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Eco-friendly approach of Natural Dyes

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***Abstract*:** Natural dyes were extracted and analysed for their application in dyeing 100% pure cotton. Aqueous, alkali and solvent (Soxhlet) extraction methods are explored to assess their efficacy in obtaining vibrant and stable colourants for textile applications. The influence of mordants on dye uptake and colour quality is also examined to optimized dyeing processes. Ferrous Sulphate, Aluminium Sulphate and Potash Alum mordants were utilized to enhance colouration using three approaches: pre-mordanting, postmordanting and Trans/Meta mordanting methods. Phytochemical analysis and functional group testing were executed on the extracted dyes to further investigate their composition and characteristics. Analytical examinations for U.V. and I.R. were conducted. Sustainability is central to this research, prioritizing environmental impact reduction and ethical practices in dyeing.

Key word: Auxochrome, mordants, Ferrous Sulphate, Aluminium Sulphate and Potash Alum, pre-mordants and post-mordants.

1.INTRODUCTION

Colour has been a fundamental aspect of human existence, influencing every facet of our lives across the ages. (Bhuyan and Saikia,2004) .Dyes are agents that impart color to textile and they can either be natural or synthetic and they are both employed for colouring fibers, yarns and fabrics. (Kajla and Srivastava,2006).Yet, natural dyes are more favored because they are safer for health and the environment, making them an eco-friendlier choice.

The ancient days people were applying crushed fruits, vegetables or other materials onto the cloth for dying textiles . However, in recent decades, there has been a significant evolution in dyeing techniques and, materials due to innovative and advanced technologies. These newer methods not only minimize harm but also prioritize environmental friendliness, reflecting a shift towards sustainable practices in the textile industry. (Choudhury,2018; Sadeghi-Kiakhani et. Al.,2020; Baig et. al.,2021;Batool et. al.,2022;). The blue dye indigo, extracted from the Indigo-fera Tinctoria plant, holds significant historical importance as a renowned natural dye, often referred to as the “King of natural dyes”. Historically used for blue hues, it remains highly sought after today, notably in denim production. 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). 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.

Nagaland, a state in North Eastern India, boasts a rich cultural heritage deeply intertwined with the use of natural dyes. These dyes derived from plants, barks, roots and animals not only imbue textiles with vibrant colours but also carry profound cultural significance.

In antiquity, it’s been documented that people used various powered mixtures of coloured minerals on their hair and body for what they believed were magical power during hunting and sometimes for adornment. (Yusuf et. al.,2017).

2. **Materials and Methods** :

GEOGRAPHY OF PLANTS:

1. Scientific name: Berberis napaulensis

Family: Berberidaceae

Common name: Nepal Mahonia

Local name: Ntho

The bark from the ‘Ntho’ tree in the Angami dialect is collected from ‘Lievagei’ in Jotsoma village, Kohima Nagaland. After collecting, it is dried and crushed then boiled with thread to create traditional clothing, with a yellow dye. Angami tribe usually use this bark technique to dye their mekhala/shawl for their traditional clothing. This bark is also utilized to dye the headgear worn by Angami men. Additionally, it serves as a material for other garments according to preference. The bark also helps inflammation, digestion and is topically used for skin conditions.




Fig:1(a)-Berberis napaulensis Fig:1(b) -Bark of Berberis napaulensis

Scientific name: Myrica esculenta
Family: Myricaceae
Common name: Bayberry
Local name: Pezie

This bark known as ‘Pezie’ in the Angami dialect, is sourced from a place called ‘Lievagei’, located in Jotsoma Village, Kohima Nagaland. In traditional practices, the bark of Myrica esculenta is harvested and processed to create natural dyes. These dyes, derived from the bark, are rich in color and are commonly used to dye fabrics and textiles. The process involves extracting pigments from the bark through various techniques, which are then applied to the materials, imparting vibrant hues. This method of natural dyeing has been passed down through generations and remains an integral part of cultural and artisanal practices. The bark is also employed for treating diarrhea.


Fig:2(a)-Myrica esculenta Fig:2(b)-Bark of Myrica esculenta

Scientific name: Castanea crenata

Family: Fagaceae

Common name: Japanese chestnut

Local name: Thezhü

The bark obtained from the tree known as ‘Thezhü’ in the Angami dialect, is found in Lievagei at Jotsoma Village, Kohima Nagaland . Once harvested, this bark serves various purposes, primarily as a key ingredient in creating natural dyes. These dyes are then utilized in diverse traditional practices for colouring different materials, including traditional clothing worn in Nagaland. The vibrant hues derived from these natural dyes play a significant role in adorning Nagaland's traditional costumes, adding cultural significance and reflecting the rich heritage and artistic traditions of the region. This method has been employed by villagers since ancient times for dyeing clothes, representing a cherished tradition within the community.

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Scientific name: Clitoria ternatea

Family: Fabaceae

Common name: Butterfly pea

Local name: Aparajita (India)

The Clitoria ternatea, is also known as butterfly pea, originates from tropical Asia and is renowned for its vibrant blue hue, making it a popular choice for natural dyeing. Its anthocyanin-rich petals are responsible for this distinctive colour, which is utilized in dyeing food, beverages, and textiles, including traditional clothing. In Nagaland, butterfly pea is widely embraced as a natural dye, where its vivid blue pigment extracted from the petals is used to colour an array of traditional textiles worn by different tribal communities. These textiles, often include shawls, mekhala and other attire worn for cultural ceremonies and festivals.

Butterfly pea, holds a significant place in Ayurvedic medicine, with various plant parts used to treat indigestion, constipation, arthritis, skin conditions, liver issues, and intestinal problems.

Fig:4- Clitoria ternatea

3. Methodology

3.1 Extracting natural dyes from organic sources

Various extraction methods were developed for these natural dyes, including aqueous, alkali, and solvent extraction. Each of these methods has its own benefits and limitations, which are influenced by the specific parameters required for the extraction process.

3.1.a Aqueous Extraction: This procedure involves crushing or grinding dye-containing materials or plant samples into smaller pieces using a mortar and pestle. The crushed sample is then placed in a beaker, and distilled water is added in a ratio of approximately 3-4 times the sample amount. The mixture is boiled for around 15-20 minutes and promptly filtered either with filter paper and funnel or by using a pump. The resulting filtrate is boiled until it reduces to half its original volume and then stored for dyeing cotton material. The remaining residue is left to dry. In this instance, 45-50 grams of the plant sample were utilized.

3.1.b Alkali extraction This method resembles the aqueous extraction method involving crushing the plant sample in a mortar and pestle and boiling it for approximately 15 to 20 minutes. However, in this variation, a 2N NaOH solution (containing 80 grams in 1 liter of water) is utilized at a ratio of about 3 to 4 times the sample amount instead of distilled water. After filtration, the resulting filtrate undergoes further boiling until it reduces to half its original volume, and it's then prepared for dyeing cotton fabric. In this instance, 45 to 50 grams of the sample were employed.

3.1.c Alcoholic Extraction: The solvent extraction procedure employs the Soxhlet apparatus, where the dried plant sample, typically around 10 grams, is enclosed in filter paper and placed within the siphon tube. Methanol is employed as the solvent, filling the round-bottom flask of the apparatus to approximately 250 ml, situated at the base. The Soxhlet apparatus operates for approximately six hours to ensure thorough extraction

3.2 Mordants Application Techniques

Natural dyes, with limited adherence to fibers, necessitate the use of mordants to enhance colourant fixation through complex formation. Key mordants include alum, potassium dichromate, ferrous sulfate, copper sulfate, zinc sulfate, tannin, and tannic acid. While these metal mordants offer a diverse range of hues when complexed with natural colour compounds, their toxicity necessitates minimal use for safety. Derived from the Latin word "*mordere*," meaning *"to bite,"* a mordant is a chemical that can bind to fibers and form chemical bonds with natural colourants. It aids in dye absorption and fixation, improving colour fastness and preventing bleeding and fading.

3.3 Methods of Mordanting

Complex formation can occur through pre-mordanting, meta-mordanting, or post-mordanting processes

3.3.a Pre-Mordanting: In this method, the cotton fabric undergoes a multi-step procedure. Firstly, it is boiled with around 100 ml of the mordant for roughly thirty minutes, then taken out and allowed to dry. Once the mordanted fabric is dry, it is submerged into the lukewarm extracted dye solution for approximately 10 minutes. Afterward, it is removed and left to air dry in the sun. This process effectively imparts the desired dye onto the fabric.

3.3.b Meta -Mordanting: This method involves preparing and combining the natural dye and mordants simultaneously, soaking the fabric in this combined solution. While simpler than pre-mordanting or post-mordanting processes, it's only suitable for a selected few dye.

3.3.c Post-Mordanting: In this method, the cloth material is initially soaked in the extracted dye and boiled for approximately thirty minutes before air drying. Afterward, the dried cloth is immersed in the mordant solution and boiled for another half an hour before being sun-dried.

This process results in obtaining the desired product.

3.3.d Preparation of mordants: Ferrous Sulphate (10g in 100ml) , Aluminum Sulphate (10g in 150 ml) and Potash Alum (10g in 100ml) in distilled water were used as mordants.

4. Results and Discussion:

Table-1: Effect of mordants on Berberis napaulensis dye

Extraction Process	Mordants	Clour obtained on cloth with mordants	pre-mordant and extracted dye	post-mordant and extracted dye
Aqueous	FeSO4	Citrine yellow	Honey yellow	Pear yellow
	Al_2(SO4)	Honey yellow	yellow green	Corn yellow
	$[KAl(SO_4)_2 12H_2O$	Greenish yellow	Bright yellow	Pear yellow
Alkali	FeSO4	Brownish orange	Light brown	Ginger
	Al_2(SO4)	White clay	White brown	Brown
	$[KAl(SO_4)_2 12H_2O$	Clay	White clay	White clay
Alcoholic	FeSO4	Honey yellow	Amber yellow	Clay yellow
	Al_2(SO4)	Honey yellow	Dark golden	Bright yellow
	$[KAl(SO_4)_2 12H_2O$	Honey yellow	Dark yellow	Bright yellow

Table- 2: Effect of mordants on Myrica Esculenta dye

Extraction Process	Mordants	Clour obtained on cloth with mordants	pre-mordant and extracted dye	post-mordant and extracted dye
Aqueous	$FeSO4$	Grayish brown	Dark brown	Charcoal
	$Al_2(SO4)$	Wood brown	Brown cream	Dark brown
	$[KAl(SO_4)_2 12H_2O$	Grey	Medium brown	Burlywood
Alkali	$FeSO4$	Dark brown	Brown	Dull brown
	$Al_2(SO4)$	Medium brown	Brown	Brownish grey
	$[KAl(SO_4)_2 12H_2O$	Brown	Dark brown	Medium brown
Alcoholic	$FeSO4$	Brown grey	Light brown	Brownish grey
	$Al_2(SO4)$	Light brown cream	Wood brown	Light brown
	$[KAl(SO_4)_2 12H_2O$	Brown cream	Wood brown	Brown cream

Table- 3: Effect of mordants on Castana crenata dye

Extraction Process	Mordants	Clour obtained on cloth with mordants	pre-mordant and extracted dye	post-mordant and extracted dye
Aqueous	$FeSO4$	Charcoal	Ink black	Denim blue
	$Al_2(SO4)_3$	Brown cream	Wood brown	Brown cream
	$[KAl(SO_4)_{2.}12H_2O$	Brown cream	Medium brown	Wood brown
Alkali	$FeSO4$	Medium brown	Denim greenish blue	Charcoal
	$Al_2(SO4)$	Brown cream	Brown cream	Brown cream
	$[KAl(SO_4)_{2.}12H_2O$	Brown cream	Brown cream	Light brown cream
Alcoholic	$FeSO4$	Charcoal grey	Charcoal grey	Light brown cream
	$Al_2(SO4)_3$	Light brown cream	Light brown cream	Brown cream
	$[KAl(SO_4)_{2.}12H_2O$	Light brown cream	Brown cream	Light brown cream

Table- 4: Effect of mordants on Clitortia ternatea dye

Extraction Process	Mordants	Clour obtained on cloth with mordants	pre-mordant and extracted dye	post-mordant and extracted dye
Aqueous	$FeSO4$	Light Coffee	Coffee	Light coffee
	$Al_2(SO4)$	Pink	Maroon linen	Pink
	$[KAl(SO_4)_2 12H_2O$	Pink	Cherry pink	Dark cherry pink
Alkali	$FeSO4$	Light brown	Light golden	Light brown
	$Al_2(SO4)$	Yellow	Light brown	Light brown
	$[KAl(SO_4)_2 12H_2O$	Yellow	Yellow	Light brown
Alcoholic	$FeSO4$	Brownish grey	Dark brown	Brownish grey
	$Al_2(SO4)$	Brown cream	Coral pink	Coral silk
	$[KAl(SO_4)_2 12H_2O$	Brown cream	Greenish brown	Greyish brown

Table-5(a) : Functional group test for Berberis napaulensis and Myrica esculenta

Functional group	Results corresponding to extract (Berberis napaulensis)			Results corresponding to extract (Myrica esculenta		
	Aqueous Extraction	Alkali Extraction	Alcoholic Extraction	Aqueous Extraction	Alkali Extraction	Alcoholic Extraction
Carboxylic acid	Absent	Absent	Absent	Absent	Absent	Absent
Alcohol	Present	Present	Present	Absent	Absent	Present
Phenol	Absent	Present	Absent	Absent	Present	Absent
Carboxyl	Absent	Present	Absent	Present	Absent	Absent
Amino	Present	Absent	Present	Absent	Present	Absent

Table- 5(b) : Functional group test for Castanea crenata and Clitoriaternatea

Functional group	Results corresponding to extract (Castanea crenata)			Results corr esponding to extract (Clitoriaternatea)		
	Aqueous Extraction	Alkali Extraction	Alcoholic Extraction	Aqueous Extraction	Alkali Extraction	Alcoholic Extraction
Carboxylic acid	Absent	Absent	Absent	Absent	Absent	Absent
Alcohol	Absent	Present	Present	Present	Absent	Present
Phenol	Present	Present	Absent	Present	Present	Present
Carboxyl	Absent	Present	Absent	Present	Present	Absent
Amino	Present	Absent	Absent	Absent	Absent	Absent

Table -6(a) : Phytochemical test for Berberis napaulensis and Myrica esculenta

	Berberis napaulensis			Myrica esculenta		
	Aqueous	Alkali	Alcohol	Aqueous	Alkali	Alcohol
Alkaloids	+++	_	+++	+	_	_
Steroids	++	_	+	+++	+	+++
Flavonoids	_	_	_	_	_	+
Tannins	_	_	_	+++	_	+++
Glycosides	_	+++	_	+	+	+
Terpenoids	_	++	_	+++	++	_
Saponins	+++	+++	_	+++	+++	_

Table-6(b) : Phytochemical test for Castanea crenata and Clitoriaternatea

	Castanea crenata			Clitoriaternatea		
	Aqueous	Alkali	Alcohol	Aqueous	Alkali	Alcohol
Alkaloids	_	+	_	_	_	_
Steroids	+	_	++	+++	_	++
Flavonoids	_	_	_	+	+	_
Tannins	+++	_	+++	+	_	+
Glycosides	+++	++	+++	_	+	_
Terpenoids	+	+	+++	_	+	_
Saponins	+++	+++	+++	+++	+++	_

Quantitative Analysis of Berberis napaulensis

Peak list:

No.	WL.(nm)	Abs	T%
1	238.0	2.651	0.22
2	244.0	2.593	0.26
3	248.0	2.540	0.29
4	253.0	2.497	0.32
5	259.0	2.425	0.38
6	271.0	2.283	0.52
7	288.0	2.176	0.67
8	360.0	1.614	2.43
9	431.0	2.118	0.76

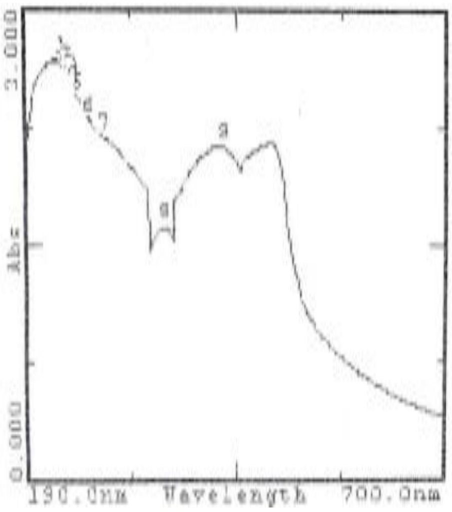


Fig:6- UV spectroscopy of Berberis napaulensis

Interpretation (IR)

Alcohol - 2916 (2500-3000)

Amines - 1020 (1000-1250)

Alkanes - 2916 (2850-3000)

Alkenes – 1642 (1600-1650)

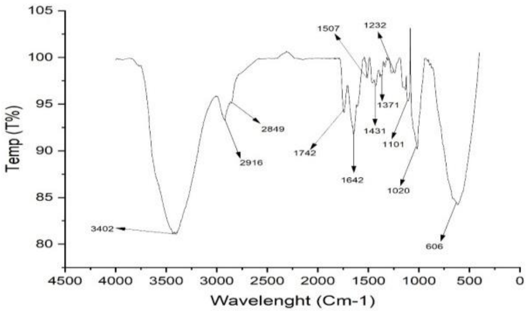


Fig:-7. IR spectroscopy of Berberis napaulensis

Quantitative Analysis of Myrica esculenta

Peak list:

No.	WL.(nm)	Abs	T%
1	233.0	2.633	0.23
2	239.0	2.619	0.24
3	242.0	2.583	0.26
4	249.0	2.515	0.31
5	278.0	2.220	0.60
6	289.0	2.158	0.69
7	314.0	2.013	0.97
8	359.0	1.594	2.55
9	435.0	2.067	0.86
10	557.0	2.462	0.34
11	589.0	2.583	0.26
12	594.0	2.536	0.29
13	601.0	2.472	0.34
14	608.0	2.410	0.39
15	620.0	2.337	0.46
16	629.0	2.290	0.51

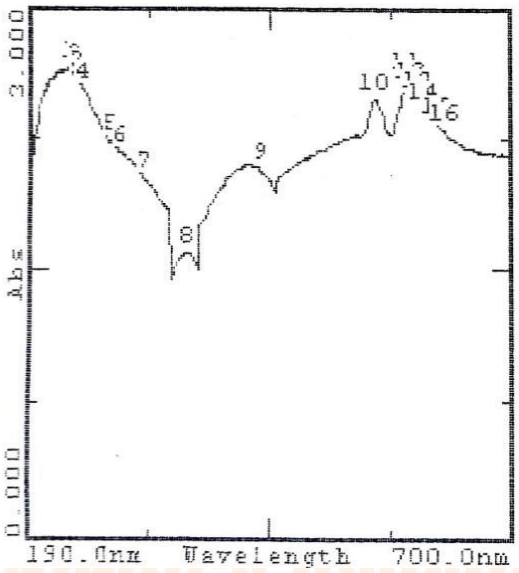


Fig :8-UV spectroscopy of Myrica esculenta

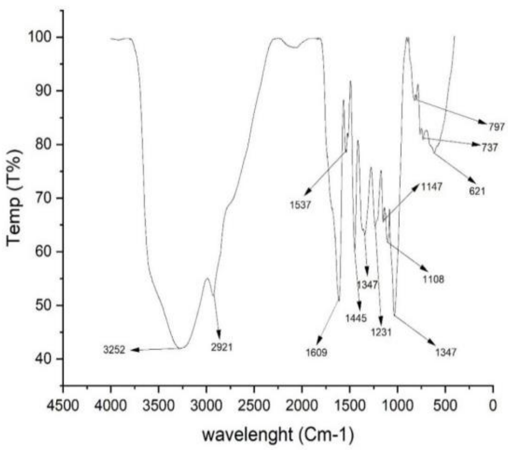


Fig:9- IR spectroscopy of Myrica esculenta

Interpretation

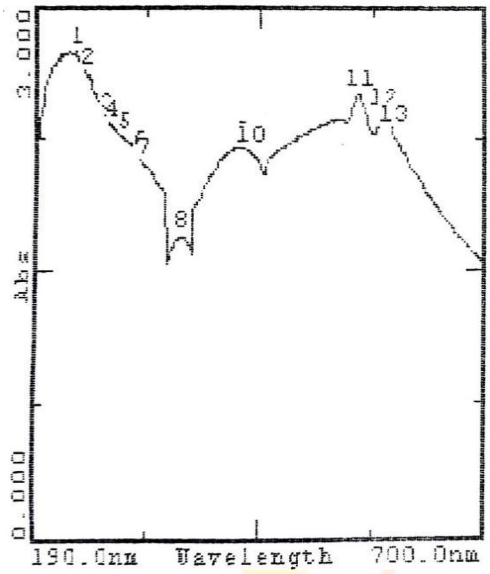
Phenol – 3252 (3200-3550)

Amines – 1108 (1000-1250)

Alkanes – 2921 (2850-3000)

Alkenes – 1609 (1600-1650)

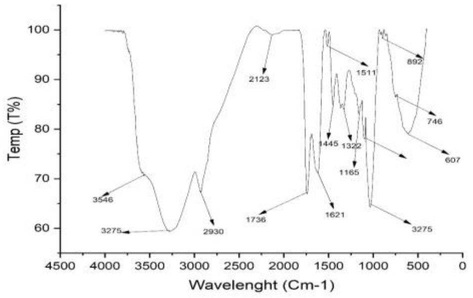
Quantitative Analysis of Castanea crenata



Peak list:

No.	WL(nm)	Abs	T%
1	239.0	2.712	0.19
2	251.0	2.598	0.25
3	274.0	2.352	0.44
4	280.0	2.308	0.49
5	294.0	2.239	0.58
6	312.0	2.133	0.74
7	316.0	2.098	0.80
8	359.0	1.688	2.05
9	428.0	2.192	0.64
10	437.0	2.157	0.70
11	559.0	2.484	0.33
12	586.0	2.375	0.42
13	596.0	2.286	0.52

Fig :10- UV spectroscopy of Castanea crenata



Interpretation

Phenol - 3275 (3200-3550)

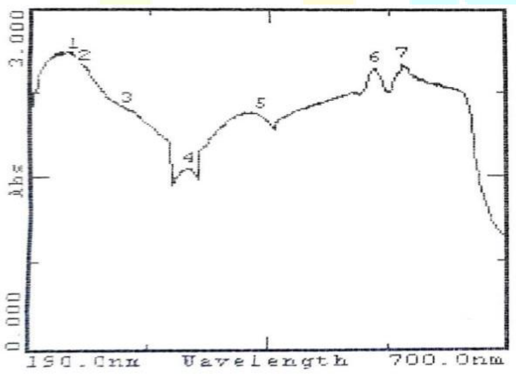
Amines - 1165 (1000-1250)

Alkanes - 2930 (2850-3000)

Alkenes – 1621 (1600-1650)

Fig:11- IR spectroscopy of Castanea crenata

Quantitative Analysis of Clitoriaternatea



Peak list:

No.	WL(nm)	Abs	T%
1	240.0	2.568	0.27
2	251.0	2.466	0.34
3	296.0	2.098	0.80
4	361.0	1.563	2.74
5	435.0	2.041	0.91
6	559.0	2.447	0.36
7	588.0	2.487	0.33

Fig:12- UVspectroscopy of Clitoria ternatea

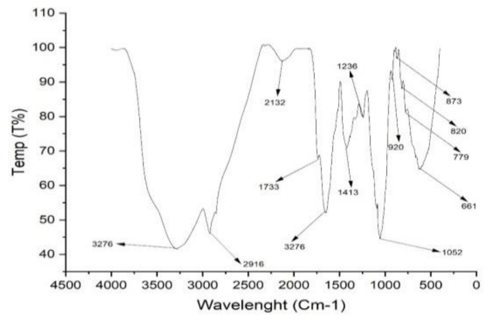


Fig :13- IR spectroscopy of Clitoriaternatea

Interpretation

Alcohol - 3276 (3400-3200)

Phenol - 3276 (3200-3550)

Amine- 1052 (1000-1250)

5. Discussion:

SAMPLE 1:

The cotton fabric yielded a dark yellow hue after extracting the plant dye through an aqueous method. On aqueous and alkali extraction, the predominant colour observed was vibrant yellow, with or without the application and various mordants. However, during alkali extraction, some brown colouration was also evident.

SAMPLE 2:

The cotton fabric obtained a light brown hue following the plant dye extraction via an aqueous method. The Myrica esculenta dye yielded various shades of brown including grey and charcoal on different cloth samples, with brown being the predominant colour. Nevertheless, solvent extraction proved less effective compared to alkali and aqueous extraction methods.

SAMPLE 3:

The cotton fabric achieved a brown hue through the extract of plant dye using an aqueous technique. The ferrous sulphate produced satisfactory darker tones like denim blue, charcoal, and brown across all the extractions. On the other hand, aluminium sulphate and potash alum mainly resulted in lighter shades of brown.

SAMPLE 4:

The cotton cloth obtained a light blue hue following the plant dye extraction via an aqueous method. The Clitoriaternatea dye produced different shades of brown, pink and some yellow with varying vibrancy on different cloth samples, with brown being the predominant colour.

6. Conclusion:

In summary, this study extensively explored the extraction of natural dyes from diverse plant sources and their application with various mordants and fibers to achieve a wide array of colours, illuminating the importance of natural dyes both ecologically and culturally, highlighting their role in supporting biodiversity, preserving traditional knowledge, and strengthening the bond between humanity and the natural world. In natural dyes, it's imperative to tackle key challenges such as scalability, standardization, and accessibility. Through the integration of modern technology, biotechnology, and interdisciplinary collaboration, one can uncover new pathways to enhance the quality, efficiency, and sustainability of natural dye production and usage. Thus, this ability highlights the potential of natural dyes as sustainable alternatives to synthetic options offering not only vibrant hues but also environmental and cultural advantages. 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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