

NATURAL DYE EXTRACTION FROM THE COMBINATIONS OF *Camellia sinensis*, *Portulaca grandiflorum*, *Macrotyloma uniflorum* *Vigna radiata*, *Punica granatum* & *Mentha piperita*

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ABSTRACT

Environmentalists are usually involved about the unbridled use of artificial dyes in the material enterprise as they lead to water, air pollutions and exceptional troubles related to waste water disposal. Natural dyes have gained a space starting in the area of fabric coloration. They are extra well matched with the environment in contrast to synthetic dyes because they are eco-friendly, non-allergic, non-toxic and also they are biodegradable. Due to the recognition amongst human beings involving the environmental and health risks elated with the use of artificial dyes, the craze for the apparels dyed with herbal dye is growing exponentially. The current study focused to produce various shades on the cotton fabric by using the combinations of *Camellia sinensis* & *Portulaca grandiflorum*, *Macrotyloma uniflorum* & *Vigna radiata*, *Punica granatum* & *Mentha piperita* with two different extraction methods such as Aqueous extraction and Alkaline extraction. The alum is used as mordant. The colour fastness was analysed and assessment confirmed that all dyed fabrics could withstand colour fastness test moderately. Further to discover precise chromophores in dye extracts and their molecular configurations due to mordants, FT-IR spectroscopy was employed.

Keywords: Natural dye, Mordant, Alkaline extraction, Aqueous extraction, Wash fastness, FT-IR Spectroscopy.

1. INTRODUCTION

Use of synthetic dyes has increased dramatically in several industries including textiles, pharmaceuticals, food processing etc. In recent years, it is more readily available and has better fastness properties with adverse effects on the human body including allergic reactions. Synthetic dyes are difficult to degrade and accumulate in the natural environment¹. They are widely employed in a variety of industries, with textile processing industries being the most significant consumers. Several new dyes have been added to the ever-growing list². It may pollute the environment leading to skin illness and possesses health risks to human and other essential organisms. Natural colours generated from plants and animals are thought to be harmless because of their nontoxic, non-

carcinogenic and biodegradable properties^{3,4}. In humans, cationic dyes can cause hypertension, shock, vomiting and also tissue necrosis⁵. During the degradation process, 130 of the 3,200 Azo dyes in use are thought to create carcinogenic aromatic amines⁶.

Natural dyes are organic compounds derived from natural sources such as plants, insects, animals and minerals without any chemical treatment^{7,8}. These colours are caused by absorption of light in the visible area^{9,10}. Natural dyes are now a days in demand no longer only in fabric industry however in cosmetics, leather, meals and pharmaceuticals. The present day dyestuff requirement from the enterprise is about three million tonnes. Considering this fact, the use of natural dyes in mainstream material processing is a massive task¹¹. Natural dyes might help you archive a delicate hue or relaxing tint if you want to opt for that style. When swallowed some natural colours such as Carmine found in lipsticks, produce no harm or health problems^{12,13}. Small portions are also used in coloration of paper, leather, shoe polish, wood, cane and such other products requiring coloration¹⁴.

MATERIALS AND METHODS

- Cotton cloth (A4 Size)
- Aluminium sulphate (1.496 g)
- Sodium carbonate (0.374 g)
- Sodium hydroxide (10 %)

Sample collection: The *Mentha piperita* leaves were collected from the plants grown at college campus. *Punica granatum* peel was collected from juice shops. Tea dust packet with trade mark name 3 Roses and legumes like *Macrotyloma uniflorum* and *Vigna radiata* were brought from grocery store. *Portulaca grandiflora* were brought from flower market.

Samples used: Combinations of 3 different natural sources were used for synthesis of a natural dye:

1. *Camellia sinensis* & *Portulaca grandiflora* (Tea powder & Rose petals)
2. *Macrotyloma uniflorum* and *Vigna radiata* (Horse gram & Mung bean)
3. *Punica granatum* and *Mentha piperita* (Pomegranate peel & Mint leaves).

Sample preparation: The above three combination of samples were subjected to pigment extraction processed by various extraction methods.

Extraction of Natural Dye was carried out by TWO methods:

1. **Aqueous extraction:** In aqueous extraction method, 10g of each combination of samples (Tea powder + Rose petals), (Horse gram + Mung bean), (Pomegranate peel + Mint leaves) were boiled individually in 200ml distilled water at 100 °C for 1 hour. The decolorized samples were taken out from extraction solvent. Then filter the solution and use it for further study.

2. **Alkaline extraction:** In alkaline extraction method, 10g of each combination of samples (Tea powder + Rose petals), (Horse gram + Mung bean), (Pomegranate peel + Mint leaves) were boiled individually in 1% Sodium hydroxide for 1 hour. The decolorized samples were taken out from extraction solvent. Then filter the solution and use it for further study.

[*Punica granatum* (Pomegranate) Peel were dried in hot air oven at 80 °C for 3 hours. The dried peel was grinded into fine powder].

Scouring of Cotton cloths: Cotton cloths used for dyeing were boiled in 10% NaOH solution for 10 minutes to dispose off starch and impurities from the cloth. The NaOH treated cotton cloths were then washed with distilled water.

Preparation of Mordant (Alum) : 0.748 g of Alum and 0.187 g of Washing soda were mixed in 100 ml of water and was stored for further use.

Dyeing and Mordanting: The clean scouring cotton cloths were treated with Alum mordant.

Washing fastness: The ISO Standard strategies were utilized to evaluate the fastness residences of dyed fabrics. The viewed requirement had been as follows: IS/ISO 105- C10:2006 A (1) for washing fastness. The test was carried out at SITRA (The South India Textile Research Association, Coimbatore) and the results obtained are discussed below.

FTIR: The samples were subjected to FTIR analysis to comprehend the different compounds and functional groups that are present. This would enable us to understand which compounds render binding properties to the cloth.

RESULTS AND DISCUSSION

Natural dyes have higher biodegradability and usually have greater compatibility with the environment. Wide range of mild and dark shades had been acquired while dyeing with natural pigments.

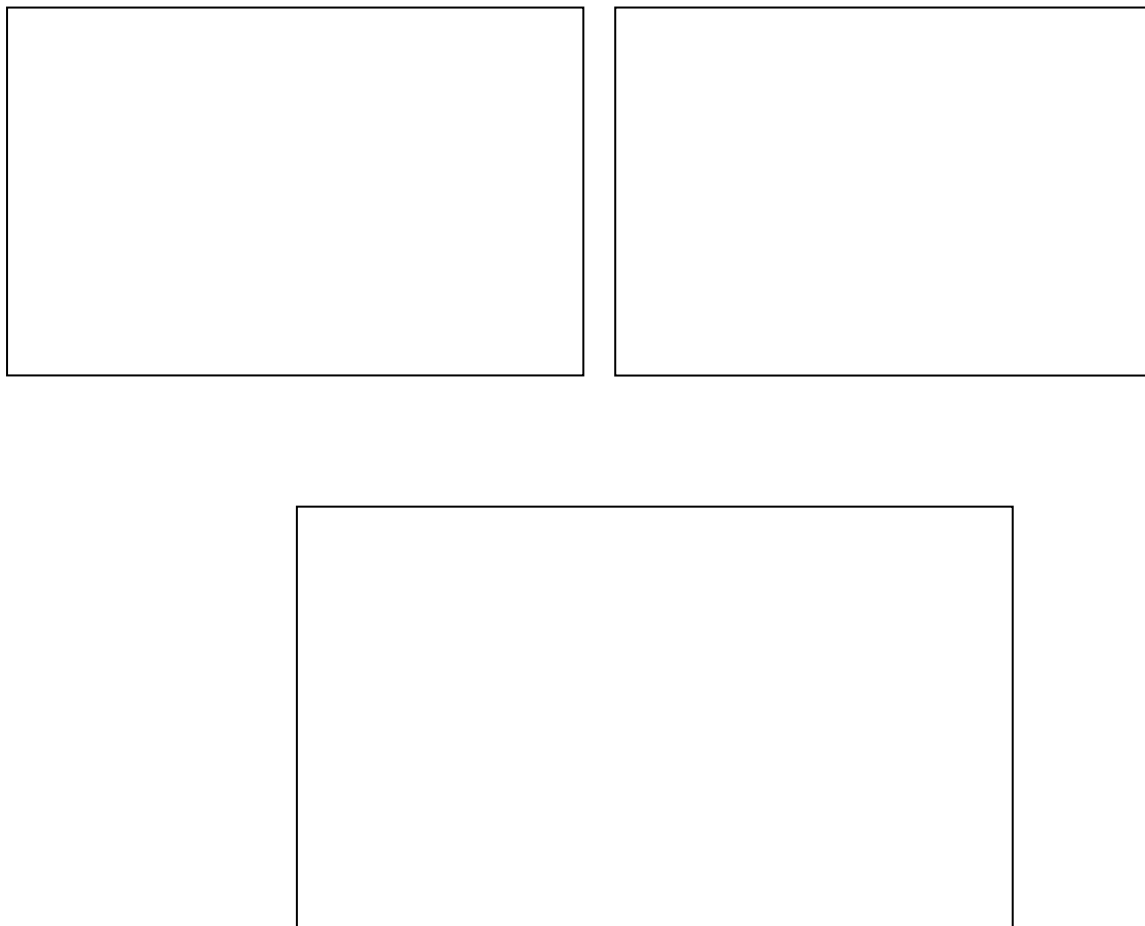
Determination of Fastness properties:

Wash fastness of dye is influenced by using the rate of diffusion of dye and state of dye inner the fibre. Another cause is that they have a tendency to aggregate inner the fibre (thereby increasing the molecular sine) and therefore exhibit suitable wash fastness. In case of the cotton fibre which is mordanted chemically with Alum mordant and dyed with the combination of samples such as Tea powder & Rose petals, Horse gram and Mung bean and Powdered Pomegranate peel & mint leaves in 2 different extraction methods like Aqueous extraction and Alkaline extraction, that confirmed precise wash fastness.

The Figures 1, 2, and 3 showed that using mordants, wash fastness of mordanted samples could be ranked according to their quality of color retention. All the 3 combinations of samples were determined to

ranges from a ranking of four to a rating of five (4-5)[The Grey scale ratings resembles that the color gets slightly changed and no changes occurs].

The washing fastness result confirmed that all the dyed samples of Saffron extract in acidic condition had proper to very good fastness homes on Pashmina fabric with and except the use of mordants was falling between 4 to 5. It is also discovered that colorings received are no longer only vivid and alluring but also quite fast to a massive extent for washing¹⁵. Followed by the fastness assay as per the ISO standards, the samples were subjected to FTIR analysis.



FTIR ANALYSIS:

The FTIR spectra of the optimized extracts of various samples were given in the figures below. The wide top inside the area $3550\text{--}3100\text{ cm}^{-1}$ is the characteristic of the --OH stretching vibration of benzene rings and methylol groups of phenolic structure like tannins and flavonoids. The peaks around 2960 and 2890 cm^{-1} are due to symmetrical and asymmetrical vibrations of --CH_3 groups. Small peaks around $1260\text{--}1085\text{ cm}^{-1}$ shows the presence of aromatic --C--C-- and --C--O-- stretch of phenols. This placement suggests that the --OCH_3 group is present on the aromatic device which refers to Eupatilin and Jaceosidin¹⁶.

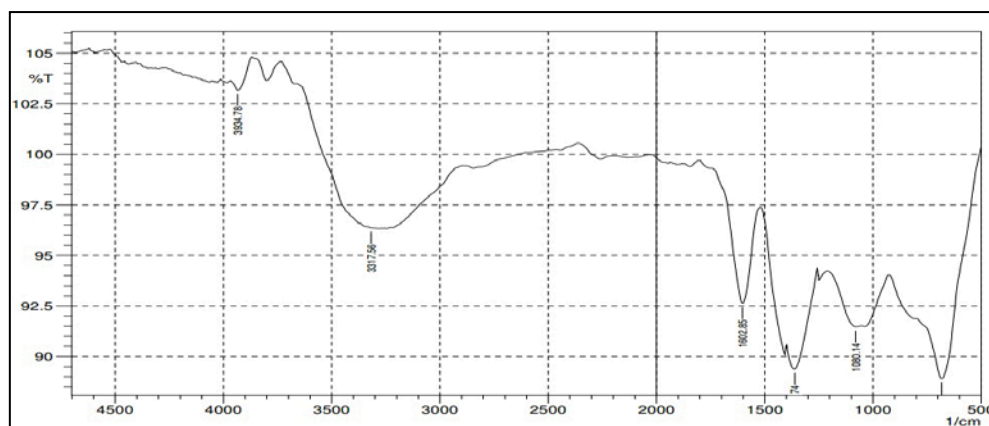


Figure 4 FTIR spectra of Aqueous Extraction (*Camellia sinensis* & *Portulaca grandiflora*)

Figure 4 shows the IR spectrum of combination of *Camellia sinensis* & *Portulaca grandiflora* (Aqueous Extraction). In that the peak ranges at 3934.78 cm^{-1} is because of the O-H stretching, which contains hydroxyl compounds. The peak ranges at 3317.56 cm^{-1} is due to C-H stretching/ contains alkyne compounds and also presence of O-H stretching contains alcohol compounds. The peak ranges at 1602.85 cm^{-1} indicates C=C stretching resembles conjugated alkene compounds. The peak ranges 1361.74 cm^{-1} is due to C-H (rock methyl) contains alkane compounds. The peak ranges at 1080.14 cm^{-1} is due to C-O stretching indicates the presence of alcohol group. The peak ranges at 682.8 cm^{-1} is because of =C-H bending contains alkene compounds.

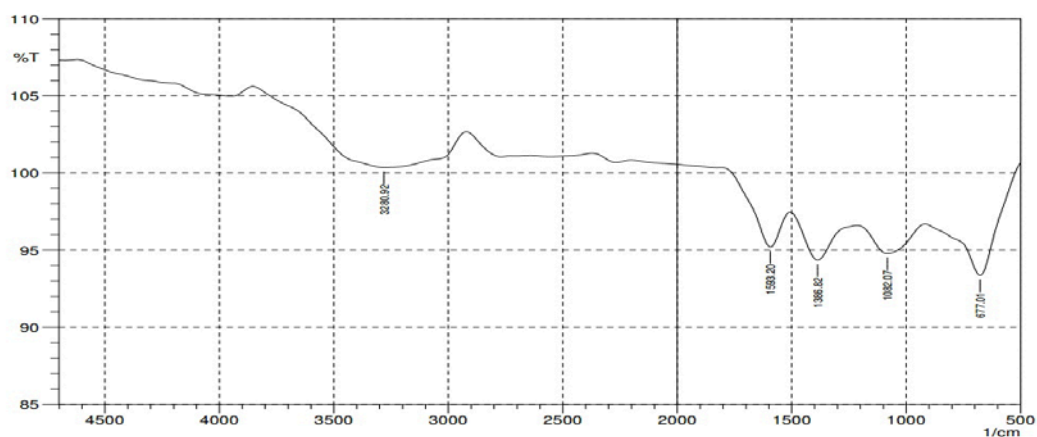


Figure 5 FTIR spectra of Alkaline Extraction (*Camellia sinensis* & *Portulaca grandiflora*)

Figure 5 shows the FTIR spectra for the mixture of *Camellia sinensis* & *Portulaca grandiflora* in alkaline extraction. The peak ranges at 3280.92 cm^{-1} is because of O-H stretching indicates the carboxyl acid and alcohol compounds. The peak ranges at 1593.2 cm^{-1} due to C-C stretching (in ring) contains aromatic compounds. The peak ranges at 1386.82 contains sulfonyl chloride and aldehyde compounds, due to S=O stretching and C-H bending. The peak ranges at 1082.07 cm^{-1} contains alcohol compounds due to C-O stretch. The peak ranges at 677.01 cm^{-1} is because of the =C-H bending and C-H “loop” indicates the presence of alkene and aromatic compounds.

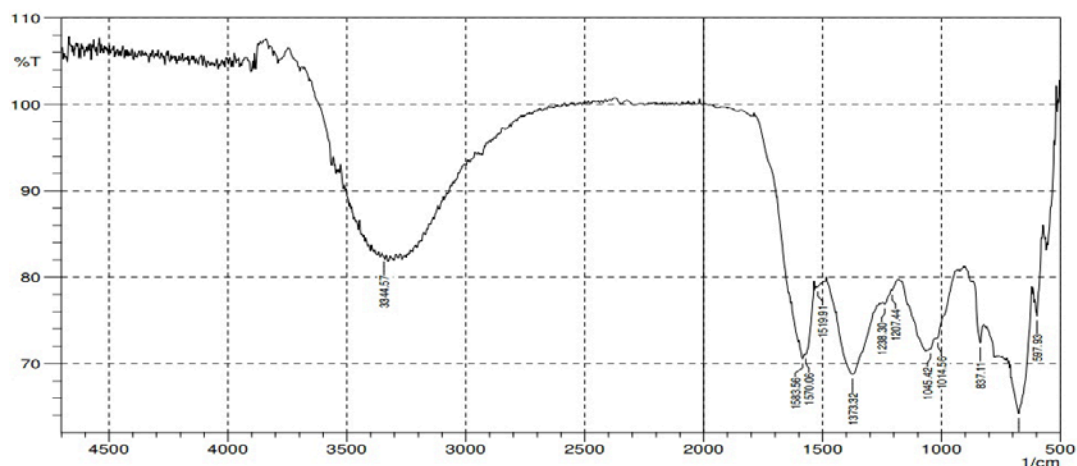


Figure 6 FTIR spectra of Aqueous Extraction (*Macrotyloma uniflorum* & *Vigna radiata*)

Figure 6 shows the FTIR spectra for the mixture of *Macrotyloma uniflorum* & *Vigna radiata* in aqueous extraction. The peak ranges at 3344.57cm^{-1} contains Alcohol compound due to O-H stretching. The peak ranges at 1583.56 and 1570.06cm^{-1} is because of N-H bending and C=C stretching contains amine and cyclic alkene compounds. The peak ranges 1519.91cm^{-1} is due to C-C stretching (in ring), contains aromatic compounds. The peak ranges at 1373.32cm^{-1} is because of O-H bending contains C=O of carboxylic acid compounds. The peak ranges at 1238.3cm^{-1} is because of C-O bending, C-O stretching and C=O stretching that contains C=O of carboxylic acid and ester compounds. The peak ranges at 1207.44cm^{-1} contains alcohol and esters, due to C-O stretching. The peak at 1045.42 & 1014.56cm^{-1} contains alkenes and esters, due to C-H bending and C-O stretching. The peak 831.11cm^{-1} due to C-H “oop” and C-Cl stretching contains aromatic and organic compounds that contain halogen. The peak ranges at 675.09cm^{-1} contains alkynes and organic compounds with halogen compounds, due to C-H bending and C-Br stretching. The peak at 597.93cm^{-1} contains due to C-Cl and C-Br stretching that contains organic compounds with halogens.

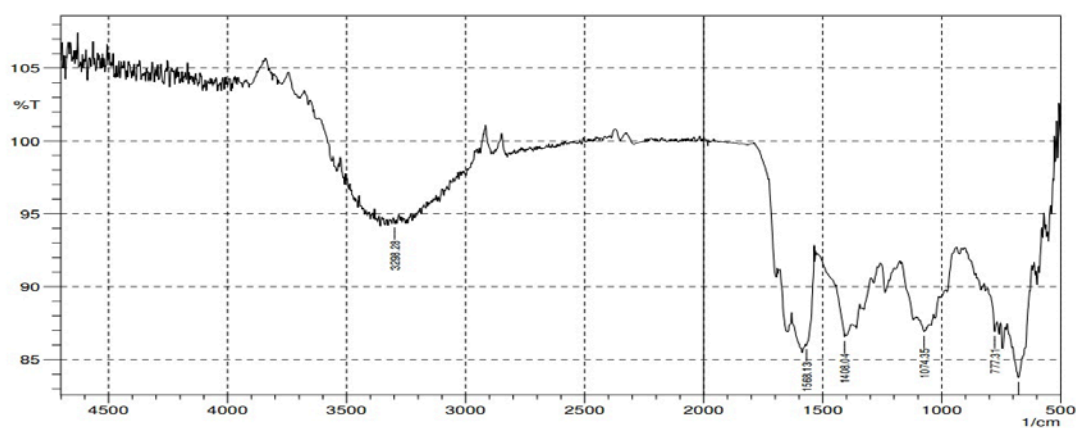


Figure 7 FTIR spectra of Alkaline Extraction (*Macrotyloma uniflorum* & *Vigna radiata*)

Figure 7 shows the FTIR spectra for the mixture of horse gram and mung bean in alkaline extraction. The peak ranges at 3298.28 and 1408.04 cm^{-1} is because of O-H stretching containing C=O of carboxylic acid compounds. The peak at 1568.13 cm^{-1} contains cyclic alkene due to C=C stretching. The peak ranges at 1074.35 cm^{-1} contains alcohol compounds due to C-O stretching. The peak ranges at 777.31 and 677.01 cm^{-1} contains C-Cl and C-Br stretching contains organic compounds with halogens.

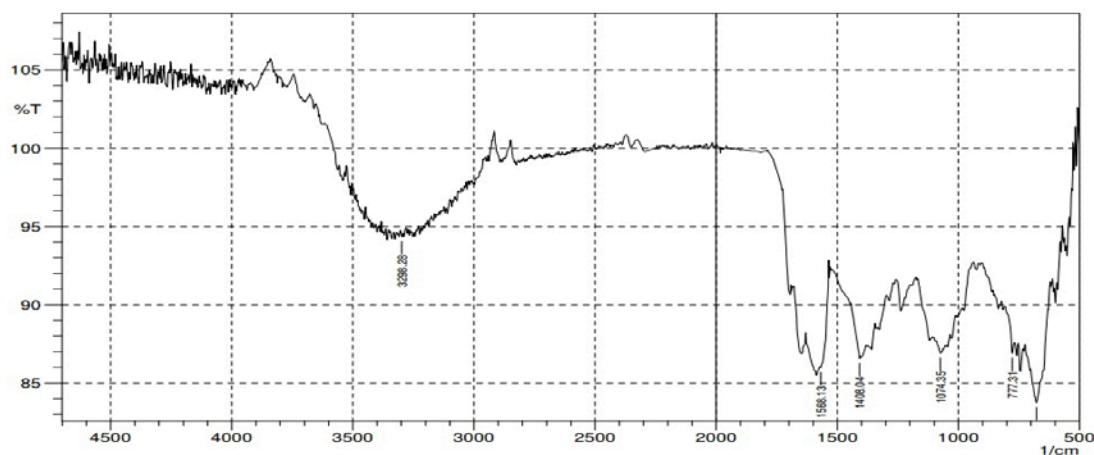


Figure 8 FTIR spectra of Aqueous Extraction (*Punica granatum* & *Mentha piperita*)

Figure 8 shows the FTIR spectra for the mixture of *Punica granatum* & *Mentha piperita* in aqueous extraction. The peak ranges at 3273.20 and 2335.80 cm^{-1} are due to O-H stretching contains alcohol compounds. The peak ranges at 1587.42 cm^{-1} is because of C-C stretching that contains aromatic compounds. The peak ranges at 1327.03 cm^{-1} contains Sulfone and aromatic amine compounds, due to S=O and C-N stretching. The peak ranges at 690.52 cm^{-1} is because of C-H stretching, contains alkenes and alkyne compounds.

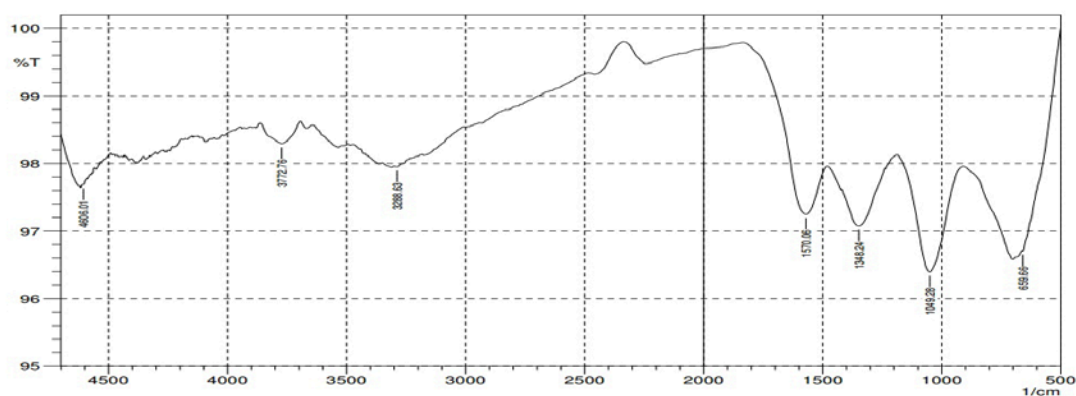


Figure 9 FTIR spectra of Alkaline Extraction (*Punica granatum* & *Mentha piperita*)

Figure 9 shows the FTIR spectra for the mixture of *Punica granatum* & *Mentha piperita* in alkaline extraction. The peak ranges at 3772.76 cm^{-1} contains hydroxyl compounds, due to O-H stretching. The peak ranges at 3288.63 cm^{-1} is because of C-H stretching contains alkynes. The peak ranges at 1570.06 cm^{-1} contains cyclic alkene compounds due to C=C stretching. The peak ranges at 1348.24 cm^{-1} due to O-H bending contains alcohol compounds. The peak ranges 1049.28 cm^{-1} because of the C-O stretching which contains esters. The

peak ranges 659.60 cm^{-1} contains halogen and alkyne compounds, due to C-Cl and C-Br stretching and C-H bending.

SUMMARY

The current research unveiled that the combination of *Camellia sinensis* & *Portulaca grandiflorum*, *Macrotyloma uniflorum* & *Vigna radiata*, *Punia granatum* & *Mentha piperita* has the dyeing potential for cotton fabrics. All the 3 combinations of natural dyes have been efficiently dyed on the cotton fabrics. The study aimed to use natural pigments as organic dyes for textiles where different chemical mordants were used. These natural pigments were found non-toxic to the humans and eco system. As herbal dyes indicates non-toxic, non-allergic effects and consequences on less air pollution as properly as fewer facet effects. Natural dyes provide a host of advantages for human use. Thus, it can be concluded from the current study that these mixtures of samples are an excellent source of practical bio-colorant for cotton fabric.

ACKNOWLEDGEMENT

The authors would like to acknowledge the financial support (seed money) rendered by the management, Principal, Dean – Research & Development and Department of Biotechnology for having motivated to materialize this article. The communication number is DRNGPASC2022-23-BS02.

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