



NATURAL DYES AND MORDANTS

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Abstract- The purpose of this work is to extract natural dyes from plant sources and study the effect of mordants on them found in Lazami village Zunheboto, Nagaland, India. The use of natural dyes for dyeing textile has decreased to a large extent after the discovery and synthesis of chemical dyes. Dyes obtained from natural sources have emerged as an important alternative to synthetic dyes. The plants used in this work were *Buddleja officinalis*, *Syzygium Syzgioide*, *Phlogacanthusthysiformis*, *Rhododendron* and *Alophyllussubfalvatusvar. distachyus*. Phytochemical screening and functional group test were also performed on the extracted dyes. Analytical studies such as IR spectrophotometer were also observed. The extracted dyes from different plant sample and parts were used for dyeing on the cotton clothes for the fixation of colour. Several mordants such as Potash Alum, Stannous chloride, Aluminium sulphate, Potassium dichromate were used for fixing colour for better colouration using two methods *i.e.* pre-mordants and post-mordants. The best results were obtained from stannous chloride giving desirable product. However fastness properties of these natural dyes should be improved for commercial use.

Key word: Auxochrome, mordants, copper sulphate, potash alum, pre-mordants and post-mordants.

Introduction:

Natural dyes are considered as one of the most important gift from nature to human as they are related with cultural practices, rituals, arts and crafts, fabrics and emotions, Whereas dye producing plants have not received much attention and care. Recent publications of research articles and reports on the vegetable dyes have generated the interest of researchers on natural dyes.

Natural's dyes or colorants are believed to be safe because of its nontoxic and biodegradable character. Many tribes of Arunachal Pradesh have been using this plant species traditionally in combination with other plants for extraction and preparation of dyes utilizing indigenous processes (Mahanta & Tiwari, 2005). Natural dyes are not so productive in commercial like synthetic dyes which are not eco friendly. Currently artificial dyes were introduced in the textiles industry. The synthetic compound are hazardous to health in living being, pollutes environment and huge effluent issues. Natural dyes will solve this problem. It is important to develop the technique for extraction of naturally occurring dyes and for their used in the apparel fibers. (Joyti Arora et.al., 2017). Besides, in the field of textile and garments, it is also used for colouring food, hand of crafts and in the application of medicine. Though natural dyes are eco-friendly, biodegradable, renewable, their intermediate bond of dyeing with the fabrics is not desirable. Effects of some mordants on natural's dyes have to be applying on the process of dyeing the fabrics for the better colouration. Some of the mordants are non-biodegradable and toxic which have the huge impact on the environment. Therefore much research is carrying on in the natural dyes for different natural fibers to get a better products and eco-friendly textile. (Virendra Kumar Gupta, 2019). Walnut dye extract can be used to dye woollen yarn with or without alum mordants to produce a variety of light and bright greyish to yellowish brown shades of varying depth (Khan et.al., 2015).

Some of the important sources of natural dyes are onion, carrot, marigold, orange peel, rose etc (Arun KautiGuta, 2019). The colour fastness of natural dyes not only depend on chemical nature and type of natural colorants, but also on chemical nature and type of mordants used (Samanta and Agarwa, 2009). Wash fastness of the dye is influenced by rate of diffusion of the dye inside the fiber (Jothi 2008). Whether pre-mordanted or simultaneous mordanted or post-mordanted, the colour depends not only on the natural colorants but also on the mordant and mordanting assistants. The replacement of synthetic dyes by natural dyes will not only be transforming the industrial fabric into handcrafted fabric, but also lead to a more environment friendly textile with the same quality standards achieved with synthetic dyes. Natural dyes are not an innovation but a revival of a rich and prudent tradition and cannot be compared with synthetic dyes only in terms of efficiency for industrial applications. However, in present days we need to ensure that the replacement of synthetic dyes by natural dyes will not only be transforming the industrial fabric into handcrafted fabric, but also lead to a more environmentally friendly textile with the same quality standards achieved with synthetic dyes. All natural dyes based on the vegetable origin are renewable (Chavan, 1995; Paul et. al., 1996 and Gupta, 1990). The strength of culture in natural colour is gaining so much popularity in textile industry due to their eco- friendly and desire colorant (Shahid et. al., 2018). Natural dyes have other functional properties such as anti-fungal, anti-viral activity (Gupta et. al., 2004), insect repellent (Ali et al., 2013), UV protection (Sun and Tang, 2011) antimicrobial activity (Khan et. al., 2012; Shahid et. al., 2013), and deodorizing agents (Lee et. al., 2009) in addition to their biodegradable and eco-friendly character.

Most of the mordants used prior to the nineteenth century contained transition metals such as Cu^{2+} and Cr^{6+} the use of which is not presently recommended in view of their eco-toxicity. With the aim of supporting an eco-friendly approach, Al^{3+} and Fe^{2+} salts have been employed to lower the potential for harm to human health and the environment. Standardization of the dyestuff is required to minimize variability in the dyeing result due to quality differences of plant material (Leitner et. al., 2012).

Materials and Methods

GEOGRAPHY OF PLANTS

All the plants were taken from Lazami village under Zunheboto district, Nagaland with a total population of 2936 with 654 household. It has a higher literacy rate compared to Nagaland 79.55% of which it has 82.76% according to 2011 census.

Types of plants

1 *Buddleja officinalis*:

Scientific name – *Buddleja officinalis*, Family - Scrophulariaceae

Common- Mi Meng Hau Local name- Ajunebo

Buddleja officinalis has green leaves and bears terminal spike of white flowers in early spring. In early days the villagers of Lazami village use the flowers and extract yellows dyes for dyeing wool, which is used for weaving traditional shawl (**Amighephi**).

2. *SyzygiumSyzgioide*:

Scientific name – *SyzygiumSyzgioide* , Family – Myrtaceae

Common name – kelat Hitam, Local name - Ayichebo

SyzygiumSyzgiodes is an evergreen shrub or a tree that can grow up to 15 meters tall. The bole can be up to 85cm in diameter, either with flutes at the base or with short buttresses up to 1.5meters high. The locals eat fruits of the plants and it is not poisons some even believe that Diabetes patient can be cure by eating a fruits.

3. *Phlogacanthusthysiformis*:

Scientific name – *Phlogacanthusthysiformis* Family - Acanthaceae

Common name- Nongmangkha Local name- Shiqhenipuniye

It is an evergreen shrub grows up to 2.4 meters high, branch lets quadrangular, leaves 13- 32cm long, elliptic-oblong, acute or acuminate, entire. Flowers in terminal elongated, thyroid panicles, up to 30cm long, corolla tubular, curve, orange or brick red villas. Capsule 3.8cm long linear- cleave. The plant leaves are used for cooking by the locals; it is one of the most common dishes.

4. *Rhododendron*:Scientific name- *Rhododendron*

Family – Ericaceae

Common name – Azalea

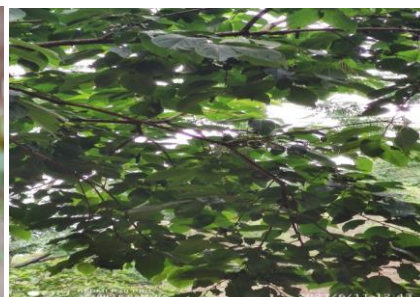
Rhododendron is a very large genus of 1024 species of woody plants in the healthy family evergreen or deciduous found mainly in Asia. It is also wide spread throughout the low land. It is characterized by having inflorescences with scarious perulae. They are best known for their many clusters of large flowers which have different varieties of colours. Ever green azaleas do not lose most of their leaves over the winter, unlike deciduous azaleas, which drop their leaves in the fall.

5. *Alophyllus subfalvatus* var. *distachyus*:Scientific name – *Alophyllus subfalvatus* var. *distachyus*

Family – Sapindaceae

Local name- Ashiqheniye

It is a large shrubs; bark lenticelled, grayish white. Leaves long petiole, 3-folited, leaflets ovatelanceolate, long-acuminate, unequal at base, lateral ones usually small. Racemes dense flowered, un-branched. Fruits clavate-ellipsoid, glabrous, orange red seeds with short aril.

*Buddleja officinalis**Syzygium Syzgioides**Phlogacanthus thyrsoformis**Rhododendron**Alophyllus subfalvatus* var. *distachyus***Materials required:**

Plant sources, Cotton cloth, Beakers, Conical flask, Knife, Funnel, Filter paper, Tripods and Mordants used are Potash Alum, Stannous chloride, Potassium dichromate, Ammonium sulphate and Reagents are Ceric Ammonium, Potassium hydroxide, Carbon sulphide, Phthalic anhydride, Sulphuric acid, sodium hydroxide, 2, 4-DNP, ferric chloride.

Methodology:

Extraction method: The collected samples (flowers, leaves, bark, stem) were sun dried for a week until it can be crashed into smaller particles and it is used to perform the experiment in two methods, Aqueous (water bath) and Alcoholic (Soxhlet)

Aqueous method (water bath): In this method, dyes from dried samples (flowers, leaves, tree bark, stem) were extracted. In 1000ml of beaker 200g of dried sample in 1/3 of distilled water was used, and the extraction process was carried out at temperature range of 80-85°C for 2 hours using water bath. It was filtered and concentrated to ¼ of it followed by Functional group test, Phytochemical screening, Infrared (IR) absorption and Dyeing process.

Alcoholic method (Soxhlet): In this method, extraction of plant samples was done using Soxhlet apparatus. 50g of plant samples were fitted inside the siphon tube of the Soxhlet apparatus using 200 ml of methanol as solvent. The soxhlet was run for about 6 hours for effective extraction and extracted dyes were then filtered and evaporated leaving it behind.

Preparation of Mordants:

Potash Alum, Aluminium Sulphate, Stannous Chloride were prepared taking 1g of each and dissolve in 100ml of distilled water . Potassium dichromate was prepared taking 1g of it and dissolves in 300ml of distilled water.

Functional group test: Test of different functional groups were done and results are shown in table- 3

Phytochemical screening: Test for Tannins, Saponins, Terpenoids, steroids and Flavonoids were done with suitable reagents and results are shown in table -4.

Dyeing process:

The extracts obtained from aqueous method (water bath) were used for dyeing cotton clothes.

Two methods were used for dyeing fabric.

Pre-mordants: The cotton clothes were put in each respective mordants for 10-20mins and dried in a sun, then it is transfer to extracted natural dyes and kept for 30mins and again sundried for better coloration.

Post-mordants: The cotton cloths were first put in extracted natural dyes for 10-20 minutes and dried in a sun, again it is transfer to each respective mordants for 30 minutes and dried in a sun for desirable outcome.

RESULTS AND DISCUSSION:

The dyes extracted from the plants and colour of the dyes is depended on the compounds present in the respective plant. The dye is then used in the cotton fabric for the fixation of colour. Different mordants were used on the same plants to give different shades of colours and then enable us to make different types of shades from one plant using the mordant.

Table-1: Effect of different mordants with dyes extracted from plant on cotton cloth

Plant and Parts	Mordants	Colour obtained with the treatment of mordants	Colour obtained with the treatment of mordants and extracted dye
<i>Buddleja officinalis</i> (Flowers)	Stannous chloride	Creamy white	Yellowish
	Potash alum	Colorless	Light Yellowish
	Aluminum sulphate	Colorless	Dark yellow
	Potassium dichromate	Dark yellow brown	Dark brown
<i>Rhododendron</i> (Flower)	Stannous chloride	Creamy white	Orange Yellowish
	Potash alum	Colorless	light Yellow
	Aluminum sulphate	Colorless	light pink
	Potassium dichromate	Dark yellow brown	Dark yellowish brown
<i>Syzygiumsyzygiodes</i> on (Tree bark)	Stannous chloride	Creamy white	Peach/Orange
	Potash alum	Colorless	Peach
	Aluminum sulphate	Colorless	Dark orange
	Potassium dichromate	Dark yellow brown	Dark brown
<i>Alophyllus subfalvatus</i> var. <i>distachyus</i> (Leaf)	Stannous chloride	Creamy white	Yellow
	Potash alum	Colorless	Light yellow
	Aluminum sulphate	Colorless	Light brown
	Potassium dichromate	Dark yellow brown	Dark brown
<i>Phlogacanthus thyrsiformis</i> (Stem)	Stannous chloride	Creamy white	Brown
	Potash alum	Colorless	Light brown
	Aluminum sulphate	Colorless	Brownish orange
	Potassium dichromate	Dark yellow brown	Dark brown

Table-2: Effect of different mordants with extracted dye

Plant	Mordants	Colour obtained with the treatment of extracted dye	Colour obtained with the treatment of extracted dye and mordants
<i>Phlogacanthusthysiformison</i>	Stannous chloride	Light brown	Brownish
	Potash alum		Brownish
	Aluminum sulphate		Brownish
	Potassium dichromate		Yellowish
<i>Buddleja officinalis</i>	Stannous chloride	Yellowish	Light Yellow
	Potash alum		Light Yellow
	Aluminum sulphate		Light yellow
	Potassium dichromate		yellowish
<i>Syzygiumsyzygiodes</i>	Stannous chloride	Peach	Peach
	Potash alum		Light peach
	Aluminum sulphate		Peach/orange
	Potassium dichromate		Reddish brown
<i>Rhododendron</i>	Stannous chloride	Light pink	White
	Potash alum		White
	Aluminum sulphate		Off white
	Potassium dichromate		yellowish
<i>Alophyllusubfalvatus var. distachyuson</i>	Stannous chloride	Brownish orange	Off White
	Potash alum		Creamy White
	Aluminum sulphate		white
	Potassium dichromate		yellowish

Table -3: Functional group test

Functional group	Test	Plant samples	Results	remarks
Alcoholic group	Xanthate test	<i>Buddlejaofficinalis</i>	present	Yellow ppt.
		<i>SyzygiumSyzgioide</i>	Absent	-
		<i>phlogacanthusthyrasiformis</i>	Absent	-
		<i>Rhododendron</i>	Absent	-
		<i>Alophyllusubfalvatusvar.distachyus</i>	Absent	-
Phenolic group	Phtalein	<i>Buddlejaofficinalis</i>	present	No colour p-cresol
	Ferric chloride test	<i>SyzygiumSyzgioide</i>	Absent	-
		<i>phlogacanthusthyrasiformis</i>	present	Green (darken rapidly)
		<i>Rhododendron</i>	present	Reddish brown
		<i>Alophyllusubfalvatusvar.distachyus</i>	present	Green present of β -naphthol
Carbonyl group	2,4-DNP-test	<i>Buddlejaofficinalis</i>	Absent	-
		<i>SyzygiumSyzgioide</i>	Absent	-
		<i>phlogacanthusthyrasiformis</i>	present	Yellow ppt. presence of aldehyde and ketone
		<i>Rhododendron</i>	Absent	-

		<i>Alophyllus subfalvatus</i> var. <i>distachyus</i>	Absent	-
Amide group	Ammonia evolution test	<i>Buddleja officinalis</i>	present	Evolution of ammonia gas and a blue colour of red litmus paper.
		<i>Syzygium Syzygioides</i>	present	
		<i>Phlogacanthus thyrasiformis</i>	present	
		<i>Rhododendron</i>	present	
		<i>Alophyllus subfalvatus</i> var. <i>distachyus</i>	present	

Table -4: Phytochemical screening

Sl/no	Plant samples	Flavonoids	Steroids	Tannins	Saponins	Terpenoids
1	<i>Buddleja officinalis</i>	+	+	-	-	-
2	<i>Syzygium syzygioides</i>	++	+	++	+	+
3	<i>Ipomoea hederacea</i>	-	+	-	+	-
4	<i>Rhododendron</i>	-	+	+++	+++	-
5	<i>Phlogacanthus thyrasiformis</i>	+	+	-	+	+

The pre-mordant i.e the cotton cloth is dyed first in the mordants and then transferred to extracted dye. In post-mordant, the cotton cloth is first dyed with extracted dye and then immersed in mordants. The intensity of colour produced on cotton cloth by pre-mordant method is showed best colouration except in the mordant of potassium dichromate.

Yellowish dye was obtained from the plant sample of *Buddleja officinalis* flower.

Peach/ orange dye was observed from the plant of *Syzygium syzygioides* tree bark.

Light pinkish dye was obtained from the plant of *Rhododendron* (Azelea) flower.

Light brownish dye was obtained from plant of *Phlogacanthus thyrasiformis* stem.

Slight brownish dye was extracted from the plant sample *Alophyllus subjalcatu* var. *distachyces* leaf.

The present work showed that flowers of *Buddleja officinalis* and bark of *Syzygium syzygioides* obtained quite an efficient dyed. These extracted dyes can be treated with different mordants, colour fastness, wash fastness properties for commercial scale in textile industry.

In Lazami village, Nagaland people have been using the flowers of *Buddleja officinalis* for extraction and preparation of dye utilizing indigenous process. The flowers are collected during winter and used for dyeing. The dye is prepared by pounding the flowers and made it paste, it is then transferred to earthen pot. The wool is immersed together with flower paste and water in earthen pot for heating till it is boiled. It is then cooled and wool is taken out from pot and dried. Now the wool is used for weaving traditional shawl called **Amighephi**.

The phytochemical screenings of the present study revealed the presence of flavonoids and steroids in flowers of *Buddleja officinalis* and absence of tannins, saponins and terpenoids. In *Syzygium syzygioides* all the phytochemical screenings are present. Only steroids and saponins are in the flower of *Ipomoea hederacea*. Saponins and tannins are high in *Rhododendron* as compared to steroids. Except tannins all are present in *Phlogacanthus thyrasiformis*. The flowers which contain much of tannin are flame coloured and yield red/ pink/ brown/ flame coloured shades of dye depending upon the fabric. Functional groups are specific group of atoms within molecules that have their own characteristic properties, regardless of the other atoms present in molecules. Natural dyes extracted from plants had functional groups such as phenol, alcohol, amide, carbonyl groups.

Quantitative Analysis of plants

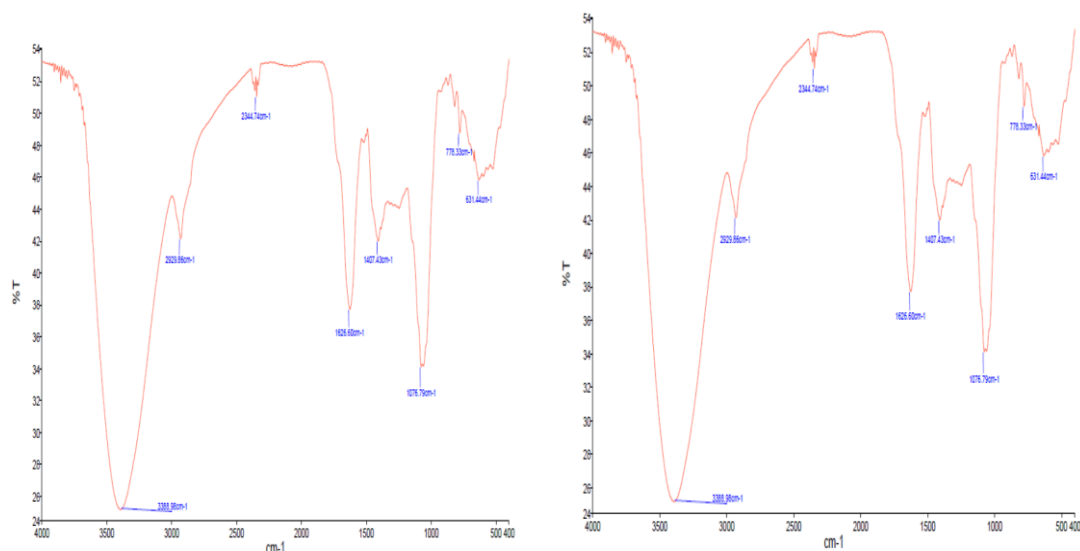


Fig1: IR spectroscopy of *Buddleja officinalis* flower *Syzygiumsyzygioides* bark

Interpretation

The IR spectroscopy of *Buddleja officinalis* flower shows the range of Phenol compound-3388nm (3200-3550nm)

Alkanes -2929nm (2850-3000nm)

Amines -1076nm (1000-1250nm)

Interpretation

The IR spectroscopy of *Syzygiumsyzygioides* bark flower shows the range of

Phenol compound-3397nm (3200-3550nm)

Alkanes -2928nm (2850-3000nm)

Alkenes -1632nm (1630-1680nm)

CONCLUSION

The main aim of this study is to extract the natural dyes from different plants found in Lazami village, Nagaland, India and treated with mordants on them for the better colorant. Some of the plant samples were found to have desirable colour which can be used for commercial purpose. Natural dyes are in high demand due to their non-toxic, eco-friendly, biodegradable and non-carcinogenic nature. Textile dyer must know the suitable solvent for extraction, good binding mordanting agents and various fiber for dyeing purpose and should follow the standard and suitable methods. Thus, with the worldwide concern over the use of eco-friendly and biodegradable materials, the use of natural dyes has undoubtedly gained interest and momentum of researcher and textile industry. The range of different colours given by the same dye-producing plant species by application of different mordants is found to be remarkable and can be considered as one of the advantages of natural dyes over synthetic dyes.

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