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Eco Friendly Dyeing with natural dye - Areca nut; enhancing colour fastness with natural mordants (Myrobalan, Lodhra and Pomegranate) and increasing the Antibacterial Activity

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ABSTRACT

The main aim of the study is to use natural mordants to improve the colour fastness of the natural dyes in tandem with environment and reproducible methods. To accomplish the formulated objective, experiments in various proportions with the natural dyes and natural mordants to achieve the colour fastness and visual inspection of the dyed samples and analyzing the colour fastness tests using standards were planned . During the pilot study, it was experiential that pre mordanting method is most effective in achieving colour shades and colour fastness than the post and simultaneous mordanting method. Comparative study was conducted between the three mordants on bases of colour change, colour (Light, wash and perspiration) fastness, depth of shade dye. Lodhra showed excellent colour fastness and also allows the dyes to show the real colours, whereas pomegranate and myrobalan covers up the dye's colour with their natural colour. Pomegranate displays excellent anti bacterial activity than the other two mordants. While conducting the study it was also observed that the effluent generated from this whole process is not harmful and can be used for farming /gardening activities. The water used for dyeing and mordanting can be reused by simple steps of recycling. The study authenticates natural dyeing with natural mordanting method is an excellent example for conserving environment yet able to colour the cotton fabric with various shades.

Keywords: Acrea Nut, Natural mordants, Colour fastness, Antibacterial and Eco friendly.

INTRODUCTION

The dyeing came into existence from ancient times. There is no written evidence in form of paper it is very difficult to know the exact date or years of its inception. During excavation clay tablets were found dated 2200 B.C. which prove the presence of dyers and weavers. Earliest written records of using dyes was found in China dated 2605 B.C. To make fabrics colourful they did tried varieties of natural materials for dyeing.

The natural dyes such as indigo and madder were used to textile materials were found in Roman graves during middle of 2nd and 3rd century. the manuscripts of Rome and Greek mention the dyeing and its use but do not disclose the dyeing processes. In Egyptian tombs fabrics with different colours were exhibited. The mummy cloths were dyed yellow by treating the fabrics with safflower natural dye or by mineral colour. The colour extracted from the insect body was applied on fabric and treated with metallic hydroxide of obtain red colour. Indigo dyes were also in use. India was the first to develop red and blue colour with madder and indigo respectively. In ancient times, vegetable dyes with tannic acid were commonly used.

In early 5th century A.D. purple coloured fabrics were used by Byzantium royal people for mourning and this coloured fabric was forbidden for common people. In Chinese manuscripts there is mention of batik i.e. wax prints in late 7th century. In 9th century the first medieval association of craftsmen was established in Europe. In 12th

century the dyeing flourished and new colours were invented. In 13th to 17th century there was vast growth of new equipments and colours throughout the world.

Many dyes were manufactured from coal tar in mid 19th century by Mauveine. Till the 19th century dyers relied on the natural sources for dyeing. During late 19th century William Henry Perkin discovered lavender dye accidentally produced artificially from coal tar and this was the first step towards the decline of natural dyes. Natural colours play a vital role from ancient times in commercial and household use. The invention of synthetic dyes and its simple way to use, cheaper in cost compared to natural dyes, easy storage and good colour yield had downfall of natural dyes.

Using of waste to extract colour and utilize for the dyeing of new fabric is a kind of recycling which is not only helping the environment but also promotes nature. Using a vegetable waste to extract colour not only gives the colour yield but also saves the environment from the hazards waste produced from the synthetic colours. The use of food waste is a small step towards saving the environment and going back to the ancient times where all these products were used.

Exhaustive work was carried out to find substitute for myrobalan (harda)[16],[20]. the researcher found a new vegetable origin to achieve the colour value of the fabric with comparable cost in pomegranate rind which gives comparable or 5 - 10% more colour value ie. deeper shade on the fabric and is commercially available in abundance in the market. Pomegranate rind is about 50% cheaper than myrobalan. it is found that apart from shades, the fastness properties obtained were equal to those treated with myrobalan. It also produces different hues not possible by myrobalan. As pomegranate rind is a vegetable waste, commercially available in abundance and has medicinal value like myrobalan, it can be used as a substitute for sustainable source of vegetable mordant.

The symplocos bark were used as mordants for their natural red dye process in Indonesia. But it is difficult to sustain the traditional craft because harvesting of symplocos bark is unsustainable. Bebali foundation has promoted the symplocos leaves to be used instead of bark through the research conducted by Royal Botanical Gardens at Kew (2002) proves that symplocos racemosa have high tannin content and gives very good fastness properties as mordants. It was found through research symplocos racemosa leaves gave brilliant effect on protein and cellulosic fibres (Maiwa & Bebali foundation).

In the present investigation an attempt has been made to optimize the dyeing condition of some natural colorants on bamboo and cotton fabrics. The chief reason of starting the study was to make use of onion peels which are available in abundance but not used. Various factors like pH of the dyebath, dyeing temperature and time, concentration of dye used were considered to standardize the dyeing condition. The preliminary examination showed the natural dye obtained from areca nut produced good colours with natural mordants on cotton fabrics.

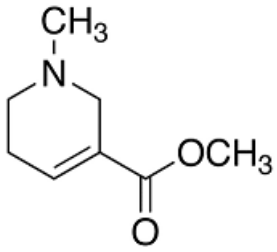
MATERIALS AND METHODS

2.1. Selection of Fabric

Cellulosic fibers was selected for the study, as these fabrics are eco friendly natural fibers and give brilliant shades with selected dye.

2.1.1.Material:

- Natural dyes: Areca Nut (Betel nut) were procured in liquid form (*chogaru*) from betel nut supplier in Hubli.



The areca catechu nut dye liquid is processed in following steps:

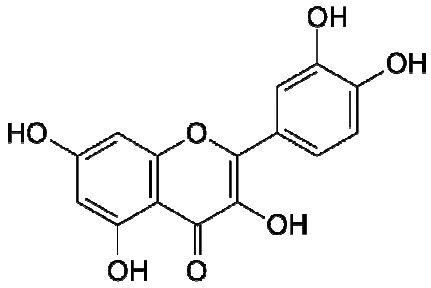
➢ Plucking: The bunches of arecanut appear near the top close to leaves. Climbing up an area palm and plucking the bunches is considered to be a special art and techniques. It is a practice that climber will test the nuts whether it has matured or not before cutting the bunches. He is called as "konegowda" in local language

➢ Dehusking: The next process is that of husking and curing. Trained women coolies are employed for husking, which they do it with help of ironed sharp knife which is fixed on wooden piece. Incase of tendered nuts, husking process is taken soon after harvest within 4 days. If it is delayed quality suffer.

➢ Boiling: The tender nuts are boiled soon after the husking in copper vessel for about a couple of hours. While boiling the nuts, the most common used in the district is that when the nuts loosen the eye. It is supposed to be fully boiled. In order, to improve the colour and quality of nuts barks of teak tree, lime and betel leaf is added. When the nuts are fully boiled, they are taken out from the vessel by means of perforated spoon and poured in cane basket. This process requires lot of firewood while boiling.

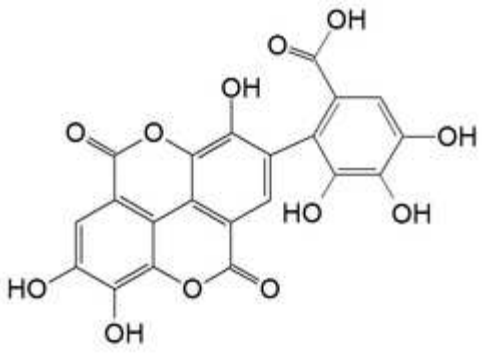
➢ Colouring Nuts: After the boiling of arecanut in copper vessel, the water remained in the vessel is not wasted, in fact, it is reboiled for couple of hours and little thick liquid is prepared, and now it is called tannin or Chogaru. The sorted and graded tender nuts are soaked or coated in this tannin to get a glossy and attractive red appearance. This process is called colouring. Alkali and wood flour was added at 1.5 parts and 5 parts added to 100 parts of solid chogaru diluted with 250 parts of water and gives good adhesive combination for a red dye could be prepared for the nut and could be used for dying cloths and ropes Ink of good quality can be manufactured from the immature nests. when the consistency of the water is thicker it is packed in dark containers and the remain of the liquid is further dried in the pit and used as areca dust. The areca dust is sold for pan masala.

• Tannin Mordants: The natural dyes are either adjective which requires mordants or substantive which does not require mordants. Tannin is an astringent vegetable product found in a wide variety of plant parts such as bark, wood, fruits, fruits pods, leaves, roots and plant galls. Tannins are defined as naturally available polyphenolic water soluble compounds of high molecular weight (500-3000) contains phenolic hrdroxyl groups to facilitate effective crosslinks between proteins and other macro-molecules[37]. Myrobalan is the fruit of trees such as Terminalia Chebula and Myrobolanus Chebula. Myrobalan was procured from the ayurvedic clinic in powder form as waste in Hubli market.



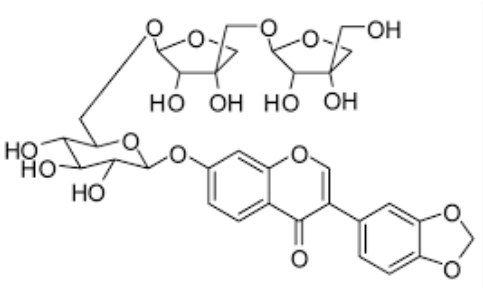
The peel of the fruit has bulk of tannin is known as ellagitannic or ellagic acid containing 58-60 % by weight tannins. Myrobalan are very rich in hydrolysable tannins. Myrobalan contains a yellowish brown colouring matter when used on textiles is a good substitute for tannic acid.

The dual use of pomegranate for dyeing with or without mordant was known by two ancient Middle Eastern civilisations. All the recipes for greens in an Indian treatise dating from the Mughal period, the Nuskha Khulasatul Mujarrebat, include pomegranate rind, used either on its own or with other yellow dyes or with other tannin rich plants (pistacia galls, cutch or myrobalan)[11].Pomegranate rinds are rich in tannin and are very good mordants to obtain the shade of brown, grey and black shades. The tannins are complex polyhydric phenols of high molecular weight has large molecules and have good affinity for cellulosic and protein fibres resulting in high light and wash fastness.



Pomegranate rind were procured from Hubli market. The dried rind were pounded in powdered form by hand pounding in Gajendragarh village.

Symplocaceae genus there are about 250 species of symplocos[10].



Lodh plant is used as great source for ayurvedic medicines for ages in India dating from end of the Vedic period (1000-500 B.C.)[16]. The leaves have a high aluminium content (13%-more than 27% of the weight of ashes) present in the plants in the form of oxalate or tartrate. Lodh leaves when dried changes natural green colour to yellow. This yellow leaves are rich source of tannin (45-60%).

Lodh leaves were procured from Agumbe rain forest in dry condition. And the dry leaves were hand pounded in powdered form in Gajendragarh village.

- Yarns: Cotton yarns were procured from Cooperative society in Gadag district. Yarns with 2/20's, 2/40s, 30s, 20s and 40s were procured. As determined during pre-testing, the fabrics were wound into samples weighing 7±0.02 g for pre-mordanting and dyeing. At least 30 samples were wound from each dyeing according to AATCC Test Methods 16-2014 and 61-2014, with the specimens were selected randomly from different bobbins of 20s count (AATCC, 2014). The calculated sample sizes not only allowed for level mordanting and dyeing, but they accommodated the specifications and replications required of specimens for subsequent colour fastness to light and laundering tests.

2.1.2. Mordanting:
The powdered mordants were packed in a mesh cotton cloth pouch according to the concentrations of each, Pomegranate 8% &10%, Myrobalan 10% &15% and Lodh 25% & 50%.

The yarns were randomly assigned to one of the six experimental treatments, representing three mordant agents. The packed cotton pouches along with the heated water and remaining 180 ml of water were inserted in the canisters along with Each 7 g sample was mordanted individually. An Launder-O-meter was used for mordanting to ensure accurate timing and consistent temperature and agitation. The machine rotates the samples in closed stainless steel canisters in a thermostatically controlled water bath.

Approximately 30 minutes before mordanting, the 7 g samples were soaked in water to wet out the fibres to ensure more even uptake and dyeing. Liquor ratio used for mordanting was 1:40. The mordant was pre-dissolved in 100 ml of boiling water. Then the remaining 180 ml of water at 25°C (77°F) was added to cool the mordant solution to approximately 30°C (86°F). The solutions were stirred when the boiling water was added, and again after the cool water was added. The solutions were poured into canister, followed by the wetted out sample that had been squeezed to remove excess water. Each canister was closed and shaken by hand 4-6 times to begin the agitation process.

They were then loaded into the machine for the initial 90-minute cycle. During this time, the temperature was increased from 25°C (77°F) to 80°C (179°F). After 90 minutes, the machine lid was opened and the drain was turned open for a few minutes while half of the water was allowed to drain out. With the heat turned off, the cold water valve in the Launder-O-meter was turned on and then the machine was turned on again for 20 minutes while cold water entered and water continued to drain out. When the temperature in the water reservoir inside the machine decreased to 25°C (77°F), the canisters were unloaded, and the samples were removed.

The samples were rinsed under a water faucet by hand for 30 seconds, then squeezed and twisted thoroughly to remove excess water. The samples were soaked in water for approximately 25 minutes while the canisters were washed and prepared for dyeing.

2.1.3. Dyeing: In order to reduce mixing errors within a specific type and concentration treatment combination, stock dye solutions were prepared as for dyeing individual samples. Betel nut already in liquid form was used directly as a dye stock.

Table no. 1: Mordanting and Dyeing Details

Table No. 4		Mordanting Details			Dyeing with Betel Nut			
Mordant	**Concentration**	**MLR**	**Temp**	**Duration**	**10%**	**20%**	**30%**	**40%**
Control	**No mordant**	1:40	80	120	MLR - 1:20, Temp - 90^0C, Duration 120minutes			
Harda/ Myrobalan	8%	1:40	80	120				
	15%	1:40	80	120				
Anar/ Pomegranate	8%	1:40	80	120				
	10%	1:40	80	120				
Lodh/ Lodhra	25%	1:20	80	120				
	50%	1:20	80	120				

To dye the pre-mordanted fabric samples, 280 ml of dye solution was poured into individual Launder-O-meter canisters, followed by the wetted out, pre-mordanted sample.

The canisters were shaken by hand 4-6 times to begin the agitation process. They were then loaded into the Launder-O-meter for the initial 9- minute cycle where the temperature was raised from 25°C (77°F) to 80°C (179°F). After 90 minutes, the machine lid was opened and the drain was turned on until half of the water had drained out. With the heat turned off, the cold water valve was then turned on, and the machine was turned on again for 20 minutes while cold water entered and water continued to drain out. When the temperature decreased to 25°C (77°F), the canisters were removed, and the samples were taken out.

The samples were rinsed under a water faucet by hand for 30 seconds, then squeezed and twisted thoroughly to remove excess water. The samples were soaked in water for approximately 25 minutes while the canisters were washed and prepared for dyeing.

2.1.5. Colour Fastness Tests
2.1.5.1. Light Fastness: The dyed samples were tested using AATCC method 16-2014, using Xenon - arc lamp at a temperature 63$\pm$ 1^0 C and relative humidity 30$\pm$5%.

2.1.5.2. Wash Fastness: The dyed samples were tested using AATCC method 61- 2014, Test no 2A, using laundrometer at a temperature of 49^0 C.

2.1.5.3. Colour Evaluation:
2.1.5.3.1.Colour Change: Color change in the dyed specimens was assessed following AATCC Evaluation Procedure 1, Gray Scale for Color Change (AATCC, 2014). The assessment was done visually by comparing the amount of colour change between the control and the exposed specimens against the steps on the Gray Scale for Color Change.

2.1.5.3.2.Staining: Staining of the undyed multifiber fabric No. 1 in the colourfastness to laundering test was evaluated according to AATCC Evaluation Procedure 2-2007, Gray Scale for Staining (AATCC, 2009).

2.1.5.3.3.Comparison of Colour Parameters in Dyed Specimens: The colour differences in the pre- and post-test specimens were assessed using AATCC Evaluation Procedure 9-2007, Visual Assessment of Color Differences of Textiles.

2.1.5.3.4.Anti bacterial: For the antibacterial study, five bacterial pathogens E. Coli, B. Cereus, B.Subtilis, Staphylococuss aureus, K. Pneumoniae were used to screen the possible antimicrobial activity of mordants and dyes.

RESULTS AND DISCUSSION

Selection of dye material: The areca nut on cotton showed a shades of brown but had poor color fastness. The mordanted cotton showed range of deep browns. The cotton mordanted with lodh showed excellent color fastness in comparison to myrobalan and pomegranate rind.

Optimization of Different Variables of Dyeing Condition: This part of study included the visual evaluation of samples on the criteria of luster, evenness of dye, depth of shade and overall appearance by a panel of judges. The

natural mordants usually over power the dye colour which can be seen in myrobalan and pomegranate rind. The lodh mordants allows the dye to give true colour to the cotton fabric.

The optimum concentration of dye material: The optimum concentration of dye material was carried out by heating 65 – 70°C at varying concentration of dye material (10%, 20%, 30% & 40%) for 120 minutes. By visual inspection by panel of judges, on basis of lustre and depth of shade. The best shades were obtained by using areca nut using lodh mordanted cotton gave good colour yield.

Optimum Time for Dyeing: In order to find the optimum time for dyeing the cotton samples were dyed for different timing (30, 60, 90 and 120 min.). On the basis of visual inspection 60 min. time was selected for dyeing, which provided best shades.

Method of Mordanting: Dyeing of cotton fabric with acrea nut was carried out in the presence of natural mordant, following three different methods, namely pre-mordanting, post mordanting and simultaneous mordanting. Out of three methods of mordanting, it was found that best shades of colour were obtained by Pre-mordanting method.

Colour Fastness to Light and Washing: The colour fastness to light and washing for cotton samples dyed with onion peel dye in absence and presence of mordanting agent.

Anti bacterial: In this study, five different bacteria were used to screen the possible antimicrobial activity of mordants and dye. Samples mordanted with pomegranate rind showed excellent anti bacterial activity lowest was seen in lodh mordanted samples.

Table no. 2 Colour Evaluation Acrea nut dyed samples

Acrea Nut					Colour Change				Staining				Comparison of Colour Parameter			
Mordant	Concentration	LQR	Temp	Duration	30%	40%	50%	60%	30%	40%	50%	60%	30%	40%	50%	60%
Control	No mordant	1:40	80	120	2	2	2	2	2	2	2	2	off Shade			
Harda/ Myrobalan	8%	1:40	80	120	3	3	3	3	3	3	3	3	3	3	3	3
	15%	1:40	80	120	5	5	5	5	4	4	4	4	5	5	5	5
Anar/ Pomegranate	8%	1:40	80	120	3	3	3	3	3	3	3	3	3	3	3	3
	10%	1:40	80	120	4	4	4	4	4	4	4	4	5	5	5	5
Lodh/ Lodhra	25%	1:20	80	120	4	4	4	4	4	4	4	4	3	3	3	3
	50%	1:20	80	120	5	5	5	5	5	5	5	5	5	5	5	5

Table no.3 Comparison of Antibacterial acitivity with various mordants dyed with Acrea Nut

Sr.No.	Bacterial Culture	MTCC No.	Pomegranate Rind	Myrobalan	Lodh
1	*E. Coli*	739	21	18	12
2	*B. Cereus*	1307	25	22	16
3	*B.Subtilis*	6910	20	20	17
4	*Staphylococuss aureus*	7405	26	22	15
5	*K. Pneumoniae*	7028	21	22	12

CONCLUSION

The experiments prove that acrea nut dye has 50-60% tannin content in dry state but on dyeing on cotton without mordant showed poor colour fastness. There is need of mordant for improve colour fastness while dyeing with acrea nut. The best shades were obtained by using Acrea nut with pre mordanting with lodh. Lodh showed excellent colour fastness and also allows the dyes to show the real colours, whereas pomegranate and myrobalan covers up the dye's colour with their natural colour. Pomegranate displays excellent anti bacterial activity than myrobalan. Lodh exhibited poor anti bacterial activity than the other two mordants. While conducting the study it was also observed that the effluent generated from this whole process is not harmful and can be used for farming /gardening activities. The water used for dyeing and mordanting can be reused by simple steps of recycling. The study authenticates natural dyeing with natural mordanting method is an excellent example for conserving environment yet able to colour the cotton fabric with various shades.

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