 7 probably increased the solubility of the dye and its diffusion coefficient resulting in higher optical density. MLR does not cause any impact on the optical density of the neem leaves extract and for ease in operational procedure of laboratory dyeing and reduction in cost (lower MLR will need lower amount of energy

for heating thereby reducing costs), lower MLR of 1:20 has been found to be optimum. Maximum optical density is rendered to the solution when the extraction is carried out at room temperature or 80^{0}C. Since room temperature is expected to reduce energy cost in dyeing, it is considered optimum. Variation in time also does not have any appreciable effect on the extraction of colourant from neem leaves and 45 min records highest optical density. Prolonged extraction after 45 min probably leads to decomposition of the extracted molecules of neem in water hereby giving lower optical density.(7)

Optimization of different dyeing process variables
Colour difference and related colour interaction parameters
Effect of different dyeing process variables (dye concentration, pH, MLR, time and temperature) on dyeing related properties of harda-treated and aluminium sulphate pre-mordanted cotton fabric dyed with aqueous neem leaves extract have been studied to optimize the dyeing conditions for obtaining maximum and uniform colour yield and good fastness properties.

Increase in the concentration of the dye from 25-100% (calculated on the basis of weight of the dried dye source i.e. neem leaves) leads to increase in the surface colour strength or K/S value of the dyed cotton fabric. When the dye-bath concentration increases, there is more dye transfer to the fibre and thus higher apparent depth of colour occurs. Varying degrees of dye uptake in terms of K/S values with the variation in the pH of the dye-bath from 2 to 11 is evident from the data in Table 6. The K/S values decrease with increase in pH from 2 to 4. There is a noticeable increase thereafter and at pH 7 the cotton dyed with neem extract shows highest K/S. Alkaline pH is not suitable for dyeing cotton with this dye and harda-treated and aluminium sulphate pre-mordanted cotton shows very low K/S values when the dyeing is carried out under pH 9-11. Increase in pH from acidic to alkaline probably retards dye ionization and the resultant dye adsorption showing poor dye uptake. Keeping other variables constant, the K/S value of the dyed samples increases till 1:40 MLR. MLR 1:40 gives maximum colour yield in terms of the K/S values. Further increase in the MLR creates a dilution effect and reduces the dye uptake by slowing down the rate of strike of dye ions on the fibre surface. Thus K/S value decreases beyond MLR 1:40. Keeping all other variables fixed, an increase in the time of dyeing (15 min to 90 min) reduces the the K/S values till 45 min after which any further increase in the duration of dyeing increases the surface colour strength (K/S) of the dyed cotton fabric. The dyeing time reaches equilibrium after 75 min indicating that maximum absorption of the dye has taken place through formation of mordant-fibre-dye complex. Any further increase in dyeing-time beyond this equilibrium stage leads to desorption or breaking up of the dye-fibre-mordant complexes, thereby showing a decrease in the dye uptake (K/S). On increasing the dyeing temperature from room temperature to 100^{0}C, variable dye uptakes can be seen (Table 6) and highest surface colour strength (K/S) is observed when dyeing is carried out at 80^{0}C. This temperature provides more energy for the transportation of the dye molecules, thereby facilitating higher rate of dye strike, dye sorption and diffusion.

Table 6. Colour strength and related parameters of harda-treated and aluminium sulphate pre-mordanted cotton fabric dyed with aqueous extract of neem leaves under variable process conditions

Varying Parameters	**K/S at λ_{max}**	**ΔE**	**ΔC**	**ΔH**	**MI (LABD)**	**CDI**	**RCR (CDImax - CDImin)**
Harda-treated and aluminium sulphate pre-mordanted cotton (Control)	1.9	----	-0.1	0.1	2.2	0.6	----
Variation in dye concentration							
15	2.2	15.1	-5.4	10.6	3.9	2.6	1.6
30	3.0	17.7	-10.6	11.0	4.00	3.4	
45	2.2	14.2	-6.3	10.0	3.7	2.5	
Variation in pH							
2	3.1	3.1	1.2	-0.4	0.5	2.1	6.5
4	2.8	2.4	-1.6	-0.4	0.5	1.2	
7	4.7	10.0	4.6	-1.9	0.8	5.2	
9	2.6	12.2	4.2	-3.7	1.4	7.7	
11	2.4	8.2	3.1	-3.2	1.3	6.5	

Variation in MLR							
1:10	2.0	10.8	5.6	-3.5	1.3	5.2	3.0
1:20	2.1	13.3	7.4	-3.5	1.3	4.8	
1:30	2.2	16.1	9.2	-3.8	1.5	4.4	
1:40	3.0	17.1	10.6	-3.5	1.5	3.7	
1:50	2.3	12.3	5.0	-3.8	1.4	6.7	
Variation in time (in min)							
15 min	5.2	8.0	5.2	-1.9	3.9	0.7	1.0
30 min	5.0	7.9	4.7	-1.9	4.0	0.8	
45 min	4.2	6.5	3.3	-1.8	3.7	1.0	
60 min	6.2	8.4	3.9	-2.3	3.8	1.3	
75 min	6.3	9.4	4.1	-2.3	3.6	1.4	
90 min	6.3	10.7	4.5	-2.6	3.5	1.7	
Variation in temperature (^{0}C)							
RT ^{0}C	2.0	6.2	5.5	-1.4	2.5	0.6	0.6
40^{0}C	4.5	8.2	5.7	-2.1	3.2	0.9	
60^{0}C	2.0	9.0	7.3	-1.9	3.1	0.8	
80^{0}C	5.7	8.6	5.5	-1.9	3.4	0.9	
100^{0}C	2.9	8.8	4.3	-1.9	3.2	1.2	
ΔE, total colour difference; ΔH, change in hue; ΔC, change in chroma; MI, metamerism index; CDI, colour difference index							

The data for ΔE indicate the differences in the colour yield or surface colour strength for varying dyeing condition in each case, as compared to the standard un-dyed pre-mordanted scoured cotton fabric. ΔE value are found to vary significantly when pH and MLR is varied indicating that these two are the major controlling parameters responsible for uniform dyeing of cotton with neem leaves extract.

Changes in hue (ΔH) for all the cases are found to be negative indicating that there is no major change in predominating hue, except showing some hypsochromic shift in the colour / tone. The general metamerism index indicates the metameric effect on cotton fabric dyed with neem leaves for different process conditions of dyeing. In all the cases, the MI varies from 0.5 to 4.0 and the data are widely dispersed both within a particular process condition that is varied and from one condition to other, indicating potent metamerism from one varying condition to the other

CDI values are widely dispersed for pH and MLR among the dyeing process variables (dye concentration, pH, MLR, time and temperature) varied and have been identified as the most important and pre-dominating process variables. Dispersion of CDI (RCR) for variation in pH it is 1.2 to 7.7 and that for variation in MLR it is 3.7 to 6.7. The order of decreasing RCR values therefore appears to be as follows - pH < MLR < dye concentration < time < temperature. Therefore, for uniform dyeing of cotton with neem leaves extract, stringent control of pH and MLR of the dye-bath is imperative.

4.4.2 Colour fastness
The light fastness of harda-treated and aluminium sulphate pre-mordanted cotton dyed with neem leaves ranges from poor to moderate (1-3) in most cases as indicated in Table 7 except in case when the dye concentration is varied. Wash fastness with respect to change in depth of colour ranges from 1 to 4, indicating fair to good fastness. Staining of the adjacent cotton fabric is very good (3-4 to 4-5) and for cotswool it ranges from fair to good (3 to 3-4). In most cases, the corresponding value are better for ISO-II compared to ISO-III probably due to the additional use of alkali in ISO-III that causes leaching out of the fixed dye. Dry rubbing fastness is very good (3-4 to 4) and is much higher than the wet rubbing fastness for corresponding variables. Wet rubbing fastness is very poor and ranges between 2 to 1-2. Addition of water while performing the wet rub fastness test supposedly facilitates migration of surface dye molecules that are easily leached out resulting in reduction in the fastness properties.

For each variable, the fastness properties (Table 7) remains the best for the values that have been identified as optimum with respect to the surface colour strength and other colour parameters (Table 6).

Table 7. Colour fastness properties of aluminium sulphate (10%) pre-mordanted cotton fabric dyed with standardized aqueous extracted solution of neem leaves (NL) using variable conditions of dyeing

Variables	Light Fastness	Wash Fastness						Rubbing Fastness	
		ISO-II			ISO-III			Dry	Wet
		LoD	ST		LoD	ST			
			C	Cw		C	Cw		
Variation in dye concentration									
25%	4	3	4-5	4-5	2	3-4	3	3-4	2
50%	5	2-3	4-5	4-5	1-2	3-4	3	4	1-2
100%	5	2-3	4-5	4-5	1-2	3-4	3-4	4-5	2
Variation in pH									
2	3	2-3	4	4	2-3	3-4	3	4	1-2
4	3	1	4-5	4-5	1-2	3-4	3	4	2
7	3	2-3	4-5	4-5	2-3	4	3-4	4	2
9	2	1-2	4	4	2-3	4	3-4	4	2
11	1	3	3-4	4	2-3	4	3-4	4	2
Variation in MLR									
1:10	2	1	4	4	1	3-4	3	4	2
1:20	3	2	4-5	4	2-3	3	3-4	4	2
1:30	3	1	4-5	4	3-4	3	3-4	2-3	2
1:40	3	1	4-5	4	2	3-4	3-4	4	2
1:50	1	2	4-5	4	1	4	3	4	2
Variation in time (in min)									
15 min	1	1-2	4	4	1-2	3	3-4	3-4	2
30 min	1	1-2	4-5	4	1-2	3-4	3	4	2
45 min	2	1-2	4-5	4	1-2	3-4	3	4	2
60 min	2	2-3	4	4	1-2	3	3-4	4	2
75 min	2	2-3	4	4	1-2	3	3	4	2
90 min	2	2-3	4	4	1-2	3-4	3	4	2
Variation in temperature (^{0}C)									
RT	1	2	4-5	4	3-4	4	3	4	1-2
40^{0}C	2	2-3	4	4	2-3	3	3	3-4	2
60^{0}C	2	4	4	4	2	3	3-4	3-4	2
80^{0}C	3	2-3	4	4	2	3	3	4	2
100^{0}C	3	4	3-4	4	2	3-4	3-4	3-4	1-2
LoD, Loss in dept of shade; ST, extent of staining; C, cotton; Cw, cotswool									

In order to assess the surface colour strength and fastness properties of cotton dyed under optimized conditions, a harda-treated and aluminium pre-mordanted (using optimum mordant concentration) cotton sample was dyed with aqueous extract of neem leaves under optimized conditions. The corresponding results on surface colour strength and fastness properties are tabulated in Table 8. Cotton dyed under optimized conditions shows remarkable increase in the surface colour strength (K/S increases to 5.3 from 1.9) compared to the mordanted but undyed cotton. Though lighter shades (positive L* values) are obtained, it becomes darker when the mordanted cotton is dyed under optimized conditions as indicated by lower L* value (61.8) for the dyed sample compared to the higher value (82.5) for the harda-treated and aluminium sulphate pre-mordanted undyed cotton. On dyeing, the shades become more redder (positive a*) and yellower (positive b*). Improved light fastness (rating of 6), comparable wash fastness (ISO-II) and much improved rubbing fastness (both dry and wet) have been obtained when cotton is dyed under optimized conditions.

Table 8: Surface colour strength, colour interaction parameters and fastness properties of harda-treated and aluminium sulphate pre-mordanted cotton dyed at the optimized conditions with neem extract

Sample	K/S at λmax	ΔE	L*	a*	b*	LF	Wash Fastness (ISO II)			Rubbing Fastness	
							LoD	SC	SCw	Dry	Wet
Sample-1	1.9	--	82.5	-3.1	-27.5	--	--	--	--	--	--
Sample-2	5.3	21.4	61.8	2.3	26.7	6	3	4	4	4-5	3-4
Sample-1, Harda treated and aluminium sulphate pre-mordanted cotton; Sample-2, Harda treated and aluminium sulphate pre-mordanted cotton dyed with aqueous neem extract under optimized conditions of dyeing(100% dye concentration, 7 pH, 1:40 MLR, 80°C temperature and 60 min time); K/S, surface colour strength; L*, lightness/darkness; a*, greenness/redness; b*, blueness/yellowness; LoD, loss in depth of shade; SC, stating on adjacent cotton fabric; SCw, staining on adjacent cotswool fabric											

Antibacterial property

Keeping in mind the demand for multi-functional properties in textiles, the antimicrobial property of cotton dyed with neem leaves was also assessed and the relevant data are tabulated in Table 9. It is very evident from the data that harda-treated and aluminium sulphate pre-mordanted cotton dyed with aqueous extract of neem leaves exhibits remarkable antimicrobial function against both gram positive (S. aureus) and gram negative (E. coli) bacteria till 72 hrs of inhibition. This effect improves with time and is better for gram negative bacteria compared to gram positive bacteria.

Table 9. Zone of inhibition against E.coli and S.aureus for cotton treated with harda, aluminium sulphate and dyed with neem extract at the optimized conditions

	Zone of Inhibition (in mm)					
	24 hrs		48 hrs		72 hrs	
	E.coli	S.aureus	E.coli	S.aureus	E.coli	S.aureus
Scoured cotton fabric	0	0	0	0	0	0
Scoured cotton fabric treated with harda	0	0	0	0	0	0
Harda-treated cotton fabric pre-mordanted with aluminium sulphate at optimized conditions of mordanting	0	0	0	0	0	0
Harda treated and aluminium sulphate pre-mordanted cotton dyed with aqueous extract of neem leaves under optimized conditions of dyeing	1.5	1.0	2.0	1.5	2.5	2.0

Conclusion

20% mordant concentration is found to be optimum. The optimal conditions of aqueous extraction of colour from neem leaves has been established as 7 (pH), 1:20 (MLR), room temperature (temperature) and 45 min (time) with respect to the highest optical density at the wavelength of maximum absorption (370nm). Mordanting cotton with aluminium sulphate at pH 7 using MLR 1:20 at room temperature for 45 min gives best results with respect to minimal loss in strength and highest colour uptake. The optimized conditions for dyeing harda-treated and aluminium sulphate pre-mordanted cotton with aqueous neem extract has been established at 100% (dye concentration), 7 (pH), 1:40 (MLR), 80^{0}C (dyeing temperature) at 75 min (dyeing time). Cotton dyed under optimized conditions shows better balance of properties with high surface colour strength (K/S), good light, wash and rubbing fastness. For uniform dyeing of cotton with neem extract, special care should be taken for controlling of pH and MLR of the dye-bath apart. Harda-treated and aluminium pre-mordanted cotton exhibits antimicrobial property against gram positive (S. aureus) and gram negative (E. coli) bacteria till 72 hours of inhibition. Finding shows that the natural dye extracted from neem leaves have good potential for dyeing and imparting antimicrobial properties on textiles and its use in this respect can be exploited further.

References

1. O. E. Ismal, Y. Leyla, and O. Esen, Use of almond shell extracts plus biomordants as effective dye, Journal of Cleaner Production, 79, 61-67 (2014).
2. R. Paul, M. Jayesh, and, S. R. Naik, Natural dyes: Classification, extraction and fastness properties, Textile Dyer Printer, 29(22), 16-24 (1996).
3. K. H. Prabhu, and A. S. Bhute, Plants based natural dyes and mordants: A review. Journal of Natural Product Plant Resource, 2(6), 649-664 (2012).
4. P. Vankar, R. Shanker, and S. Wijaypala, Utilization of temple waste flower- Tagetus erecta for dyeing of cotton, wool and silk on industrial scale, Journal of Textile & Apparel, Technology and Management, 6(1), 1-15 (2009).

5. A. K. Samanta, and Priti Agarwal, Application of natural dyes on textiles, Indian Journal of Fibre Textile Research, 34(4), 384-399 (2009).
6. Sultan Ahmed, M. Parvaz, Rahul Johari, and M. Rafat, Studies on activated carbon derived from neem (azadirachta indica) bio-waste, and its application as supercapacitor electrode, Materials Research Express, 5, 045601 (2018).
7. H. Bukhari, M. Heba, and Q. Khadijah, Eco-friendly dyeing textiles with neem herb for multifunctional fabrics- Part 1: extraction standardization, International Journal of Technical Research and Applications, 2(2), 51-55 (2014).
8. Kausik Biswas, Ishita Chattopadhyay, Ranajit K. Banerjee, and Uday Bandyopadhyay, Biological activities and medicinal properties of neem (Azadirachta indica), Current Science, 82(11), 1336-1345 (2002).
9. N. S. Rawat, Neem plantation for better pesticides for reducing poverty and for protecting environment in India, Indian Journal of Environmental Protection, 14, 433-439 (1994).
10. R. R. Chattopadhyay, R. N. Chattopadhyay, and S. K. Maitra, Possible mechanism of anti-inflammatory activity of Azadirachta indica leaf extract, Indian Journal of Pharmacology, 25, 99-100 (1993).
11. A. M. Majumdar, A. S. Upadhyay, and A. M. Pradhan, Effect of Azadirachta Indica leaf extract on carbon tetra chloride induced hepatic damage in albino rats, Indian Journal of Pharmaceutical Sciences, 60, 363-370 (1998).
12. J. P. Mathur, Anjula Mehta, R. Karnawat, and C. S. Bhandari, Use of neem bark as wool colourant- Optimum conditions of wool dyeing, Indian Journal of Fibre & Textile Research, 28(3), 94-99 (2003).
13. G. G. Pandhare, N. Trivedi, Rajesh R. Pathrabe, and S. D. Dawande, Adsorption of colour from a stock solution using neem leaves powder as a low-cost adsorbent, International Journal of Enggineering Science and Emerging Technology, 5(2), 97-103 (2013).
14. Krishna G. Bhattacharyya, and Anurima Sarma, Adsorption characteristics of the dye brilliant green, Dyes and Pigments, 57(3), 211-222 (2003).
15. Mohammad Zuber, Shahid Adeel, Fazal-Ur Rehman, Fozia Anjum, Majid Muneer, Muhammad Abdullah, and Zia Khalid Mahmood, Influence of microwave radiation on dyeing of bio-mordanted silk fabric using neem bark (Azadirachta indica)-based tannin natural dye, Journal of Natural Fibres, doi.org/10.1080/15440478.2019.1576569 (2019).
16. Ajay Kumar, P. K Paeek, V. V. Kadam, and D. B. Shakyawar, Anti-moth efficacy of neem (Azadirachta indica A.Juss) on woolen fabric, Indian Journal of Traditional Knowledge, 15(2), 272-277 (2016).
17. Abdel-Zaher Nabawia A,, Manal T. H. Moselhey, and Osiris W. Guirguis, Eco-dyeing of wool fabrics with natural dye extracted from leaves of neem, Journal of Materials Science Research and Reviews, 4(2), 1-14 (2019).
18. B. H. Patel, Dyeing and antibicrobial finishing of polyurathane fibers with neem leaves extract, Manmade Textiles in India, 52(4), 112-116 (2009).
19. W. Rojanapo, S. Suwanno, R. Somaree, T. Glinsukon, and Y. Thebtaranonth, Mutagenic and antibacterial activity testing of nimbolide and nimbic acid, Journal of Science Thailand, 11, 177-188 (1985).
20. C. Devakumar, and SukhDev, Neem, eds Randhawa and Parmar, B. S., 2nd edn, 77-110 (1996).
21. T. Lakshmi, Vidya Krishnan, R. Rajendran, and N. Madhusudhanan, Azadirachta indica: A herbal panacea in dentistry – An update, Pharmacogn Review, 9(17), 41-44 (2015).
22. Marc Schumacher, Claudia Cerella, Simone Reuter, Mario Dicato, and Marc Diederich, Anti-inflammatory, pro-apoptotic, and anti-proliferative effects of a methanolic neem (Azadirachta indica) leaf extract are mediated via modulation of the nuclear factor-jB pathway, Genes & Nutrition, 6,149-160 (2011).
23. A. N. Panche, A. D. Diwan, and S. R. Chandra, Flavonoids: An overview, Journal of Nutrition Science, 5, e-47 (2016).
24. A. K. Samanta, D. Singhee, G. Basu, and K. K. Mahalanabis, Hydrogen peroxide and potassium per-oxo-disulphate combined room temperature bleaching of jute, cotton and jute-cotton union fabrics – An energy-efficient ecofriendly process, Indian Journal of Fiber & Research, 32, 221-231 (2007).
25. Vasanth D. Kumar, N. Boopathi, N. Karthick, and P. Ramesh, Aesthetic finishes for home textile materials, International Journal of Textile Science, 1(3), 5-9 (2012).
26. ISI (BIS) Handbook of Textile Testing, Bureau of Indian Standards, New Delhi, 24, 221, 255, 536, 553, 571, 573 (1982).
27. Annual Book of ASTM Standards (American Society for Testing & Materials Philadelphia, Pennsylvania, U.S.A), E23-02, 219 (1979).
28. Kerui Zhu, John Kanu Philip, Pierre Claver Irakoze, Kexue Zhu, Haifeng Qian, and Huiming Zhou, A method for evaluating hunter whiteness of mixed powders, Advanced Powder Technology, 20, 123-126 (2009).
29. Ashis Kumar Samanta, Priti Agarwal, and Siddhartha Dutta, Physio-chemical studies on the dyeing of jute and cotton fabrics using jackfruit wood extract: Part II- Dyeing kinetics and thermodynamic studies, Indian Journal of Textile & Fibre Research, 33(1), 66-72 (2008).
30. P. T. Tomer, and S. J. Singh, Utilisation of banyan leaves for wool dyeing, Textile Trends, 46 (11), 25-27 (2004).
31. S. D. Bhattacharya, and A. K. Shah, Metal ion effect on dyeing of wool fabric with catechu, Journal of Society of Dyers and Colourists, 116(1), 10 (2000).
32. H. S. Fairman, Metameric correction using parameric decomposition, Colour Research Application, 12, 261-265 (1987).
33. Ashis Kumar Samanta, Adwaita Konar, Sarmistha Chakraborti, and Siddhartha Dutta, Dyeing of jute fabric with tesu extract: Part I- Effects of the different mordants and dyeing process variables, Indian Journal of Fibre Textile Research, 36(1), 63-73 (2011).
34. ISI (BIS), Handbook of Textile Testing, SP: 15 (Part-4-1988), Bureau of Indian Standards, New Delhi, 115, 157-158 (1989).
35. S. S. Ahmed, A. Gogoi, R. Phukon, and N. Gogoi, Effect of natural dyes on the physical properties of silk, Indian Silk, 36(11), 17-19 (1997).
36. Deepti Gupta, and Ankur Laha, Antimicrobial activity of cotton fabric treated with Quercus infectoria extract, Indian Journal of Fibre and Textile Research, 32, 88-92 (2006).
37. S. Jayapriya, and G. Bagyalakshmi, Textile antimicrobial testing and standards, International Journal of Textile and Fashion Technology, 4(1), 1-10 (2013).
38. A. Nabawia, Abdel Zaher, T. H. Moselhey Manal, and W. Guirguis, Study the effect of different dyeing conditions of extracted natural dye from leaves of neem on silk fabrics, Journal of Chemical Metrology, 12(1), 34-58 (2018).
39. E. P. G. Gohl, and L.D. Vilensky, Textile Science- An explanation of fibre properties, Nazia Printers, Delhi, 2nd ed., reprint (2003).

A Study on Dyeing of Silk Fabric with Almond Shells (P. Amygdalus L.) Waste

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ABSTRACT

The present study is an endeavour towards making the process of dyeing with natural dyes cost effective through the use of easily available waste products like outer shells of the almond fruit. Degummed crepe silk fabric has been pre-mordanted with 25% (owf) aluminium sulphate and subsequently dyed under different conditions of time, temperature, pH, material to liquor ratio and dye concentration using aqueous extract of almond shells. The effect of variation in the extraction conditions and dyeing process variables on surface colour strength, other colour related parameters as well as fastness properties (light, washing and rubbing) have been optimized. Dyeing under acidic pH gives darker shades and the fatness properties of the dyed samples range from moderate to good. Temperature and pH have been found to be the most predominating dyeing process variables for dyeing silk with aqueous extract of almond shells as indicated by the widely dispersed CDI values of the dyed samples indicating that special care should be taken to control these parameters while dyeing silk and other protein fibers using aqueous extract of almond shell.

Keywords: Almond Shells, Aluminium Sulphate, Colour Dispersion Index (CDI), Dyeing Process Variables, Fastness Properties, Natural Dyes, Mordant, Crepe Silk Fabric.

Introduction

Natural dyes have been used since time memorable for dyeing textiles. Although natural dyes have the advantage of being a renewable source with bio-degradable nature and low environmental impact (Alam et al., 2007), they are still associated with problems of poor to moderate colour fastness (Samanta et al., 2007), absence of standardized procedure for application and extraction (Naz et al., 2011), non-reproducibility of shades (Samanta et al., 2007), pollution caused by use of metallic mordants (Mohammad et al., 2014), high energy consumption during extraction and exhaust dyeing (Bhargava, 2013) and high cost (Ozlenen et al., 2012). Natural dyes also have a significantly lower affinity for fibres that causes their lower dye-exhaustion from bath on to the fibre surface (Knizova, 2015). Moreover, the content of the colour component in most natural dyes is limited and large quantities of the dye source are needed to colour small quantities of the textile material (Knizova, 2015). For this, enormous amounts of the dye source has to be procured which may lead to over exploitation of natural resources, specifically from vegetable origin, to obtain dyes. It would also threaten some endangered species (Saxena et al., 2014). Various efforts have been undertaken all over the world to address these shortcomings of natural dyes and find suitable alternative sources in view of the tremendous environmental advantages that they offer. One such solution lies in the use of waste products.

The food and beverage industry releases considerable amounts of wastes which contain natural dyes. Such wastes could serve as sources for the extraction of natural dyes for textile-dyeing operations without causing any adverse impact on the environment and also avoid substitution food crops for cash crops. Some studies have already been undertaken to use such wastes effectively for dyeing wool with wastes, e.g. pressed berries, pressed grapes, distillation residues from strong liquor production, and wastes and peels from vegetable processing using iron mordants (Bechtold et al., 2006). Use of walnut shells in dyeing textiles has also been report by (Mirjalili et al., 2013). Such studies are however limited and scanty.

Although literature has reported the use of organic wastes such as almond shells for production of bio-energy and other valuable compounds (Chen et al., 2010), as a biosorbent for dye removal (Deniz, 2013a; Deniz, 2013b; Majib et al., 2012; Sundaram et al., 2012; Atmani et al., 2009), to absorb metals and organic substances in waste water treatment (Bechtold et al., 2006; Laufenberg et al., 2003; Pehlivan et al., 2008) and to dye wool in the presence of bio-mordants (Işmal et.al, 2012) or metal mordants (Işmal et al., 2014), there are very few and scanty reports in literature for its use for dyeing textiles.

Almond is consumed as a valuable food and its production generates millions of tons of residues (shells, hulls, pruning, leaves, skin and inedible kernel disposition), the bulk of which are the almond shells. The almond shell is the woody endocarp of the almond fruit (P. Amygdalus L.). High performance liquid chromatographic analysis has revealed the presence of quercetin, isorhamnetin, kaempferol-3-O-rutinoside, isorhamnetion-3-O-glucoside and morin as the major flavonoids in all extracts (Esfahlan et al., 2010).

The structure of the main colouring component found in the woody endocarp of the almond fruit, is given below in Figure 1.

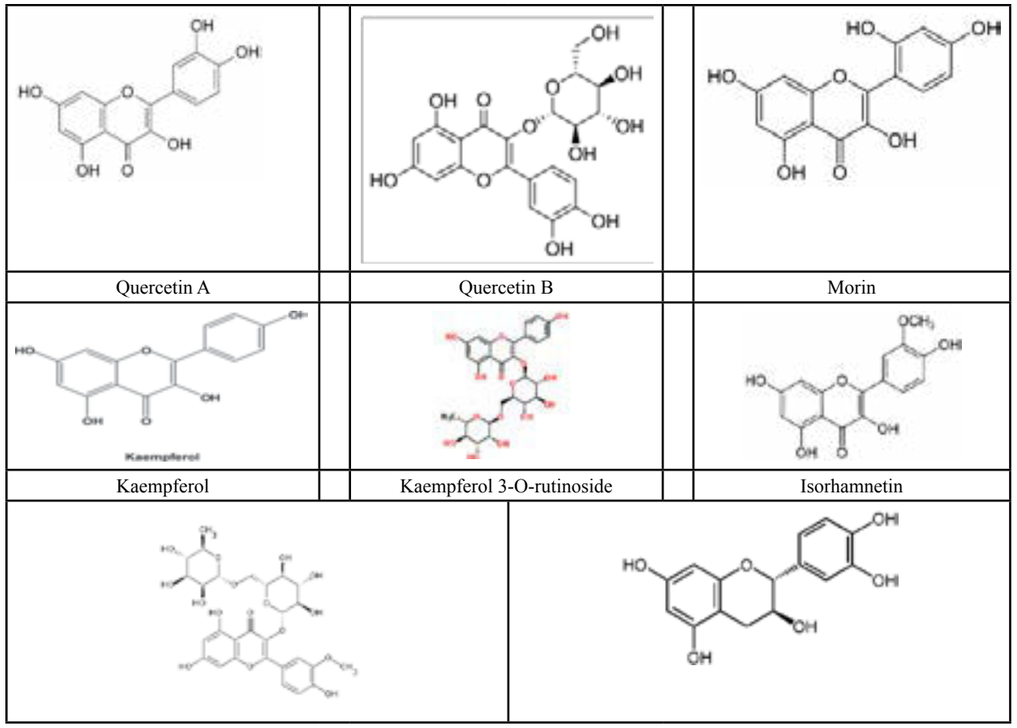


Figure 1: Structure of the colouring components found in almond shell extract.

Thus, in the present study, almond shells which are abundantly available as a waste product and free of cost have been used to dye crepe silk fabric with an objective to identify a new potential natural dye source which is not costly and can be used to dye silk at lower cost.

Methodology

Materials

Fabric: Crepe silk fabric having 9 tex silk yarns as warp and 6 tex silk yarn as weft with 32 ends/dm and 52 picks/dm and fabric area density of 66g/m2 obtained from M/s Handloom Cottage Pvt. Ltd., Kolkata (India) was used for the study.

Dyes and Chemicals: Almond shells obtained from Barabazar area in Kolkata (India) was ground to a powdered form after drying and was used for dyeing. Aluminium Sulphate 16-hydrate [$(Al_2(SO_4)_3.16H_2O$] was used as the mordant. Sodium carbonate, non-ionic detergent (Lux flakes), acetic acid were also used.

Degumming of crepe silk fabric: Bleached crepe silk fabric was degummed using 6 gpl non-ionic detergent and 2 gpl Na_2CO_3 at 90^0C for 90 min using material-to-liquor ratio (MLR) 1:20.

Pre-mordanting of degummed crepe silk: The degummed crepe silk fabric was was pre-mordanted, before dyeing, using optimized 25% (owf) aluminium sulphate (Al_2SO_4) at 60^0C for 30 min using MLR 1:20. The mordant concentration was optimized on the basis of highest colour yield, minimum loss in strength and good colour fastness of the silk fabric pre-mordanted with various concentrations of the mordant and subsequently dyed with aqueous extract of the dye.

Aqueous extraction of dye from almond shells: The colouring matter from powdered almond shells was extracted under aqueous conditions at variable temperature (RT-100^0C), time (15-90 min), material-to-liquor ratio (1:10-1:50) and pH (2-11) and each extraction condition was optimized on the basis of the highest optical density observed for 1% of the solution at the maximum wavelength. While studying a particular variable, the other variables were kept constant (temperature- 80^0C, time- 30 min, MLR- 1:20 and pH- 7). All further experiments were carried out at the optimized conditions.

Exhaust dyeing of pre-mordanted crepe silk: Pre-mordanted fabric samples were dyed using aqueous extract of the dye under variable parameters of dyeing dyeing temperature (RT-100^0C), time (15-90 min), material-to-liquor ratio (1:10-1:50), pH (2-11) and dye concentration (25-800%) and the optimum values for each condition established. While studying a particular variable, the other variables were kept constant (temperature-100^0C, time- 60 min, MLR- 1:50, pH- 11, dye concentratio- 100%). After dyeing, the samples were rinsed thoroughly under running water and air dried in shade.

Determination of maximum absorbance wavelength of the dye solution: The maximum absorbance wavelength of 1% aqueous extract of almond extract (natural dye) was identified by evaluating the relative optical densities of the solution (extracted at 90^0C for 30 min using 1 gm of dry source material of the dye in 100 ml of water) at different wavelengths (360-700 nm visible range) using Hitachi-U-2000 UV-VIS absorbance spectrophotometer.

The aqueous extract of onion peel showed maximum optical density at 370 nm (wavelength in the visible range) indicating that this natural dye shows maximum absorbance at this wavelength as shown in Figure 2. Thus, all further tests on colour parameters (K/S values, ΔE, ΔL, Δa, Δb, ΔC, ΔH, MI, etc.) were assessed at 370 nm.

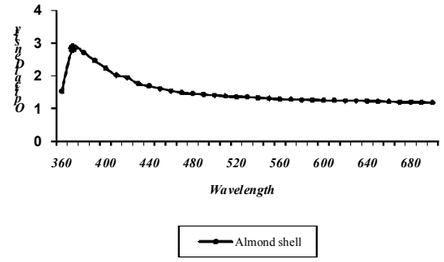


Figure 2: Colour yield (optical density) of 1% aqueous solution of almond shells extracted at 90^0C for 60 min using 1 gm of dye source in 100 ml of water at different wavelengths in the visible zone (360 nm to 700 nm)

Estimation of surface colour strength, dyeing uniformity and other related colour interaction parameters:

Surface colour strength of dyed crepe silk samples was estimated in terms of K/S values (Kubelka Munk function) (Bhattacharya et al., 2000; Tomer et al., 2004) by measuring surface reflectance of each of the dyed samples at the λmax (460 nm) using a Premier Colour Scan (model SC 5100A) reflectance spectrophotometer along with associated colourlab plus colour matching software.

Total colour difference (ΔE), lightness/darkness (ΔL*), redness/greenness (Δa*), blueness/yellowness (Δb*), change in chroma (ΔC*), and change in hue ((ΔHab), values were measured before and after dyeing to compare the shade depth and colour differences of each dyed sample against particular undyed (bleached / mordanted) standard sample using the following CIE-lab equations (Samanta et al., 2008).

General metamerism index (MI) was calculated employing the Nimeroff and Yurow's equation (Samanta et al., 2009).

Measurement of colour difference index

A newer colour interaction parameter called Colour Difference Index (CDI) postulated earlier (Samanta et al., 2011) that indicates the combined effect of different known individual colour difference parameters between any two samples when dyed in varying shade under different conditions of dyeing has also been used in the present work to understand the combined effects of different dyeing variables on a single dyeing parameter.

$$\text{Colour Difference Index (CDI)} = \frac{\Delta E \times \Delta H}{\Delta C \times MI}$$

Where ΔE I the total colour difference, ΔC is the change in chroma, ΔH is the change in hue and MI is the metamerism index.

For the application of same concentration of dye between two sets of dyeing under varying conditions, only the magnitudes of the respective ΔE, ΔC, ΔH and MI values (irrespective of their sign and direction) may be considered to calculate CDI values using the following empirical relationship:
Evaluation of colour fastness: Colour fastness to washing (ISO-II and ISO-III) of the dyed samples was determined as per the IS: 764-1984 methods (Ahmed et al., 1997). Colour fastness to rubbing (dry and wet) was assessed as per the IS: 766-1984 method (Ahmed et al., 1997).

Results and Discussion

Optimization of the conditions of aqueous extraction of colour: Before dyeing silk with extracted solution of almond shells, the conditions of extraction were optimized.

It can be seen from Table-1 that optical density of the solution of almond shells when extracted under aqueous conditions decreases with increase in the time, while it initially increases when temperature is raised to 80^{0}C after which it decreases.

Table 1: Optical densities (colour yield) at λmax (370 nm) of aqueous extracted solution of ground almond shells extracted at different time and temperature

Time (in minutes) (at 80^{0}C with 1:20 MLR and pH 7)						**Temperature (in ^{0}C)** (for 30 min with 1:20 MLR and pH 7)				
15	30	45	60	75	90	RT	40	60	80	100
3.33	3.11	3.06	3.04	2.99	2.58	2.53	2.54	2.55	2.58	2.55

Material-to-liquor ratio (MLR) (at 80^{0}C for 30 min in with pH 7)					**pH (adjusted by addition of CH_3COOH / Na_2CO_3)** (at 80^{0}C for 30 min with 1:20 MLR)				
1:10	1:20	1:30	1:40	1:50	2	4	7	9	11
2.67	2.63	3.16	2.60	2.50	1.55	2.41	2.51	2.49	2.93

The highlighted data correspond to the optimum values

There is an initial increase in the optical density of the extracted solution of almond shells with increase in material-to-liquor ratio, which reaches the maximum at an MLR of 1.30 ratio and thereafter it decreases as is evident form the data in Table-1. Colour extracted from almond shells increases with increase in the pH indicating that this natural dye is sensitive to acidic pH and can be easily extracted under alkaline conditions.

The conditions of extraction of colours from almond shells with respect to the highest optical density obtained at λ_{max} (370 nm) has thus been optimized at temperature- 80^{0}C, time-15 min, MLR-1:30 and pH-11.

Optimization of the mordant concentration: In the initial part of the present work, degummed silk fabric samples have been mordanted using aluminium sulphate and the resultant changes in tenacity and surface colour strength have been assessed. The corresponding data are shown in Table-2 and Table -3.

Effect of mordant concentration on mechanical properties
Treatment with higher concentrations of the mordant results in some loss of strength in both the warp and weft directions as can be seen from Table-3; in most cases this strength loss is always higher in the warp direction as compared to the weft direction. The higher loss of strength in the warp direction compared to that in the weft direction may be due to more shrinkage that arises in the warp direction as a result of exposure of the warp yarns to more tension during weaving rendering it more vulnerable to strength loss than the relatively more relaxed weft yarns.

Table 2: Effect of mordant (alum) on the mechanical properties of degummed crepe silk fabric pre-mordanted with alum at 60^{0}C for 30 min using MLR 1:20

Mordant Concentration	**Tenacity (cN/tex)**	
	Warp	**Weft**
NIL (degummed crepe silk sample)	13.22	10.09
10%	11.71	8.29
15%	11.73	8.14
20%	11.75	8.16
25%	11.89	8.47
35%	10.36	8.38
50%	10.20	6.64
The highlighted data correspond to the optimum values.		

Effect of mordant concentration on colour yield and colour fastness properties:

The surface colour strength (K/S value) of mordanted crepe silk when subsequently dyed with aqueous extract of almond shells initially increases with increase in the mordant concentration upto 25% (owf), after which it starts decreasing as is evident from Table-3. Mordanting with 25% (owf) aluminium sulphate gives maximum surface colour strength (K/S value).

Table 3: Effect of mordant (alum) on the dyeing efficiency of degummed crepe silk fabric pre-mordanted with alum and dyed with standardized aqueous extracted solution of almond shells at 100^{0}C for 1 hr using MLR 1:50

Mordant Concentration (Owf)	**Almond Shell**			
	K/S at λ_{max} (370m)	**Wash Fastness (ISO-II)**		
		LOD	**ST**	
			Cotton	**Silk**
NIL (degummed crepe silk sample)	1.778	2-3	4	4
10%	1.868	3	4	4
15%	1.998	2	4	4
20%	2.486	3	4	4
25%	2.977	4	4-5	4
35%	2.089	3	4-5	4
50%	2.083	3-4	4-5	4

LOD – Change in depth of shade, ST – Extent of staining
The highlighted data correspond to the optimum values

Table-3 also shows the effect of different concentrations of the mordant on the colour fastness of silk fabric dyed with the said dye. Generally the use of aluminium sulphate as a mordant gives good wash fastness with respect to staining of the adjacent fabrics (both silk and cotton). Although the colour fastness to change in the depth of colour ranges from poor to moderate, data in Table-3 indicates that the fastness improves with the increase in the concentration of the mordant and reaches maximum when 25% (owf) aluminium sulphate is used. This may be due to the initial availability of more mordant that leads to increased chances of complex formation between the said dye and mordant that is anchored to the fibre (silk). However, fastness with respect to the change in depth of shade decreases when more than 25% (owf) mordant is used. This indicates that after a certain (maximum) level of mordant concentration, the saturation value of complex formation between the dye and the mordant is reached, the excess un-fixed mordant although complexes with more dye molecules, it remains unfixed to the fibre and comes out easily during washing thereby causing wash fastness to be reduced.

Thus, keeping all the above parameters (tensile strength, K/S and wash fastness) in mind, 25% (owf) mordant concentration has been selected as the most efficient for mordanting and subsequently dyeing silk fabric with aqueous extract of almond shells.

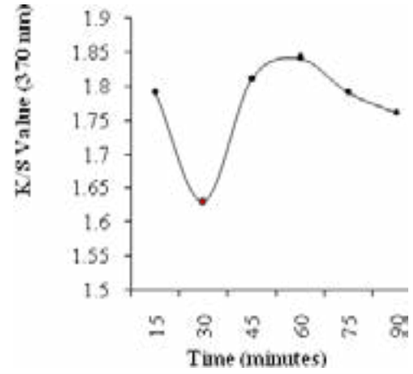
Optimization of different dyeing process variables
Effect of different dyeing process variables was optimized on the basis of uniform colour yield and maximum fastness properties obtained on crepe silk fabric samples pre-mordanted with 25% (owf) aluminium sulphate and subsequently dyed with aqueous extract (extracted at the optimized conditions) of almond shells.

Analysis of colour difference and related colour interaction parameters for application of onion peel extract under different dyeing conditions:
It was observed that keeping all other dyeing process variables fixed, with an increase in the dyeing time (15-90 min.) the K/S values shows a corresponding up and down trend (crests and troughs) till 30 min, thereafter which the K/S values increases up to 60 min. Beyond 60 min it again starts decreasing with further increase in the dyeing time. This may possibly be due to the achievement of dyeing equilibrium at 60 min for this dye, depending on the rate of dye diffusion. There may also be some desorption / breaking of dye-fibre-mordant complex beyond 60 min dyeing time, which leads to the dropping trend above 60 min of dyeing (Figure-3, Plot a).

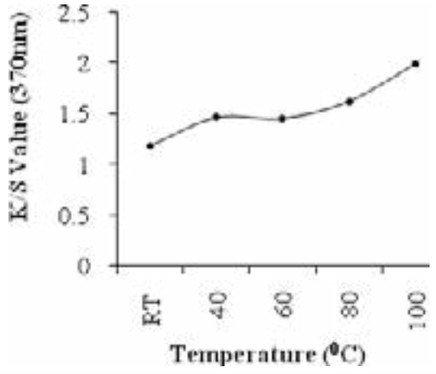
With increase in the dyeing temperature (from room temperature to 60^{0}C), there is a linear increase in the surface colour strength (K/S) (Table-4 or Figure-3, Plot b) that reaches maximum at 100^{0}C. Increase in the temperature of dyeing inevitably supplies more energy for the transportation of the dye molecule, thereby facilitating higher rate of dye sorption and diffusion into the fibre. Hence, it has been found that at 100^{0}C there is maximum absorption of colour from almond shells and hence it gives highest K/S values.

Keeping other variables constant, the K/S value of the dyed samples increases with an increase in pH from 2 to 11 (Table-4 / Figure-3, Plot c), pH 2 gives maximum colour yield in terms of the K/S

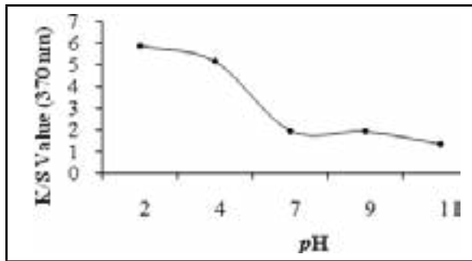
values and has been selected as the optimum In presence of acidic pH (2-4), the extract of almond shell presumably gives higher dye transportation, absorption and diffusion resulting in a higher colour yield (Figure-3, Plot c).



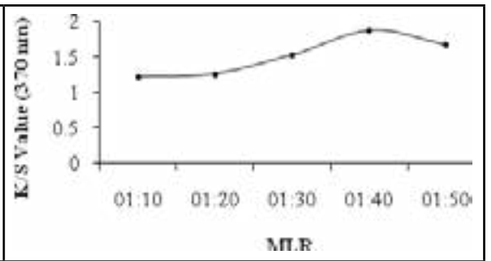
Plot a: Time (minutes)



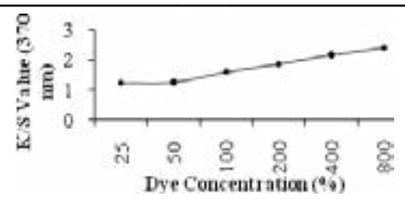
Plot b: Temperature (^{0}C)



Plot c: pH



Plot d: MLR



Plot e: Dye concentration

Figure 3: Effects of time temperature, pH, MLR, dye concentration in the dye-bath on colour yield of alum pre-mordanted crepe silk fabric using aqueous extract of almond shell.

Keeping other variables constant, the K/S value of the dyed samples increases with increase in the material-to-liquor ratio (MLR) from 1:10 to 1:50, after which it decreases. MLR (Figure-3, Plot d). MLR-1:40 gave maximum colour yield in terms of the K/S values and has been selected as the optimum.

There is a sharp linear increase in K/S value with the increase in dye concentration from 25-800% (on the basis of weight % of dried solid almond shell). This increase is noticeably higher at 800% concentration. Hence, 800% dye concentration is taken as the optimum value.

Table 4: Colour strength and related parameters of alum (25%) pre-mordanted crepe silk fabric dyed with standardized aqueous extracted solution of almond shell(AS) using variable conditions of dyeing

Varying Parameters	**K/S at λ_{max}**	**ΔE**	**ΔL**	**Δa**	**Δb**	**ΔC**	**ΔH**	**MI (LABD)**	**CDI**	**RCR (CDImax - CDImin)**
Degummed and alum pre-mordanted silk (CONTROL)	0.97	6.33	-5.47	2.86	1.39	1.31	-2.89	1.00	--	--
Variation in TIME (in min) [dyed at 100^{0}C, pH- 11, MLR-1:50 using 100% dye (on the basis of weight % of dried solid AS)]										
15 min	1.79	11.77	-11.75	0.53	-0.44	-0.36	-0.58	0.34	55.77	49.44
30 min	1.63	11.60	-11.54	0.16	-1.18	-1.15	-0.29	0.26	11.25	
45 min	1.81	12.87	-12.83	0.32	-0.93	-0.88	-0.43	0.24	26.20	
60 min	1.84	13.21	-13.17	0.26	-0.97	-0.93	-0.37	0.30	17.52	
75 min	1.79	11.79	-11.79	0.20	0.16	0.18	-0.18	0.51	23.12	
90 min	1.76	10.34	-10.29	0.44	0.90	0.95	-0.32	0.55	6.33	
Variation in Temperature (^{0}C) [dyed at pH- 11, MLR-1:50 using 100% dye (on the basis of weight % of dried solid AS) for 60 min]										
RT ^{0}C	1.18	4.47	-4.45	0.43	-0.25	-0.19	-0.46	0.16	67.64	66.37
40^{0}C	1.46	6.76	-6.67	0.41	1.01	1.05	-0.28	0.57	3.16	
60^{0}C	1.45	6.77	-6.69	0.21	0.99	1.02	-0.10	0.52	1.27	
80^{0}C	1.62	9.34	-9.31	0.27	0.75	0.78	-0.18	0.60	3.59	
100^{0}C	1.99	12.14	-12.08	0.44	1.14	1.18	-0.30	0.70	4.41	
Variation in pH [dyed at 100^{0}C, MLR-1:50 using 100% dye (on the basis of weight % of dried solid AS) for 60 min]										
2	5.89	28.32	-28.06	1.70	3.41	3.63	-1.16	0.94	9.63	109.01
4	5.16	28.40	-28.38	0.91	0.44	0.57	-0.84	0.38	110.14	
7	1.95	14.11	-14.05	0.74	-0.99	-0.87	-0.88	0.14	101.94	
9	1.91	10.82	-10.58	0.75	2.09	2.17	-0.48	0.76	3.15	
11	1.33	5.43	-5.28	0.02	1.23	1.22	-0.15	0.59	1.13	
Variation in MLR [dyed at 100^{0}C, pH- 11 using 100% dye (on the basis of weight % of dried solid AS) for 60 min]										
1:10	1.22	4.10	-1.93	0.36	3.60	3.62	-0.02	0.79	0.03	5.53
1:20	1.26	4.47	-3.45	0.45	2.80	2.83	-0.14	0.75	0.29	
1:30	1.53	7.41	-7.23	0.59	1.54	1.60	-0.40	0.57	3.25	
1:40	1.88	8.85	-8.26	0.95	3.01	3.10	-0.55	0.84	1.87	
1:50	1.67	9.16	-9.07	0.52	1.13	1.18	-0.38	0.53	5.56	
Variation in DYE CONCENTRATION [(on the basis of weight % of dried solid AS) dyed at 100^{0}C, pH- 11, MLR-1:50 for 60 min]										
25%	1.22	4.42	-2.34	0.48	3.72	3.75	-0.07	0.84	0.09	4.02
50%	1.24	4.38	-3.97	0.01	1.86	1.85	-0.17	0.53	0.76	
100%	1.58	8.21	-8.11	0.49	1.17	1.22	-0.34	0.56	4.08	
200%	1.84	8.42	-5.28	0.75	6.51	6.56	-0.05	1.43	0.04	
400%	2.16	10.24	-4.96	1.07	8.89	8.96	-0.09	1.77	0.06	
800%	2.39	11.94	-6.41	2.22	9.82	10.04	-0.85	1.92	0.53	
ΔE – total colour difference, ΔL – lightness/darkness, Δa – greenness/redness difference, Δb – blueness/yellowness, ΔH – change in hue, ΔC – change in chroma, MI – metamerism index, CDI – colour difference index										
The highlighted data correspond to the optimum values										

Table-4 also shows the effect of the different process variables on other colour interaction parameter, including total colour difference (ΔE), changes in chroma (ΔC), general metamerism index (MI) and the colour different index (CDI) values. It is interesting to note that among the dyeing conditions (time, temperature, MLR, pH and dye concentration) varied, the most important and pre-dominanting variable has been identified as dyeing time, temperature and pH of the dye-bath as indicated by the wide dispersion of CDI values. The order of increasing CDI values therefore appears to be as follows:

Dye concentration < MLR < Time < Temperature < PH

The corresponding data on ΔE, ΔL, Δa, and Δb for crepe silk fabric dyed with aqueous extract of almond shells indicate are also listed in Table-4. Higher range of ΔE value is observed for the variation in pH, indicating that it is the major controlling-parameter that is responsible for uniform dyeing. The data on ΔL, Δa, and Δb indicate further implication of the colour difference in terms of lightness/darkness, redness/greenness and blueness/yellowness respectively and analysis of these through individual colour difference parameters for dye from almond shells show that variations are more for the lightness/darkness scale rather than redness/greenness scale followed by blueness/yellowness. This indicates that the major colour component (of the predominating hue) of the natural dye from almond shell has been subjected to more varied absorption / anchoring and thus gives higher ΔL values.

Changes in hue (ΔH) for all the cases are found to be negative (Table-4), indicating that there is no major change in the predominating hue, except for some hypsochromic shift in the colour/tone. However, the maximum negative ΔH value (> -0.50) is again observed in case of the variation in time (15 min), MLR (1:40), pH (4 & 7) and dye concentration (800%), which further indicates the colour yield for the natural dye obtained from almond shells is sensitive to these four dyeing process variables.

Therefore, for uniform dyeing of silk and other protein fibres with aqueous extract of almond shells extract, special care should be taken to control temperature, pH of the dye-bath and dye concentration.

Analysis of colour fastness (washing, rubbing, light and perspiration) for application of almond shell extract under different dyeing conditions:

The wash fastness data of silk dyed with almond shell is tabulated in Table-5. It can be observed that in all cases, the wash fastness with respect to change in colour depth ranges from moderate to good (2-3 to 4-5) for both ISO-II & ISO-III methods. Again in most cases, the corresponding fastness ratings for each variable are either same or slightly better in case of ISO-III as compared to ISO-II, with the exception when pH and temperature is varied. Irrespective of the different dyeing process variables studied, the wash fastness with respect to extent of staining of the adjacent non-mordanted cotton and silk fabric is very good (4 and 4-5) in both cases of ISO-II and ISO-III as revealed by the data in Table-5 although this fastness is slightly better in case of ISO-II where no alkali has been used.

The wet and dry rub fastness of dyed silk samples with almond shell extract has been found to be good (4 to 4-5). Also there is not much variation in the wet and dry rub fastness of dyed silk samples with respect to the variation in the conditions of the dyeing parameters with the exception in case of pH variation. It is also interesting to note that the wet rubbing fastness is either same or marginally lower in few cases (when temperature and MLR is varied) compared to the corresponding dry rubbing fastness; in few exceptional cases the former is higher.

It is interesting to note from Table-5 that the best results with respect to colour fastness (wash and rubbing) matches with that of the optimized dyeing process parameters i.e dyeing dyeing temperature- 100^{0}C, time- 60 min, MLR- 1:40, pH- 2 and dye concentration- 800% (owf).

Table 5: Colour fastness properties of alum (25%) pre-mordanted crepe silk fabric dyed with standardized aqueous extracted solution of almond shell using variable conditions of dyeing

Variables	**Wash Fastness**						**Crocking Fastness**	
	ISO-II			**ISO-III**			**Dry**	**Wet**
	LOD	**ST**		**LOD**	**ST**			
		Cot	**Sil**		**Cot**	**Sil**		
Variation in TIME (in min) [dyed at 100^{0}C, pH- 11, MLR-1:50 using 100% dye (on the basis of weight % of dried solid AS)]								
15 min	3	4-5	4-5	4	4-5	4-5	4	4-5
30 min	4	4-5	4-5	4-5	4-5	4	4-5	4-5
45 min	3	4-5	4	3-4	4-5	4-5	4-5	4-5
60 min	4-5	4-5	4-5	4	4-5	4-5	4-5	4-5
75 min	4	4-5	4-5	4	4-5	4	4-5	4-5
90 min	4	4-5	4-5	4	4-5	4-5	4-5	4-5

Variation in Temperature (^{0}C) [dyed at pH- 11, MLR-1:50 using 100% dye (on the basis of weight % of dried solid AS) for 60 min]								
RT	3-4	4-5	4-5	2-3	4-5	4-5	4	4-5
40^{0}C	3	4-5	4	3	4-5	4	4-5	4-5
60^{0}C	3	4-5	4-5	3	4-5	4	4-5	4-5
80^{0}C	3-4	4-5	4-5	4	4	4-5	4-5	4
100^{0}C	3-4	4-5	4-5	4	4	4-5	4-5	4-5
Variation in pH [dyed at 100^{0}C, MLR-1:50 using 100% dye (on the basis of weight % of dried solid AS) for 60 min]								
2	4	4-5	4-5	3-4	4-5	4	4-5	4-5
4	4	4	4-5	3-4	4-5	4	4-5	4-5
7	4	4-5	4-5	3-4	4	4	4-5	4-5
9	3-4	4	4-5	4	4-5	4	4-5	3-4
11	2-3	4	4-5	3	4-5	4	4-5	4-5
Variation in MLR [dyed at 100^{0}C, pH- 11 using 100% dye (on the basis of weight % of dried solid AS) for 60 min]								
1:10	4-5	4	4-5	4	4-5	4-5	4-5	4-5
1:20	4-5	4-5	4-5	3-4	4-5	4-5	4-5	4-5
1:30	4-5	4	4-5	3-4	4	4-5	4-5	4-5
1:40	4-5	4-5	4-5	3-4	4-5	4-5	4-5	4-5
1:50	3-4	4	4-5	3-4	4-5	4-5	4	4-5
Variation in DYE CONCENTRATION [(on the basis of weight % of dried solid AS) dyed at 100^{0}C, pH- 11, MLR-1:50 for 60 min]								
25%	3	4	4-5	3	4-5	4-5	4-5	4-5
50%	3-4	4	4-5	4-5	4-5	4-5	4-5	4-5
100%	3-4	4	4-5	4	4-5	4-5	4-5	4-5
200%	2-3	4	4-5	2-3	4-5	4	4-5	4-5
400%	4-5	4	4	3	4-5	4	4-5	4-5
800%	4-5	4-5	4	4	4-5	4	4-5	4
LT – Light fastness, LOD – Loss in dept of shade, ST – Extent of staining, Cot – Cotton, Sil – Silk								

Conclusions

From the present study the following may thus be concluded:

The shades developed on crepe silk fabric samples dyed with almond shell extract ranges from various shades of light beige to dark beige. Darker shades are produced when dyeing is carried out under acidic pH.

The optimized extraction condition with respect to the highest optical density at maximum wavelength for onion peel has been established at 80^{0}C (extraction temperature), 15 minutes (extraction time), 1:30 (MLR) and 11 (pH).

25% (owf) mordant concentration has been optimized in terms of the minimum strength loss and maximum surface colour strength of the treated and subsequently dyed fabric.

The optimized dyeing condition with respect to with respect to surface colour strength and fastness properties for dyeing silk crepe fabric using almond shell extract was established at 100^{0}C (dyeing temperature), 60 min (dyeing time), 1:40 (MLR) and 2 (pH) and 800% (dye concentration).

For uniform dyeing with onion peel extract of silk and other protein fibres, special care should be taken for controlling of pH and temperature of the dye-bath apart from the dye concentration as indicated by a wide dispersion of CDI values.

References

1. Ahmed, S.S., Gogoi, A., Phukon, R. and Gogoi, N. (1997). Effect of natural dyes on the physical properties of silk. Indian Silk, 36(11), pp. 17-19
2. Alam, M.M., Rahman, M.L. and Haque, M.Z. (2007). Extrication of henna leaf dye and its dyeing effect on textile fibre. Bangaldesh Journal of Scientific and Industrial Res, 42(2), pp. 217-222.
3. Atmani, F., Bensmaili A. and Mezenner N.Y. (2009). Synthetic textile effluent removal by skin almonds waste. Journal of Environmental Science and Technology, 2(4), pp. 153-169.
4. Bechtold, T., Mussak, R., Mahmud, Ali., A. Gabglberger, E. and Geissler, S. (2006). Extraction of natural dyes for textile dyeing from coloured plant wastes released from the food and beverage industry. Journal of Science of Food and Agriculture, 86(2), pp. 233-242.
5. Bensmaili, A. and Mezenner, N.Y. (2009). Synthetic textile effluent removal by skin almonds waste. Journal of Environmental Science and Technology, 2(4), pp. 153-169.

6. Bhargava, Deepti., and Jalan, Shahnaz. (2013). Microbial Dyes: A new dimension to natural dyes. Colourage, 60(7), pp. 42-43.
7. Bhattacharya, S.D. and Shah, A.K. (2000). Metal ion effect on dyeing of wool fabric with catechu. Journal of Society of Dyers Colorists, 116 (1), pp. 10-12.
8. Chen, Paul., Cheng, Yanling., Deng, Shaobo., Lin, Xiangyang., Huang, Guangwei and Ruan, Roger. (2010). Utilization of almond residues, International Journal of Agricultural and Biological Engineering, 3(4), pp. 1-18
9. Deniz, F. (2013). Dye removal by almond shell residues: studies on biosorption performance and process design. Material Science and Engineering C, 33(5), pp.2821-2826.
10. Deniz, F. (2013). Adsorption properties of low cost biomaterial derived from Prunus amygdalus L. for dye removal from water. The Scientific World Journal, 2013, pp. 1-8.
11. Esfahlan, A.J., Jamei, R. and Esfahlan, R.J. (2010). The importance of almond (Prunus amygdalus L.) and is by-products. Food Chemistry, 120(2), pp. 349-360.
12. Knizova, Hana. (2015). Natural dyes: their past, present, future and sustainability. Recent Developments in Fibrous Material Science, Křemenáková D. (ed.), Militký J. (ed.), Mishra R. (ed.), Ops Kanina, pp. 59-71
13. Laufenberg, G., Kunz, B. and Nystroemm, M. (2008). Transformation of vegetable waste in value added products: (A) the upgrading concept; (B) Practical Implementations. Bioresource Technology, 87(2), pp.167-198.
14. Majid, Aliabadi., Imane, Khazaei., Mohsen, Hajiabadi and Fazel, Shahrzad. (2012). Journal of Biodivesity and Environmental Sciences, 2(9), pp. 29-44.
15. Mirjalili, Mohammad. and Loghman, Karimi. (2013). Extraction and characterization of natural dye from green walnut shells and its use in dyeing polyamide: Focus on antibacterial properties. Journal of Chemistry, 2013, pp. 1-9.
16. Mohmmad, Gias Uddin. (2014). Effects of different mordants on silk fabric dyed with onion outer skin extracts, Journal of Textiles, 2014, pp. 1-8.
17. Naz, Saima., Bhatit, Ijaz A. and Adeel, Shahid. (2011). Dyeing properties of cotton fabric using un-irradiated and gamma irradiate extracts of Eucalyptus camaldulensis bark powder. Indian Journal of Fibre & Textile Research, 36(June), pp. 132-136.
18. Özlenen, İşmal Erdem and Yıldırım, Leyla. (2012). Almond shell as a natural colorant. Indian Journal of Fibre & Textile Research, 37(4), pp. 358-363
19. Özlenen, İşmal Erdem, Yıldırım, Leyla. and Ozdogan, Esen. (2014). Use of almond shell extracts plus biomordants as effective textile dye. Journal of Cleaner Production, 70, pp. 61-67.
20. Pehlivan, Erol, and Altun, Turkan. (2008). Biosorption of chromium (VI) ion from aqueous solutions using walnut, hazelnut and almond shell. Journal of Hazardous Matter, 155(1-2), pp. 378-384.
21. Samanta, Asis Kumar and Agarwal, Priti. (2007). Dyeing of jute and cotton fabrics using Jackfruit wood extract: Part I – Effect of mordanting and dyeing process variables on colour yield and colour fastness properties. Indian Journal of Fibre & Textile Research, 32(4), pp. 466-476.
22. Samanta, A.K., Agarwal, P. and Dutta, S. (2008). Physio-chemical studies on the dyeing of jute and cotton fabrics using jackfruit wood extract: Part II- Dyeing kinetics and thermodynamic studies. Indian Journal of Fibre and Textile Research, 33(1), pp. 66-72.
23. Samanta, A.K., Agarwal, P., Singhee, D. and Dutta S. (2009). Application of single and mixtures of red sandalwood and other natural dyes for dyeing of jute fabric: studies on colour parameters / colour fastness and compatibility. Journal of Textile Institute, 100(7), pp. 565-587
24. Samanta, A.K., Konar, A., Chakraborti, S. and Dutta, S. (2011). Dyeing of jute fabric with tesu extract: Part I- Effects of the different mordants and dyeing process variables. Indian Journal of Fibre and Textile Research, 36(1), pp. 63-73.
25. Saxena, S. & Raja, A.S.M. (2014). Natural dyes: sources, chemistry, application and sustainability issues. Roadmap to sustainable textiles and clothing, Springer Singapore, pp. 37-80
26. Sundaram, Meenakshi, M. and Sivakumar, S. (2012). Use of Indian almond shell waste and groundnut shell waste for the removal of azure a dye from aqueous solution. Journal of Chemical Pharmaceutical Research, 4 (4), pp. 2047-2054.
27. Tomer, P., Singh, J. and Saroj. (2004). Utilisation of banyan leaves for wool dyeing. Textile Trends, 46(11), pp. 25-27.