

## SUSTAINABLE APPROACH FOR SYNTHESIS OF NATURAL DYE FROM DRIED FLOWER WASTE OF *AZADIRACHTA INDICA*

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### ABSTRACT

The different parts of *Azadirachta indica* has significant medicinal values in Ayurveda field. They are commercially used for synthesis of different therapeutic drugs in pharmaceutical sector. In this study, dried flowers of *Azadirachta indica* RUBL21378 has been used for synthesis of natural dye. The phytochemical screening was performed in different extract of their dried flower in organic solvents like methanol, ethanol, and acetone. These phytochemical screening had confirmed the presence of different bioactive components like saponins, quinone, flavonoids, and alkaloids. They showed specific antimicrobial property with effective zone of inhibition 0.5mm and 16mm against *Escherichia coli* and *Staphylococcus aureus* respectively. Copper sulphate was added as mordant during incubation period of dyeing process of cotton fabrics with flower extract. These fabrics exhibited a significant level of natural dye absorption at 50°C after incubation period up to 3 hours. The colour fastness was observed after treating them with sunlight at different interval of time period for 24, 48, and 72 hours. These fabrics showed stability in detergent solution (2%) for 30 minutes at 50°C. These fabrics showed equal colour intensity after dyeing with green tea as mordant. Their color intensity was increased after dyeing with flower extract of *Acacia catechu* as bio mordants. The functional groups are investigated by FTIR spectrum analysis. This methodology for synthesis of natural dyes could be efficiently employed in textile industry for colouring cotton fabrics at large scale.

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### INTRODUCTION

The majority of ingredients used in textile manufacturing are synthetic color made from petroleum-based petrochemicals, which are neither biodegradable nor renewable. [25]. It was predicted that the world's textile consumption would require 30 million tonnes of dye, with annual growth of up to 3%. Meanwhile, 70,000 tonnes of dyes were dumped into the environment. [1]. According to a business week, the percentage of the population that is allergic to chemicals will increase to 60% by 2020 [2]. [25] In addition, approximately 280,000 tons of textile dyes are discharged annually worldwide and approximately 10,000 different dyes have been produced and an estimated 8x10<sup>5</sup> tons of synthetic dyes are used in the textile industry worldwide [25,13]. Natural dyes become an alternative way to the industries when synthetic dyes caused

severe problems such as visible residues in the effluent [13] and toxic amines were created. [31]. On the other hand, natural dyes can return to nature at the end of use, such as wastewater from dyeing process. Therefore, researchers are interested in natural dyes because it is more compatible with the environment and has an antibacterial and ultraviolet protection functions [31],[26]. Synthetic dye production involves a large number of cancer-causing synthetic chemicals and effluents that are released into the water or produced into the air resulting in contamination. Besides that, interest in the usage of natural dyes has been revived worldwide due to the toxic nature and effect of synthetic dyes on all forms of life. Cotton fabrics have cellulose and abundant hydroxyl groups structure where it will make hydrophilic properties where the fiber can provide an appropriate environment for microorganism growth when in contact with water and sweats. Besides that, cellulose as 94%, 1.3% protein, 0.9% pectin, 1.2% oil, 0.6% wax and 2% other substances are the general composition of cotton fibers. Cotton fabric is widely used in various application due to their biodegradability, renewability and environmentally friendly where it does not contain any harmful composition. [4]. Dyes

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are used for imparting colour to an infinite variety of materials like textiles, paper, wood, varnishes, leather, ink, fur, foodstuff, cosmetics, medicine, etc. Dyes are broadly categorized into two types namely; synthetic dyes obtained from chemical substance or derived through chemical process and natural dyes obtained from natural sources. The term Natural dyes covers all the dyes derived from natural sources such as plants, insects and minerals [7].

The Sanskrit word “nimba” means (Nimbati sincati svasthyamiti, svastha vrtikaramiti yavat) that which gives health, [9]. The neem tree is an extraordinary plant that has been proclaimed by the United Nations as the ‘Tree of the 21st Century’. The neem twigs are used as tooth brushes as it possess prophylactic effect widely used by 500 million people of India alone [27][12]. Neem is used in Ayurveda, Siddha, Unani and Homeopathic medicine. In India, pharmaceutical chemists isolated margosic acid during 1919, an acidic principle in neem oil containing three active constituents, such as nimbin, nimbidin and nimbinene, since they were chemically identical and biogenetically derived from tetracycliterpenes[19]. The components are also referred to as liminoids as it contains azadirachtin, melianthol, (salanin etc., [5]. These compounds also exhibit a wide range of biological activity. Medicinal uses of neem Neem has more than 140 compounds isolated from different parts of the tree [28]. The parts of neem such as leaves, flowers, seeds, fruits, roots and bark have been used traditionally in treating inflammation, infections, fever, skin diseases, dental disorders and removing the evil effects of spirits. Quercetin (flavonoid) and nimboesterol ( $\beta$ -sitosterol) are present in the leaves, as is of the liminoids (nimbin and its derivatives). Quercetin is a polyphenolic flavonoid that tends to have antibacterial and antifungal properties and possess curative effects for sores and scabies [24]. New matured leaves develop an odorous viscous essential oil that exhibits fungal antifungal activity against Trichophyton mentagrophytes at invitro conditions [14]. Proteins (7.1 percent), carbohydrates (22.9 percent), minerals, calcium, phosphorus, vitamin C, carotene, etc. are the main constituents of neem leaves, [3]. Amino acids such as glutamic acid, tyrosine, aspartic acid, alanine, praline, glutamine, cystine and other fatty acids such as dodecanoic acid, tetradecanoic acid, elcosanic acid, etc. are present.

There are several amino acids in the neem pollen, including glutamic acid, tyrosine, arginine, methionine, phenylalanine, histidine, arminocaprylic acid, and isoleucine, [17]. Nimbin (0.04 percent), nimbinin (0.001 percent), nimbidin (0.4 percent), nimboesterol (0.03 percent), essential oil (0.02 percent), tannin (6.0 percent), bitter margosin and 6-desacetyl nimbinene are found in the trunk bark [23,24]. The stem bark is rich in tannins (12-16 Percent) and non-tannin (8- 11 Percent). The bark contains anti-inflammatory polysaccharide consisting of glucose, arabinose and fructose at a molar ratio 1:1:1 with molecular weight of 8,400, [6]. The bark also yields an antitumor polysaccharide [21,20]. Stem bark and root bark yields diterpenoids such as nimbinone, nimbolicin, margocin, nimbidiol and nimbine. Though heartwood contains iron salts, potassium and calcium, heartwood provides charcoal (30 percent) and pyroligeneous acid (38.4 percent) by the process of destructive distillation. [10]. Neem wood contains cellulose, hemicellulose (14.00%) and lignin (14.63%),

while wood oil contains  $\beta$ -sitosterol, cycloeucaenol and 24-ethylenecycloartenol[16]. The gum exuded by the tree on hydrolysis yields, D-glucuronic acid, Larabinose, D-galactose and L-fucose as the older tree exudes a sap which has free sugars such as glucose, fructose, mannose and xylose and amino acids includes asparagines, praline, alanine, aminobutyric acid, arginine, aspartic acid, glycine, norvaline and organic acids viz., citric, malonic, succinic and fumaric acids. [10]. The sap is reported to be useful in the treatment of general weakness and skin diseases. Seed is very important both because of its high lipid content and because of the presence of a large number of bitter principles that include, in substantial amounts, azadirachtin, azadiradione, fraxinellone, nimbin, salannin, salannol, vepinin, and vilasinin. Azadirachtin has been shown to be effective against around 200 insect species as a pesticide and is confirmed to be nontoxic to humans [18]. Neem kernel lipids are similar to the normal glycerides from other oilseeds and contains oleic acid (50-60 percent), palmitic acid (13-15 percent), stearic acid (14-19 percent), linoleic acid (8- 16 percent) and arachidic acid (1-3 percent) [24]. Neem kernel oil is brownish yellow, non-drying oil with unpleasant odour and acid taste. The oil content varies with the processing system. Depending on the raw material used for expulsion, the composition of neem cake after oil extraction varies widely. The range of the proximate composition in neem leaves includes crude protein 13-35, carbohydrates 26-50, crude fibre 8-26, fat 2-13, ash 5-18, acid insoluble ash 1-7 [19]. As feed for animals or poultry, the bitter cake has no appeal. Cake extraction followed by hexane with 70 percent alcohol produces a meal free of bitterness and odor that will be adequate as feed. Neem cake is rich in amino acids. It is one of the potential organic manure source which was rich in plant nutrients, including 2-3 percent nitrogen, 1.0 percent phosphorus and 1.4 percent potassium, 1.0-1.5 percent tannic acid and sulphur content of 1.07-1.36 percent[24]. The bark of neem, leaves, fruits, flowers, oil, everything (bark, leaves, fruit, flower and oil) combination. Researchers reported that tender neem leaves are effective against parasitic infections and anti-viral properties have been discovered in a 10 percent of aqueous extract dosage. Plasma coagulation period tests using Russel’s viper venom have shown that a clotting agent is present in the leaf extract which was used in treating poisonous bites. [8]. Neem leaves total extract is a potent hepatoprotective agent, as neem leaves water extract showed significant antiulcer activity, while mild fungicidal action was observed in fresh leaves essential oil, significant anti-inflammatory effect, decreased frequency and severity of gastric mucosal lesions induced by stress. The extracts of neem leaf, bark and seed on intraperitoneal administration revealed immunostimulatory properties which are responsible for its anti-HIV effect. Among more than 100 compounds found in Neem (Neem seed kernel) [27]. Twelve Azadirachtin compounds are identified while all of them shows high biological activity levels. Azadirachtin in only single low dose was reported to have immunized a transmission of Chagas disease from the kissing bug. Azadirachtins have been shown to inhibit the larval, pupal and adult moults and reproduction and fitness of both plantfeeding and aquatic larvae such as mosquitoes. It has been shown that Gedunin, contained in whole fruit possess antimalarial activity. The antipyretic and non-irritant, nimbidin found in neem bark proved to be effective in



treating of skin diseases such as eczema, furunculosis, arsenic dermatitis, burn ulcers, Herpes labialis, scabies and seborrheic dermatitis [27]. It is also effective in treating skin diseases, such as warts, dandruff and skin diseases of unknown origin. Bark extracts have potent properties that are diuretic and anti-inflammatory. Spermicidal activity is reported to include nimbidin and sodium nimbidinate found in neem bark. Neem Bark has demonstrated antibacterial activity against different species that are gram-positive. Nimboesterol and flavonoids such as kaempferol, melicitrin are present in flowers which are one of the essential oils consisting of sesquiterpene derivatives while flowers yield a waxy material which contains fatty acids such as behenic (0.7 percent), arachidic (0.7 percent), stearic (8.2 percent), palmitic (13.6 percent), oleic (6.5 percent) and linoleic (8.5 percent), [22]. There is no work publication or study found on natural dye production from neem dried flower. In this study we collect the dried flower of neem plant for the production of natural dye.

## METHODS MATERIALS AND METHODS

### Selection of Fibre Cotton

Cotton plant (*Gossypium hirsutum*, L.) belongs to Family Malvaceae. The cotton blossom, which appears about hundred days after plants are a beautiful creamy white or light-yellow flower.

### Selection of Mordants

Copper sulphate, iron sulphate and green tea as a bio mordant was used as mordants for the dye. To help dyes bind better to fibres, dyes typically use different mordants, most of which are salts of metals like aluminium, iron. The word mordant actually comes from the Latin word mordere, meaning "to bite"; because early dyers thought these substances enabled the dye to get a better bite on fibres. (Sundari, 2015; Pervaiz et al., 2016). Selection of Dyes Flowers of neem were collected and dried. Then it was powdered. Before extraction of natural dyestuff, the samples were dried at room temperature. After cleaning and drying, the samples were powdered by grinding machine.

### Preparation of Raw Materials

Dyeing in Cotton Dye powder is weighed. Cotton fibre was soaked in distilled water and ionic detergent solution containing two gram per litre each of soap in soda ash at 80°C for 1 hour to remove starch. The material to liquor (M: L) ratio was maintained at 1:30, after which fabric was washed with distilled water, dried and iron. Prior to dyeing or mordant the fibre sample were soaked in water for half an hour. Mordant used as copper sulphate, iron sulphate and green tea. In case of cold-water extract, the powder was soaked in water and kept for 48 hours. The resultant liquid was then filtered. The extracted dye solutions were then concentrated and then dried in hot air circulatory oven at 60-70°C dry material is obtained. The dry material is then removed from trays. The solid mass so obtained was then subjected to extraction with 70:30, alcohol: water mixture and then filtered. Then filtrate was evaporated under reduced pressure to get concentrated mass of colour component. For dyeing experiment cotton fibres were pre-treated with distilled water at 80°C for 30 minutes. The fibre was then dyed bath containing each of dye extracted from flower. The dyeing was carried out at 97-98°C for 45 minutes

in which 2% sodium chloride solution basis of material was added to bath and the system was further heated for 15 minutes. Then the fibres were washed thoroughly and with distilled water and dried at room temperature. Mordanting was carried out for 30 minutes at 90-95°C.

### Phytochemical Investigation of Neem flower

The preliminary phytochemical tests were carried out to study the main compounds which are present or absent in flower. Tests for alkaloids, polyphenol, flavonoids, glycosides, phenolics, saponins, lipophilics, and tannin were performed according to the methods and procedures prescribed in the [36].

### Antimicrobial activity

#### Antibacterial Activity of Extracts

The agar well diffusion technique was used to determine the antibacterial activity of neem extracts. The plates were incubated at 37°C for 24 hours and observed for the zone of inhibitions. The antibacterial activity of flower extract of neem was expressed as the mean diameter of zone of inhibition (mm) against growth [35].

### Mordanting methods

#### Pre- mordanting Method

In this method, mordant solution was prepared by heating 1.25g alum with 200 ml water to obtain a volume of material to liquor ratio of 10:200. This solution was heated to a temperature of 80°C and 10g cleaned fabric was simmered in mordant solution for about 45 minutes. During mordanting, the fabric was frequently stirred to obtain good penetration of mordants into the materials. After that, the fabric was rinsed with water. After mordanting, the dye liquid prepared was added to a beaker to obtain material to liquor ratio 10:200 and heated at 80°C. Then the mordanted fabric was simmered in this solution for about 30 minutes. Then the fabric was rinsed with water and allowed to air dry. The same experiment was carried out with different mordants such as CuSo4 and FeSo4 and bio mordant [32], [35]

#### Post - mordanting Method

After dyeing, mordanting bath was prepared by heating 1.25 g alum with 200ml water to obtain material to liquor ratio of 10: 200. This solution was heated to 80°C and 10g cleaned fabric was simmered in it for 45 minutes. After that, similar experiments were carried out as pre-mordanting method. Different mordants such as CuSo4 and FeSo4 were used for dyeing process.

#### One pot Dyeing and Mordanting Method

The dye liquid was added to a beaker containing 1.25 g alum to make a volume of material to liquor ratio 10:200. Then, the dye bath was heated at 80°C and 10g cleaned fabric was simmered in that solution for about 30 minutes. After that, the fabric was rinsed with water and allowed to air drying. The same experiment was carried out with different mordants such as CuSo4 and FeSo4 and bio mordant [34].

We used pre-mordanting method for our all mordanting experiments.



### Testing the Colour Fastness of Dyed Fabrics

After dyeing, testing for colour fastness of dyed fabrics was carried out. In the dyeing process, fastness of textile substrates to the effects such as light, washing, and rubbing were determined. Launder Meter, Model L-4 (4-rack testing bottle) washing machine was used and Test No.3 of ISO 105 was used to assess the colour fastness to washing. 2g/litre of soap solution was added to the jar a material to liquor ratio of 1:50 and the soap solution was preheated at 60°C. Then, the composite sample was placed in the jar and treated at 60°C for 30 minutes. After washing, the specimen was rinsed with water for (3) times and dried at room temperature. For the test of the colour fastness to rubbing, a Crock Meter, JIS.L 0823 / 0849 with a rubbing finger, comprising a cylinder of 1.6 cm diameter moving to and fro along a straight line of 10 cm track on the specimen with a load of 900 g was used. An outdoor exposure cabinet was used for light fastness test. The cabinet consists of a glass-covered enclosure of wood to protect the specimens from rain and weather. The dyed fabrics measuring (2 in × 1.5 in) of the material to be tested were cut out. These specimens were placed on the rack and the cabinet was covered. Then the specimens were exposed on sunny days between the hours of 9 a.m. and 3 p.m. for 7 days. The change in colour is assessed by comparing with the original dyed fabrics.

### Dyeing Fastness

The fastness properties of fabrics were tested by washing the dyed cotton fabrics thrice with warm detergent solution. The characterization of dyed fabrics by FTIR confirmed the presence of sulfoxide group from metal mordant and -OH (hydroxyl), -COOH (Carboxyl), keto, aromatic ring from natural dye extractant. These functional groups were bound to cotton fabrics either by hydrogen bond, covalent and van der Waals bond.[32,33].

## RESULTS AND DISCUSSION

In this work, neem blossoms were gathered, and bioactive components were determined using a phytochemical screening test in several organic solvents such methanol, ethanol, and acetone (Table 1). This test revealed a greater concentration of saponins, quinone, flavonoids, and alkaloids in the ethanolic extract. *Escherichia coli* (05mm) and *Staphylococcus aureus* (16mm) growth were effectively inhibited by this extract's antibacterial activity. Copper sulphate was used as a mordant throughout the one-hour incubation phase while cotton garments and unprocessed wool threads were coloured with floral extract (Fig1). Cotton textiles exhibited a significant level of natural dye absorption at 50°C. The impact of the incubation period was further investigated by changing the time period from 1 to 4 hours. Following a 3-hour incubation time at 50°C, the dye's hue diminished to a lesser amount. By subjecting them to sunlight for 24, 48, and 72 hours, the colour fastness of these colored materials was evaluated. A comparable level of hue intensity was seen over 72 hours. The impact of detergent was investigated by immersing raw wool threads and colored cotton textiles in a solution of Tide detergent (2%) for 30 minutes at 50°C. The stability of raw wool threads and cotton-dyed garments in detergent solution was greater as shown in Fig2. Using green tea and Acacia Catechu (RUBL20387) as bio-mordants and ferrous sulphate as a metallic mordant, researchers concurrently examined the

effects of additional mordants. Cotton textiles and raw wool threads were discovered to have equal colour intensity when dyed with green tea, and it increased when combined with Acacia Catechu (Babul flowers) (RUBL20387) bio mordants (Fig 3). On cotton fabric dyed with *Azadirachta indica* (Neem flower) [RUBL21378] and mordanted with Green Tea and Acacia Catechu (RUBL20387) extracts that were both metallic and non-metallic, analytical tests using IR spectrophotometry (FTIR) were carried out. The textile sector might effectively replace synthetic dye for cotton garments with natural dye by valorising dried neem blossoms. Therefore, this methodology is employed to first identify plants from which dyes can be extracted and to test the properties of the plant dyes and introduce them into the textile fabrics in a faster and effective manner.

Different shades of color dye were obtained from neem flowers. Mordants play very important role in imparting color to the fabric. With the use of copper sulphate brown coffee colour, ferrous sulphate the black coloured shade, dyeing with bio mordant light brownish color was obtained for cotton cloth. Strong co-ordination tendency of Cu and Fe enhances the interaction between the fiber and the dye, resulting in high dye uptake. The mordanted cotton cloth was immediately used for dyeing because some mordants are light sensitive. The chromophore in the dye makes the resistant to photochemical attack, but the auxochrome from dye may alter the fastness (Jothi, 2008). Cotton fibre and cotton cloth showed excellent dyeing. All the dyed fabrics were evaluated for their surface color reported in Table 4 & Table 5 and the results of assessment of colour fastness behaviour to light, washing and resistance are given in table 3 The treated samples subjected to light showed appreciable results with all different types of mordants of optimum concentration. The fastness test of cotton samples to rubbing under dry condition showed that the sample have fair to good fastness as compared to wet rubbing. Fastness towards perspiration has fair to good fastness to cotton when compared to raw wool. Washing Fastness test showed fair to excellent fastness to cotton when compared to raw wool. However, shows cotton good color fastness and bright color ranges while compared to wool. Dyeing absorbency of cotton with mordants is good while in raw wool dyeing absorbency is less when compared. A detail study on the neem flowers was carried out for the dye material concentration for raw wool and cotton.

In the extraction of natural dyes from neem flower, the phytochemical investigation of neem flower powder is tested and the results are shown in table (1). According to the results, are present in neem flower. Alkaloids, amino acids, quinone, flavonoids, carbohydrates, and saponin were found to be the primary phytochemicals in the extract according to the findings of the phytochemical screening.

*E. coli* and *Pseudomonas aeruginosa* were inhibited to the greatest extent by *Azadirachta indica* (RUBL21378) floral parts. Infections in wounds and diabetic foot ulcers are frequently caused by these organisms. These antimicrobial dyes will benefit patients as a result of their inclusion. Using the agar diffusion method, the blossom of the *Azadirachta indica* (RUBL21378) plant was tested for its ability to combat both gram-positive (*Bacillus cereus*) and gram-negative (*Escherichia coli* and *Pseudomonas aeruginosa*)



**Table 1** Phytochemical Analysis of flower extract of *Azadirachta indica* in different solvents

Constituent	Aqueous solvent	Ethanolic solvent	Methanolic solvent	Acetone solvent
Carbohydrate				
1. Molish's	+	+	+	+
2. Felling's	-	-	-	-
3. Benedict	+	+	+	+
Saponins				
1. Forth test	+	-	+	+
2. Foam test	+	+	+	+
Amino acid	+	-	+	-
Quinones	+	-	+	+
Flavonoids	+	-	-	-
Alkaloids				
1. Wagner's test	+	+	+	+

bacteria. The width of the growth inhibition zones varied from (mm), indicating the *Azadirachta indica* floral extract's (RUBL21378) strong activity against the tested bacterium strains. It had excellent effectiveness against *Pseudomonas aeruginosa* (mm), *E. coli* (mm), and *Bacillus cereus* (mm) at the highest dose tested out of the 4 cultures tested. (100 mg/ml). The flower's methanol extract was also able to exhibit some respectable efficacy against gram-positive rather than gram-negative organisms. In contrast to our findings, which showed that *E. coli*, P.A. has a highly sensitive antimicrobial activity, the ethanolic (flower) extract of *Azadirachta indica* (RUBL21378) significantly inhibited Gram-positive and Gram-negative bacteria.

Cotton fabrics were dyed with dye extracted from dried flowers of *Azadirachta indica* (RUBL21378) using copper sulfate, ferrous sulphate and green tea as mordant agent. Cotton fabrics was dyed differently with different mordanting agents. Green tea as bio-mordants showed more intensive color after dyeing the cotton fabrics in compare to control cotton fabric. The color intensity for fabrics had increased and observed maximum for pre-mordanting in compare to post-mordanting and meta-mordanting methods. From table 7, it can be seen that the trend of color strength is found to be different. Mordant dyed cotton samples have the different color strength owing to the interaction of dye and bio-mordant molecules in dye bath solution and low amount of dye interacts to Fiber. Green tea as a bio-mordant on the basis of % dye exhaustion, respectively. It can be observed that there is a gradual increase in % dye exhaustion of pre-mordanted cotton samples when the concentration of *Azadirachta indica* (RUBL21378) reached to 5%, and thereafter, a little increment occurred. Thus, 5 % bio mordant is believed to be the optimized mordant concentration for subsequent dyeing experiments with *Azadirachta indica* (RUBL21378) natural dye.

From the Results of Table (2), Cotton Fabrics Dyeing By Post-Mordanting Method Using Cooper Sulphate, Ferrous Sulphate and green tea as a bio mordant Are Most Suitable Because It Has A Change Of Shade 4-5 (Good-Excellent). Table (3) indicates the colour fastness results of dyed cotton fabrics mordanted with copper (II) sulphate. From the results, post-mordanting method is suitable because it has a change of shade 4 (Good) and staining on cotton fabrics of 4-5(Good-Excellent). In Table (4), colour fastness of dyed cotton fabrics

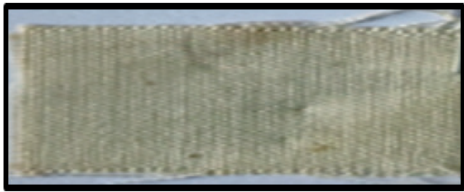
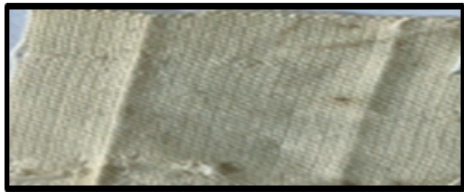

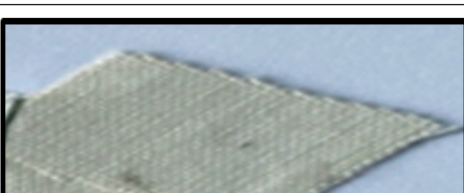
dyeing by post-mordanting method with lime gives better result. This is due to the observation of change of shade mark in grade 4 (Good) and wet rubbing grade of 4 (Good). According to the fastness results of dyed cotton fabrics, dyeing by post-mordanting method with alum is found to be most suitable due to change of shade mark in washing fastness. From the study of light fastness in Tables (2) to (4), the results show that using postmordanting with alum and copper (II) sulphate is more suitable than lime. The result is confirmed by the fact that post-mordanting with a metal salt can result in an improvement in the light fastness of some natural dyes [35].




**Table 2** Effect of different time period and temperature on colour extent of dyed cotton fibre

Temperature	Incubation Time	Colour Variation
1.30 °C	1 hour	Brown
2.50 °C	1 hour	Light brown
3.70 °C	1 hour	Creamy light brown
4.80 °C	1 hour	Creamy light brown
5.90° C	1 hour	Creamy light brown
6.100° C	1 hour	Creamy light brown


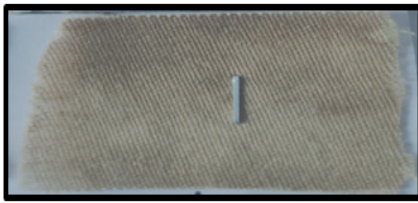

Spectroscopic examination using the Fourier transform infrared (FTIR) method Long known as a potent tool for illuminating structural information, Fourier transform infrared spectroscopy (FTIR) is a branch of spectroscopy. Clarifying conformational and environmental changes in polymers at the molecular level may be done by examining the location, strength, and shape of vibrational bands. In general, an organic molecule will absorb and convert infrared light into the energy of molecular vibration in the 10.000 to 100 cm<sup>-1</sup> range. Stretching and bending are the two primary molecular vibration modes. Stretching vibration is an irregular motion that moves in a repetitive pattern along the bond axis and increases or decreases the interatomic distance. The bond angle between bonds with the same atom. The FTIR spectra of the dyeing and mordanting of cotton fabric in the wavenumber range 4000-500 cm<sup>-1</sup> are shown in Figures 4-7. The FTIR-ATR spectra of raw cotton and dyed cotton using *Azadirachta indica* (RUBL21378) flower extract in presence of different mordants (CuSo<sub>4</sub>, FeSo<sub>4</sub>, Green tea as a bio-mordent).

**Table 3** Effect of temperature on color stability of dyed cotton fabrics with dye extracted from flowers of *Azadirachta indica*



S.No.	Temperature	Fabric analysis
1	70°C	
2	80°C	
3	90°C	
4	100°C	



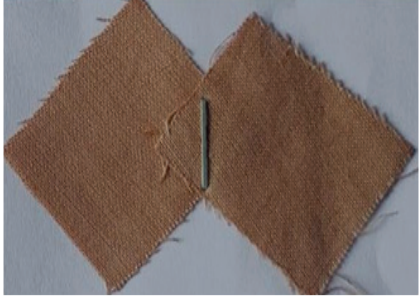
3.	Raw Wool Threads with <i>Azadirachta indica</i>	
4.	Raw Wool Threads with <i>Azadirachta indica</i> + CuSo4	
5.	Raw COTTON Threads with <i>Azadirachta indica</i> + ACACIA CATECHU AS A BIO-MORDANT	

**Table 5** Effect of different mordant on colour intensity of dyed Fabric

S.No.	Extract+ Mordant	Fabric analysis
1	Blank Cotton Fabric	
2	Cotton Fabric Dyed with <i>Azadirachta Indica</i> + CuSO4	
3	Cotton Fabric Dyed with <i>Azadirachta Indica</i> + FeSO4	

**Table 4** Effect of copper sulphate associated flower dye extract of *Azadirachta indica* against dyed Fabric and Colour Stability in raw wool and cotton

Sn.No	Plant extract + Mordant	RAW Fabric Analysis
1.	Raw Cotton Threads with <i>Azadirachta indica</i>	
2.	Raw Cotton Threads with <i>Azadirachta indica</i> + CuSo4	

4	Cotton Fabric Dyed with Green Tea (Biomordent)	
5	Cotton Fabric Dyed with Azadirachta Indica +Bio-mordent	
6	Azadirachta indica+ acacia catechu + green tea (bio mordent) +cuso4 mordent	

ketone, carboxylic acid) vibrations. In addition, the strong peak of 721 cm<sup>-1</sup> in CuSo<sub>4</sub>, FeSo<sub>4</sub>, with bio-mordent shows C=C Bending, the characteristic absorption band due to C-F Fluro compound stretching vibration was observed at 1016.69 cm<sup>-1</sup>, 1095.10 Cm<sup>-1</sup>,1092.37 Cm<sup>-1</sup>,1017.09 Cm<sup>-1</sup> in Blank Cotton Fabric as well as in CuSo<sub>4</sub>, FeSo<sub>4</sub>, Green tea as a bio-mordent. There is a change in a peak of 1371.08 Cm<sup>-1</sup> (S=O) Stretching Sulfonate in both CuSo<sub>4</sub> and FeSo<sub>4</sub> this conclude that there is an entrapment of So<sub>4</sub> compound in dyed metallic mordanted cotton fabric because there is absence of sulfonate in bio-mordanted cotton fabric It can be seen that the spectra of the raw cotton and dyed cotton using different mordants look similar, except for the changes in their intensities. It can be also seen that the bands specific to the raw cotton fabric groups decreased in intensity after dyeing.

### CONCLUSION

Natural dyes are biodegradable, non-toxic and eco-friendly approach for textiles industry. The use of wasted natural resources is the subject of the current inquiry. This study showed the effectiveness of these extracted dyes for cotton fabric in association with specific chemical and biological mordants. Alkaloids, amino acids, quinone, flavonoids, carbohydrates, and saponin were found to be the primary phytochemicals in the extract according to the findings of the phytochemical screening. The findings of the study showed that using a mix

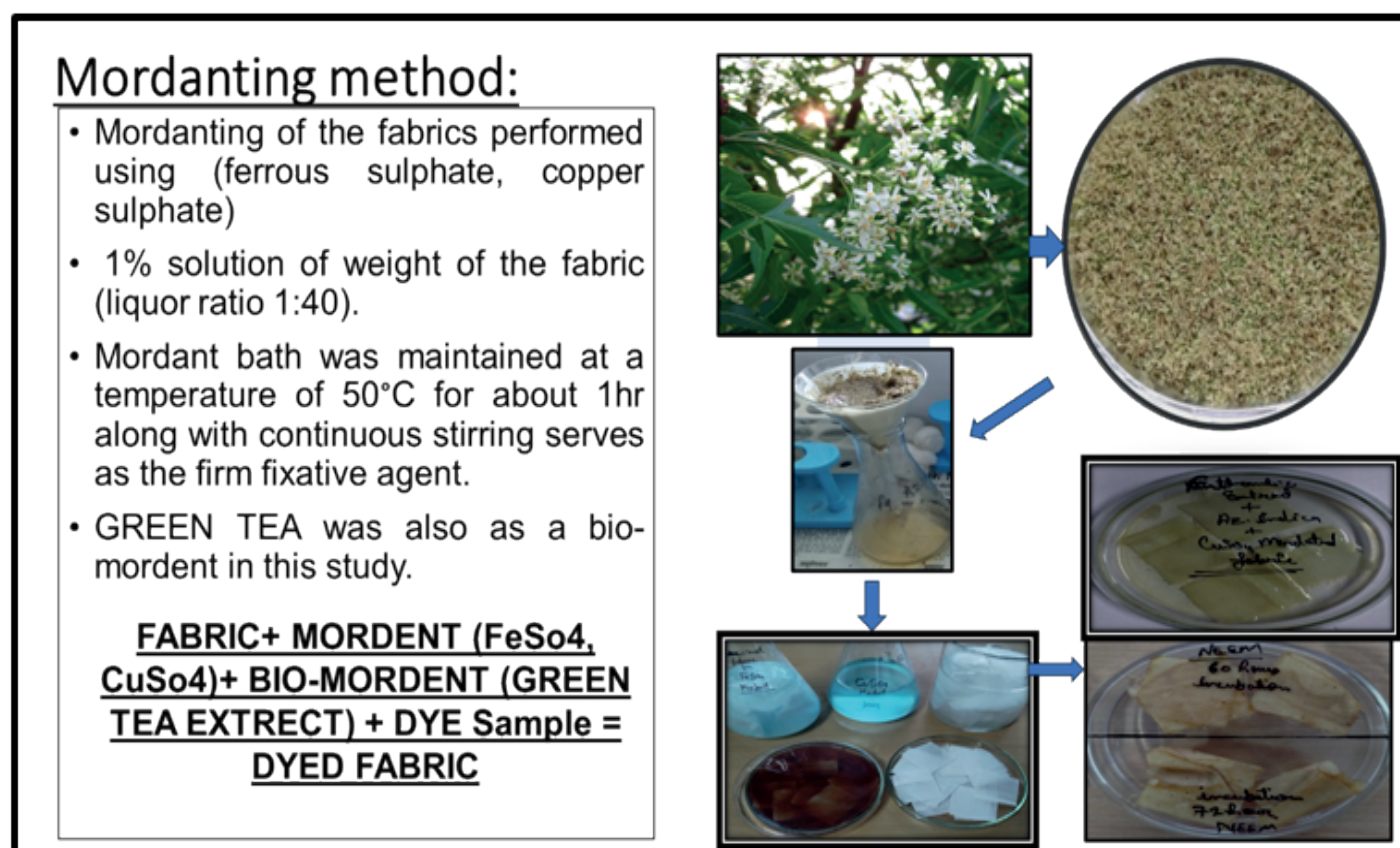


Fig. 1 Effect of chemical and biological mordant for Dyeing Process on Cotton Fabrics using dyes extracted from Azadirachta indica

was observed at strong broad peak of 3550-3200 cm<sup>-1</sup> and weak broad peak of 3200-2700 cm<sup>-1</sup> due to O-H stretching. The absorption band at 1720-1706 and 1725-1705 cm<sup>-1</sup> strong peak observed corresponds to the C=O Stretching (Aliphatic

of chemical and biological mordants, the extracted dye was successfully bonded to cotton fabric. Azadirachta indica flower dyes would be used in the subsequent experiments, along with various bio-mordants (green tea) and mordanting processes, and the natural dyes' colour fastness characteristics would also

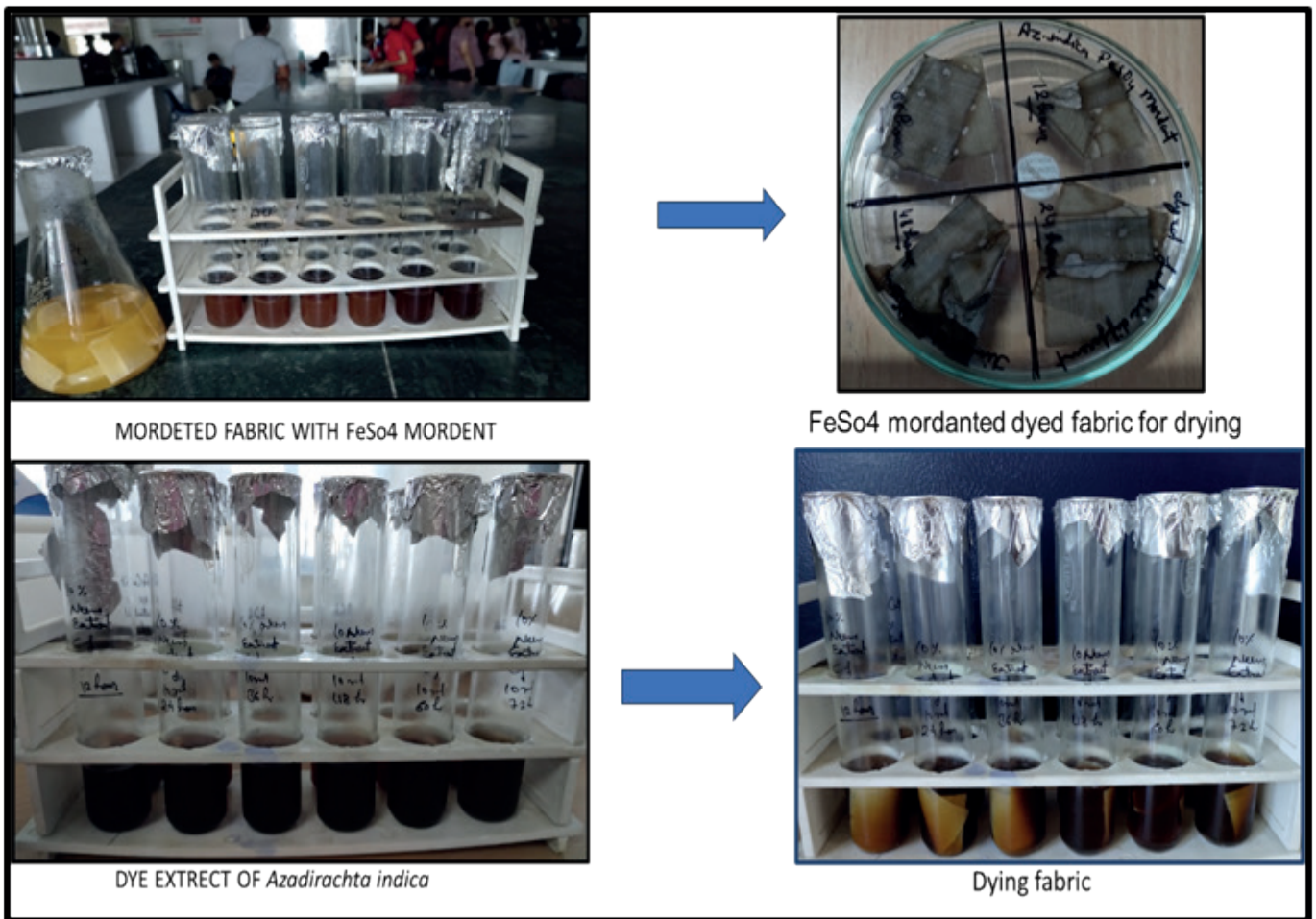


Fig. 2 Dying process of cotton fabrics with ferrous sulphate as mordant for dye extracted from Azadirachta indica

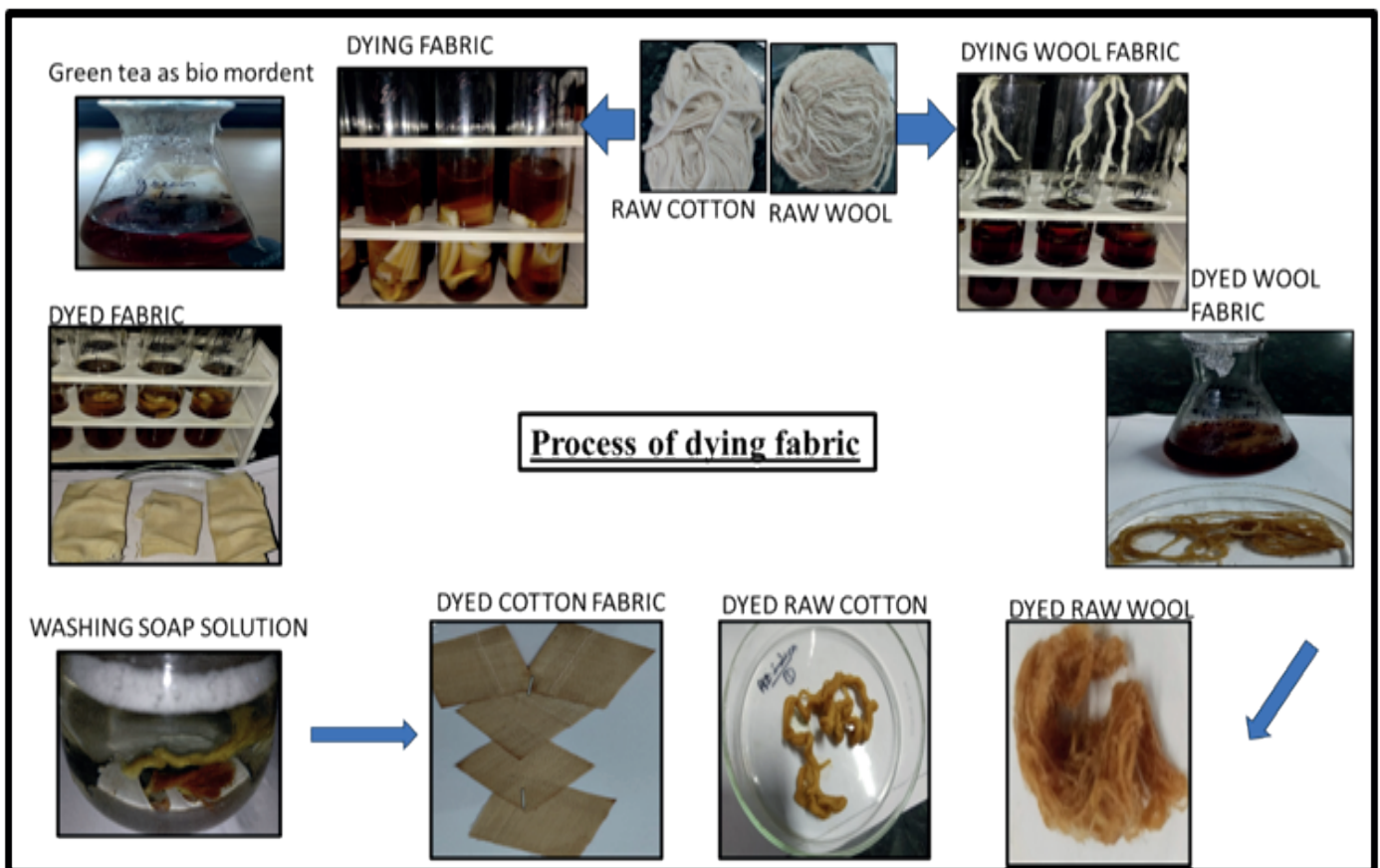


Fig. 3 Color variation in different types of cotton fabrics after using green tea as biomordant



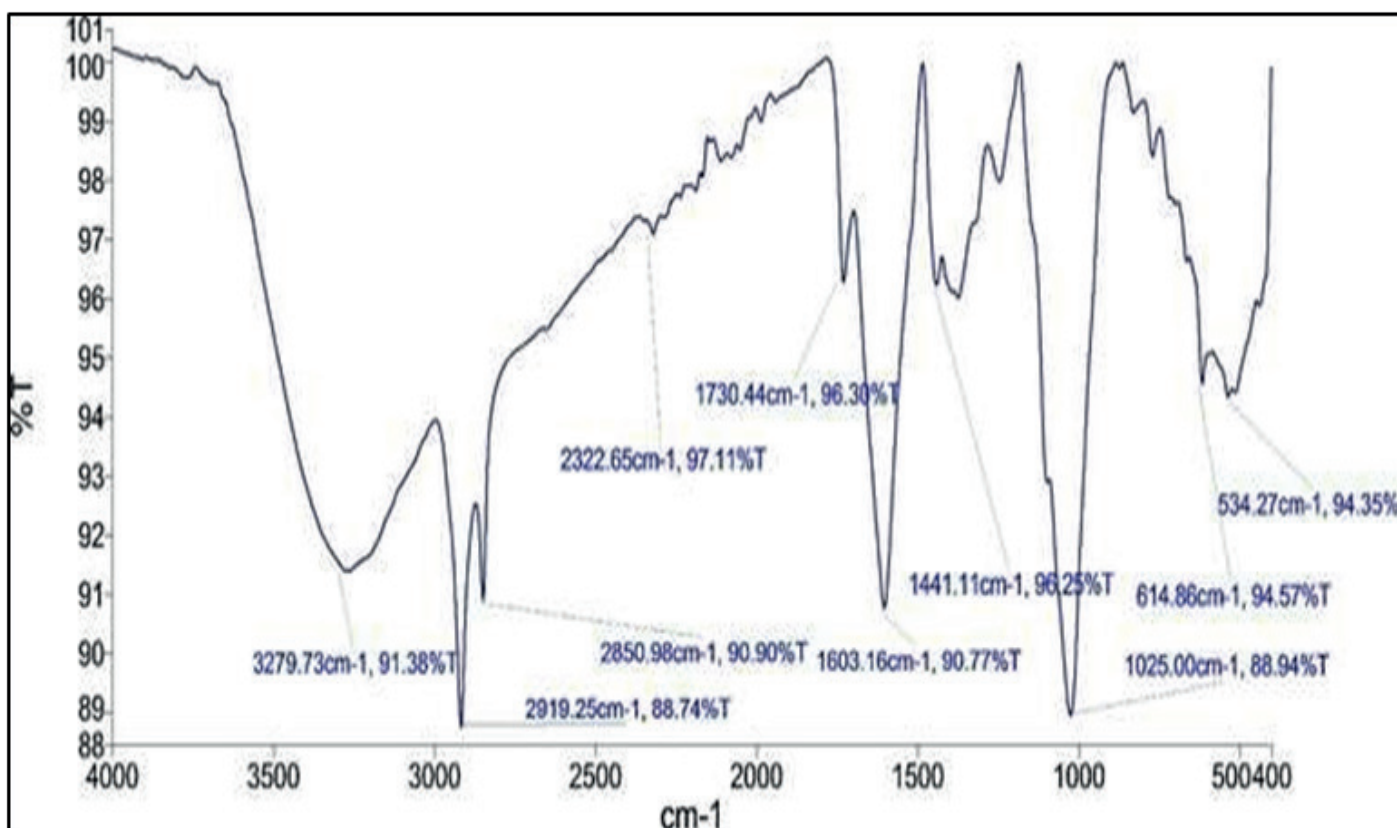


Fig. 4 FTIR spectra analysis of dye extracted from flowers of Azadirachta indica RUBL21378

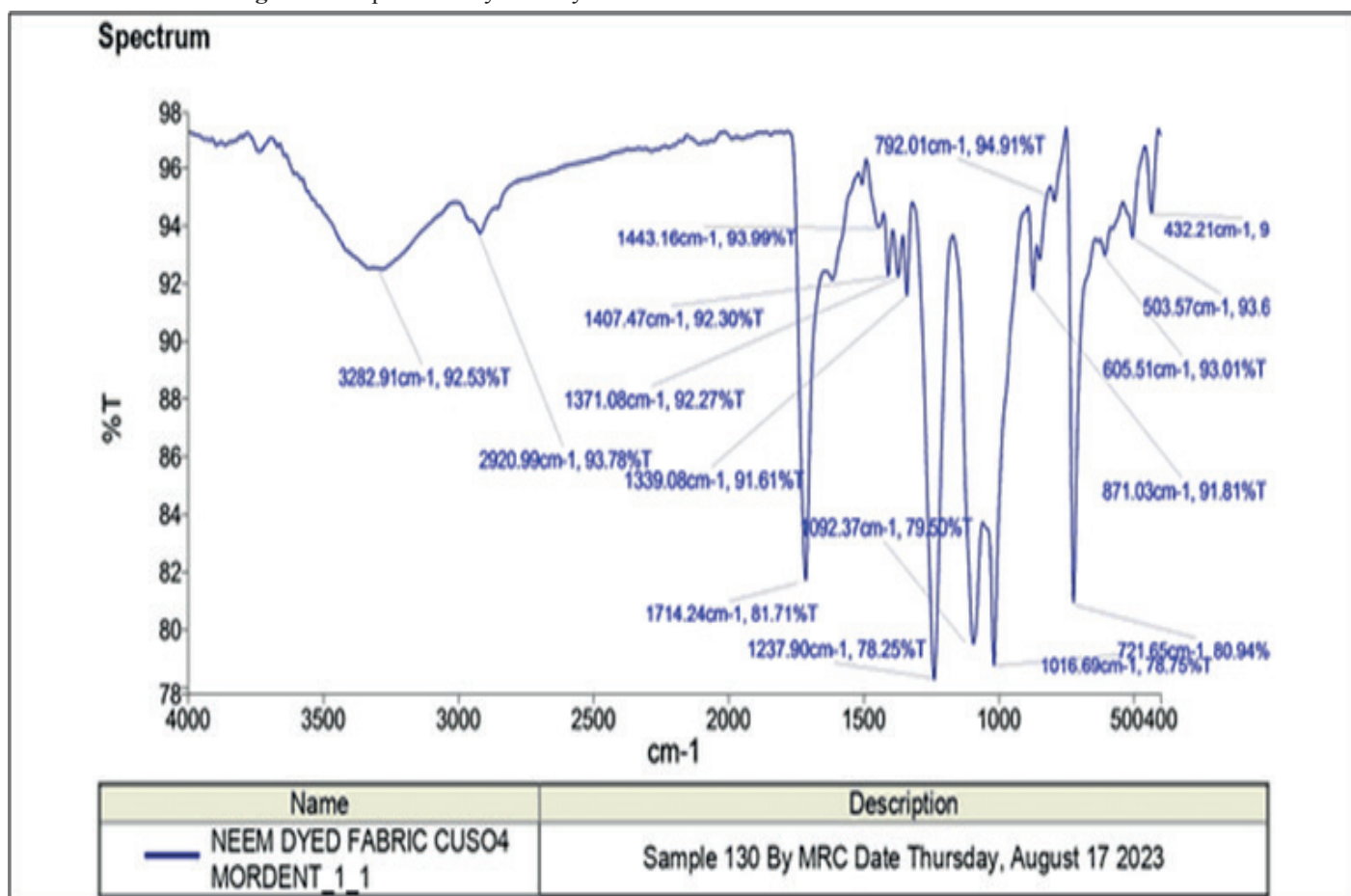


Fig. 5 FTIR spectra analysis of cotton fabric with dye extracted from Azadirachta indica RUBL21378-CuSo4 mordant

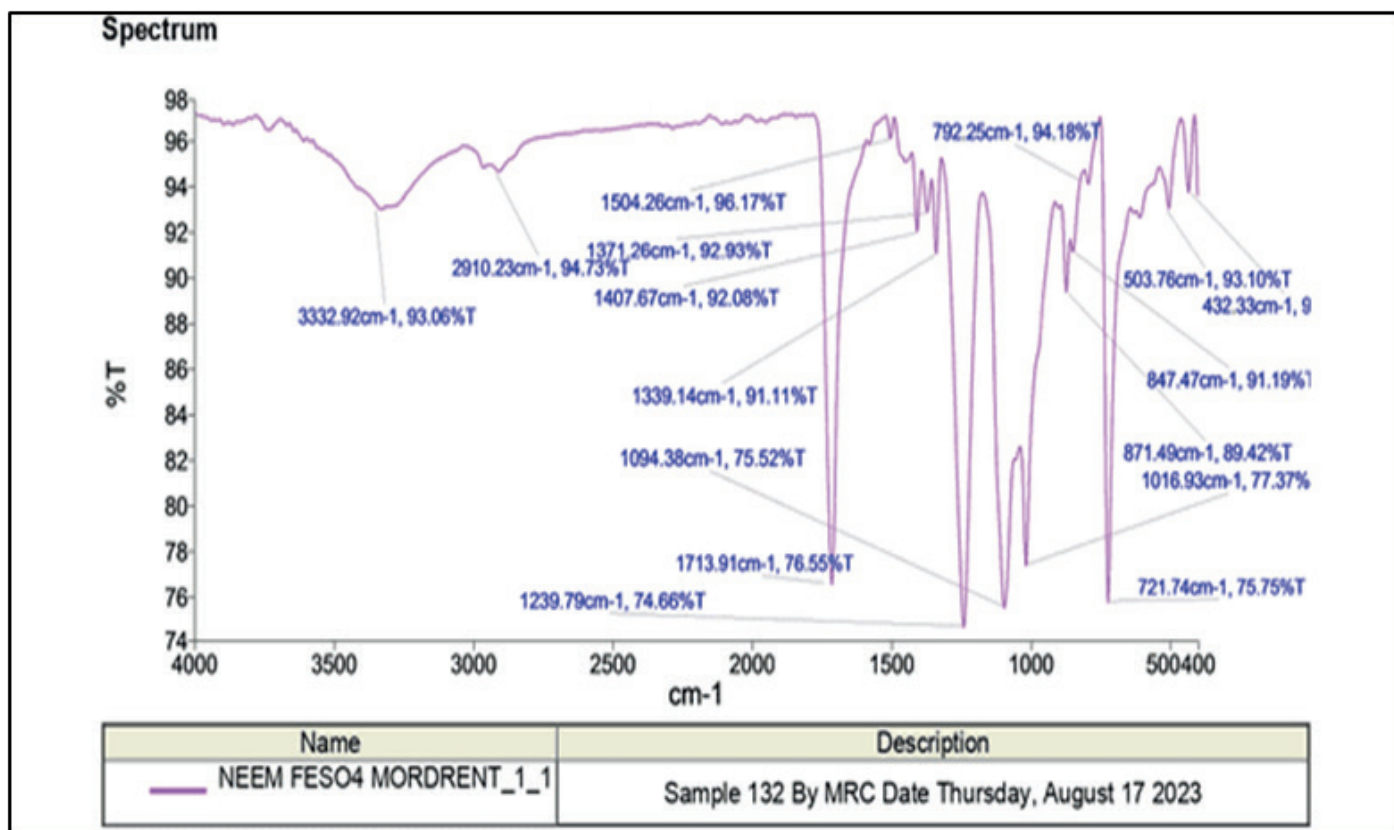


Fig. 6 FTIR spectra of cotton fabric with dye extracted from flower of Azadirachta indica RUBL21378- FeSo4 Mordant

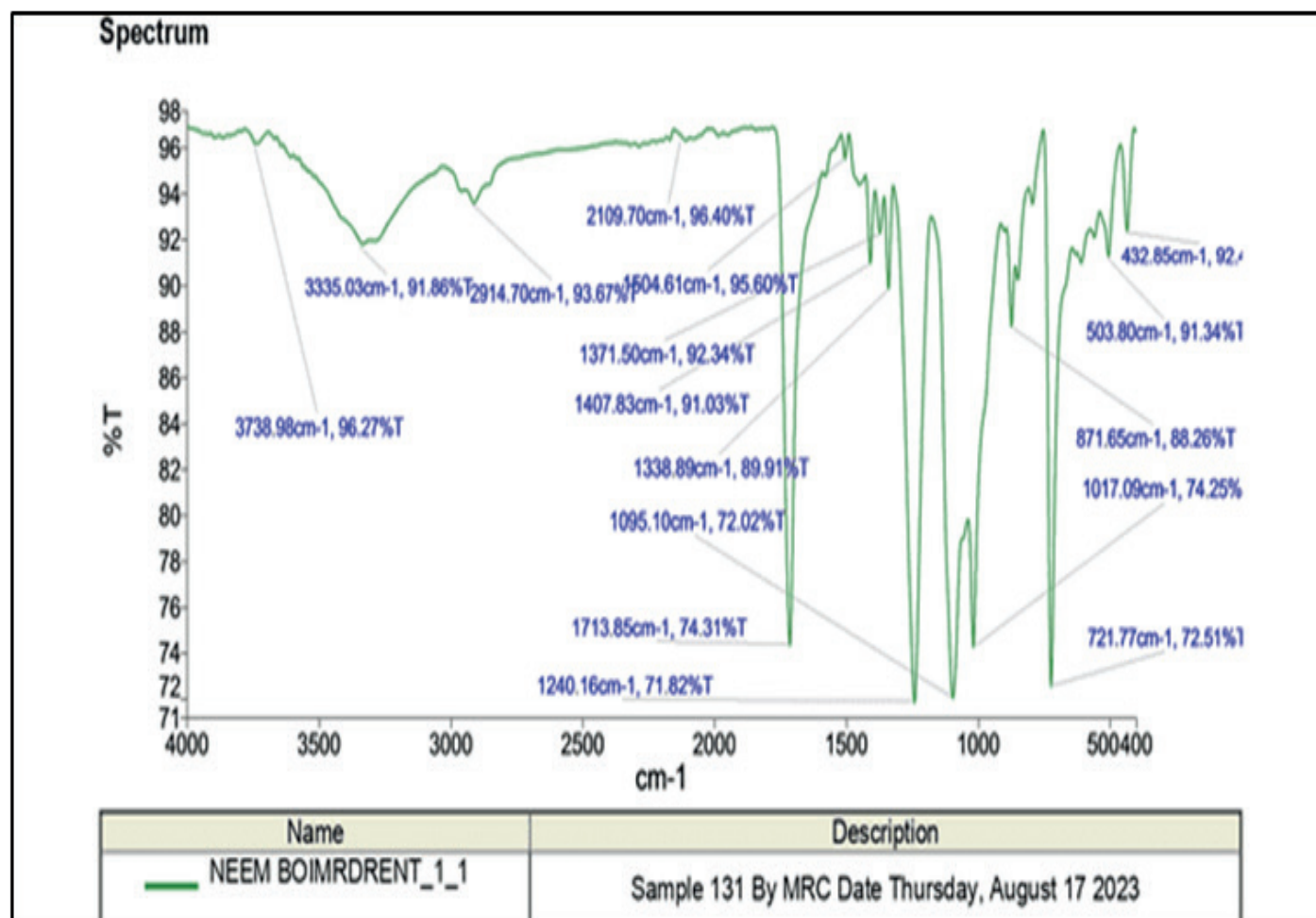


Fig.7 FT-IR spectra of cotton fabric with Azadirachta indica (Neem flower) (RUBL21378) extract mordanted with bio mordant (Green Tea).

be examined. To create an eco-friendly atmosphere, the extraction and use of the extracted natural dye would be quite beneficial. The current technique will benefit from commercialization to find a practical use in the handloom and textile sectors. Additionally, this will have a significant effect, particularly on the rural dyer communities' economic development.

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#### Competing Interests

The authors have no relevant financial or non-financial interests to disclose.

#### Ethical Responsibilities of Authors

All authors declare that this research article is original and has not been published elsewhere in any form or language (partially or in full). This study has not included any study related to humans and/or animals.

#### Data transparency

All authors agree that all data, materials as well as software application has supported their published claims and complied with field standards.

#### Credit authorship contribution statement

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Ms. Meena Choudhary. Dr. Priyanka Singh (as supervisor) has guided her for designing of experiment, compilation of data and drafting of manuscripts. All authors have read and approved the final manuscript.

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