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EXTRACTION AND CHARACTERISATION OF NATURAL DYE FROM ORANGE PEEL FOR TEXTILE APPLICATIONS

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ABSTRACT. Awareness of the need to protect the environment and people's health has led to an intensification of concerns for obtaining sustainable products and processes. Toxic waste created during the production and use of synthetic dyes has an impact on both human and environmental health. As a result, natural dyes are more secure and safer than synthetic dyes. This study is significant because it has the potential to help develop sustainable and environmentally friendly textile dyeing techniques. In this study, a natural dye was successfully extracted from orange peel (*Citrus recticulata* Rutaceae) which was applied on textile fibre and was found to be partly effective for eco-friendly dyeing applications. The extracted dye does not have a good wash fastness, which is the ability of the fabric to retain its colour after washing. This weakness is demonstrated by carrying out a Fourier transform infrared (FTIR) analysis where the potential binding mechanisms between the dye and the textile fibres was revealed. Different functional groups can interact with the fibre’s functional groups, affecting the dye's affinity for the fabric and its overall colour fastness properties. Findings show that dyes from orange peels showed promise in this study, but need to be improved further. Therefore, the study suggests that further research is needed to optimize the dyeing process and improve the fabric's resistance to washing and other environmental factors.

Keywords: mordant; natural dye; orange peel; synthetic dye; textile industry.

INTRODUCTION

Ecological alarms and cognizance have favoured a change to sustainable

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products and processes that are less impactful to the environment and living organisms' well-being (Muniz *et al.*, 2020). Natural colourants have been utilized, mainly for textile colouration, since very old times (Helmy, 2020). Natural dyes are currently in demand not only in the textile industry but also in the cosmetics, leather, food and pharmaceutical industries (Chungkrang *et al.*, 2021). In the field of obtaining textile materials, the consequences of processes and products that raised suspicions were evaluated, and in many cases improved solutions or even new alternatives, friendly to the environment and people, were found (Tayyab *et al.*, 2020). Neglect of natural dye utilization started in 1856 due to the advent of synthetic dyes that yielded uniform colour shades (Naveed *et al.*, 2020). Its current revival can be attributed to worldwide environmental awareness (Edeen, 2015; Pizzicato *et al.*, 2023; Talib *et al.*, 2023). About half (50-65%) of the total orange fruit weight is orange peels (*Citrus recticulata* Rutaceae), which is highly rich in pigments (Devi and Saini, 2020; Kodal and Aksu, 2017). Indonesia recorded more than 2 million tons of orange production in 2019 (Kusumawati *et al.*, 2020); China's annual yield is around 15 million tons, ranked third in the world (Li *et al.*, 2021; Wei *et al.*, 2013) and Nigeria produces about 1.626 million tons annually (Akpan *et al.*, 2014). Globally, the production figure is put at over 100 million tons yearly (Brezo-Borjan *et al.*, 2023; Che *et al.*, 2024).

There are over 500 dye-yielding plant species, and China occupied the top seat in dyestuff exports, followed by India (Parikh *et al.*, 2019). Thus, sustainability of the feedstock is not an issue.

Natural dyes are usually less allergenic and toxic (Gupta, 2020; Kumar and Dhinakaran, 2017; Parikh *et al.*, 2019) than synthetic dyes and generate wastewater that can be treated by biodegradation (Baaka *et al.*, 2017), all reasons why these kinds of colourants have been preferable as environmentally less impactful alternatives to certain synthetic dyes (Khan *et al.*, 2018). Knowing the potential of local plant-based substrates for natural dye extraction is critical for their use and value in native species (Muniz *et al.*, 2020). Toxic waste generated during the synthesis and application of synthetic dyes affects the health condition of humans and the environment (Sayem *et al.*, 2021). As such, the use of natural dyes is safer and more secure compared to artificial synthetic dyes. The aim of this study is to develop a healthy, sustainable and environmentally friendly alternative for artificial dye production and application. The objectives are to extract natural dye from orange peels, use a pH meter to measure the degree of acidity or alkalinity, determine the important functional groups using Fourier transform infrared (FTIR) spectroscopy and test the effectiveness of the produced dye on textiles. Already, quality shades and fastness have been achieved using lemon and orange peel waste in dyeing (Aishwariya, 2020). FTIR was previously tested on wool dyeing using combinations of walnut husk and myrobalan (Hosseinnezhad *et al.*, 2023) and mordanted cotton with *Syzygium cumini* fruits (Periyasamy, 2022) as bio-mordants. Kim *et al.* (2014) examined the rubbing fastness, water fastness, alkaline colour change, dry cleaning fastness, light fastness and washing fastness of citrus peel extract on cotton, wool, rayon and silk fabrics. There is a need to experiment with the forgone properties to compare amongst onion peel, *Areca concinna* peel, walnut shell, pine nut shell, olive fruit peel, tangerine peel, onion peel, orange peel, avocado seed and lemon peel in one study. The present study concentrated only on the effect of pH, functional group, temperature, acting-mordant concentration and textile weight after dyeing with orange peel pigment. Conventional mordants are iron sulphate, alum, copper sulphate, stannous chloride and potassium dichromate (Zubairu and Mshelia, 2015).

MATERIALS AND METHODS

Orange Peel Collection and Pretreatment

Fresh orange peel waste samples were collected from a local fruit seller at Gamboru Market in Jere Local Government, Borno State, Nigeria. The orange peels were washed rigorously with water to free them of contaminants and spread on a tray to dry under sunlight. The dried peels were crushed into powder form using a grinder (mortar and pestle and grinding machine), as carried out by Kumar and Dhinakaran (2017). This was later screened into different sizes by a vibrating screen to obtain fine dried powder.

Dye Extraction

About 200 mL of distilled water was poured into a 400 mL beaker and 12 g of dried and pulverized *citrus* peels (puree) were soaked in the distilled water for 48 h, as described by Kusumawati *et al.* (2020), before heating to 60°C for 30 min to yield what is called 'dye extract'. Next, the dye extract was left for 30 min at ambient temperature and filtered (Parikh *et al.*, 2019). Coloured crude dye solution (150 cm^3) was diluted with distilled water (50 cm^3) and immediately used for dyeing. Normally, the extraction efficiency is improved by soaking the dried powder in the cold distilled water before the extraction step.

pH Determination

An electronic device called a pH meter was used to measure the quantifiable parameter 'pH' in liquids or semisolid compounds in certain situations. Calibration of pH meters was done using buffer solutions with known hydrogen ion activity before the pH of the sample was measured.

FTIR Spectroscopy Analysis

A UV-1800 FTIR spectrometer was used to determine the functional groups within the samples taken.

Dyeing and Mordanting

Initially, a cloth was cleansed with 'Good Mama powder' detergent and rinsed with cold distilled water to remove impurities. The cotton fabric has a yarn count of 40, making it finer and smoother with high density compared to a coarser yarn count. Such a high-density plain weave pattern cloth contributes to its smoother texture and a more compact fabric structure. Rinsing was carried out by gentle soaking of the cotton fabric and swirling at room temperature to remove impurities with a sufficient volume of water inside a small container. In this experiment, three dyeing and mordanting procedures were followed. First, dyeing without mordant, where a cotton cloth is immersed in dye at temperatures of 60 and 90°C for 45 min before it is dried and analysed.

In the absence of conventional mordants, sodium hydroxide (NaOH) was used to increase the solubility of the dyes as it acts like a colour modifier by changing the pH of its environment. However, it does not act as a mordant in the traditional sense of fixing the dye to the substrate. Second in line is mordanting before dyeing, where the cloth is treated with varying molarity (0.5, 1.0 and 1.5%) of NaOH solutions at 60 and 90°C for 30 min. It was then dyed for 45 min at the same temperatures and analysed after drying. Third, or last, in the list is dyeing followed by mordanting, where the cotton cloth was dyed for 45 min at 60°C and 90°C, dried, and then treated with NaOH solutions (0.5, 1.0 and 1.5%) for 45 min at the same temperatures. After drying, the fabric was stored for analysis. The treated cloth's weight was measured before and after dyeing, and various tests were conducted, including washing, exposure to sunlight, and manual rubbing, to evaluate its performance. ISO 105 methods for testing a few fastness properties were used, as followed by Periyasamy (2022).

RESULTS AND DISCUSSION

Extracted Dye

The procedure described above is summarized in *Figure 1*.

Figure 1 – Laboratory steps followed

As observed in *Figure 1e*, the orange peel was heated and filtered for the extraction of dye. It was found that the colour quality of the dye solution was comparable to that of powdered colouring materials because its colour did not change following the 30 min heating process, leading to high quality components. Orange peels were found to be the best plant part for extracting natural colour. Berry *et al.* (1971) advised that colour extract from the peel should be stored at ≤ 40°F (4.4°C) at all times. For sustainability, the abundance of oranges is a great merit for dye production, which would help reduce waste associated with its consumption and processing and spur economic growth.

Effect of pH

pH is a measurement of the acidity or alkalinity of a solution and it ranges from 0 to 14, with 7 being neutral. Values below 7 are acidic, while those above 7 are alkaline. The pH of the extract was measured in triplicate with the pHS-3C precise pH meter. *Table 1* displays the pH values of the 3 tests carried out.

Table 1 – pH level measurements

Sample	1st Test	2nd Test	3rd Test
Orange peels dye extract	3.34	3.36	3.34

A slightly acidic pH, showcased by *Table 2*, indicates that the sample is somewhat stable. The findings' dependability was further supported by the repeat measurements carried out in the laboratory. A pH of 3 was recorded previously by Che *et al.* (2024) as optimal after a response surface design run. Dyeing of fabrics at acidic pH was also reported by Baaka *et al.* (2017).

FTIR Functional Group

Absorption peaks are displayed in *Figure 2* with wavenumbers ranging from 4000-500 cm^{-1} and percentage transmittance from 40-80%. A broad, moderate and sharp absorption bands are located at 3446.9657 cm^{-1}. Sharp, moderate and weak absorption bands are located at 1728.2182 cm^{-1}, 1551.3697 cm^{-1}, 1360.6465 cm^{-1}, 1212.8404 cm^{-1}, 1156.1859 cm^{-1}, 1062.3111 cm^{-1}, 851.8788 cm^{-1} and 700.6141 cm^{-1}.

The frequency at which the IR radiation interacts with the sample is indicated by the wavenumber. The given range, spanning from 4000 to 500 cm^{-1}, includes a broad variety of frequencies that correlate to various chemical bonds. The existence of N-H stretching vibrations are indicated by the broad and moderate signal at 3446.965 cm^{-1}, pointing to the presence of compounds or molecular fragments containing amine or amide functional groups. A sharp moderate signal (2388.1051 cm^{-1}) suggests the presence of C≡C stretching vibrations, demonstrating the presence of certain alkyne functional groups in the dye. Sharp and weak signals at a wavenumber of 1728.2182 cm^{-1} show the existence of C=O stretch*ing* vibrations, which identifies compounds or functional group containing carbonyl groups (e.g., esters). A wavenumber of 1551.3697 cm^{-1} defines the presence of C=C stretching vibration and also describe either a low concentration or less intense vibration of these bonds. The value at 1360.6465 cm^{-1} was caused by stretching vibrations of N-O bonds within the nitro group (NO_2*)*. Those at 1212.8404 cm^{-1}, 11556.1859 cm^{-1} and 1062.3111 cm^{-1} signal C-F bonds, fluorine or other compounds containing fluorine. Bands such as, 851.8788 cm^{-1} and 700.6141 cm^{-1} indicate the presence of different C-H chemical bond bending vibrations in alkenes and aromatics.

Weight After Dyeing

The results of weighing cotton fabrics both before and after dyeing at various temperatures and concentrations of mordant are given in *Table 2*. ‘Plain cotton’ weight is the initial weight of the cotton (0.42 g) before any dyeing or treatment that is used for comparing weight changes following dyeing procedures. ‘Free-mordant dyed cotton’ refers to cotton dyed without any mordant and dyed at two different temperatures; the weight increased in the absence of a mordant, perhaps because of moisture or a little amount of dye adhesion. ‘Pre-mordant dyed cotton’ was dyed after being treated with a mordant at varying concentrations and varying temperatures; the weight increases with higher NaOH concentrations at both temperatures, suggesting that the NaOH was facilitating better dye absorption. ‘Post-mordant dyed cotton’ is similar to pre-mordant dyeing, but the cotton was dyed first and then treated with mordant at varying concentrations and temperatures; at higher temperatures there was a considerable increase in weight, which was particularly visible with the maximum concentration of NaOH, indicating improved dye absorption. Li *et al.* (2021) stated that, at a temperature > 110 °C, greater than the maximum used in this study (90°C) in *Table 2*, the orange peel pigment ceases to be stable. The optimum dyeing temperature for most textile fabrics is 100°C (Hou *et al.*, 2013).

It is evident that the weight of the fabric varies depending on the NaOH concentration and temperature. The data reveals a clear trend where increasing the temperature leads to an increase in the weight of the fabric after dye application. For the "free-mordant dyed cotton" material, fabric weights rose from 0.42-0.49 g at 60°C and further to 0.50 g at 90°C. Similarly, for the "pre-mordant dyed cotton" material, fabric weights increased with higher temperatures, reaching 0.520, 0.528 and 0.530g at 60°C for mordant concentrations of 0.5, 1.0 and 1.5%, respectively (*Figure 3a*). A temperature of 60°C is also the temperature tagged as optimum in Che *et al.* (2024) and Kim *et al.* (2014) during orange peel extraction and utilization as dye. At 90°C (*Figure 3b*), fabric weights were 0.521, 0.510 and 0.522 g for the same mordant concentrations. In the case of "post-mordant dyed cotton" material, fabric weights also showed an increasing trend with temperature, with weights ranging from 0.510-0.610 g at 90°C for mordant-like NaOH concentrations of 0.5-1.5%, as shown in *Figure 3 (c-d)*. These results suggest that higher temperatures likely enhance dye uptake and binding to the fabric, resulting in increased fabric weight post-dyeing. Such weight gains represent the dye and NaOH binding to the cotton fibres, enhancing colour fastness and the overall quality of the dyed textiles. It is found in Ivanovska *et al.* (2022) that pH, time, temperature and extract concentration affect the cloth colour depth, colour hues and colour fastness. Therefore, controlling temperature during the dyeing process is crucial for achieving optimal dye uptake and fabric properties.

Table 2 – Textile Weight Before and After Dye Application

S/N	Material	Acting-Mordant concentration (%)	Temperature	Weight (g)
1.	Plain cotton	-	-	0.42
2.	Free -mordant dyed cotton	-	60ºC	0.49
			90ºC	0.50
3.	Pre-mordant dyed cotton	0.5	60ºC	0.520
		1.0		0.528
		1.5		0.530
		0.5	90ºC	0.521
		1.0		0.510
		1.5		0.522
4.	Post-mordant dyed cotton	0.5	60ºC	0.510
		1.0		0.522
		1.5		0.530
		0.5	90º C	0.550
		1.0		0.540
		1.5		0.610

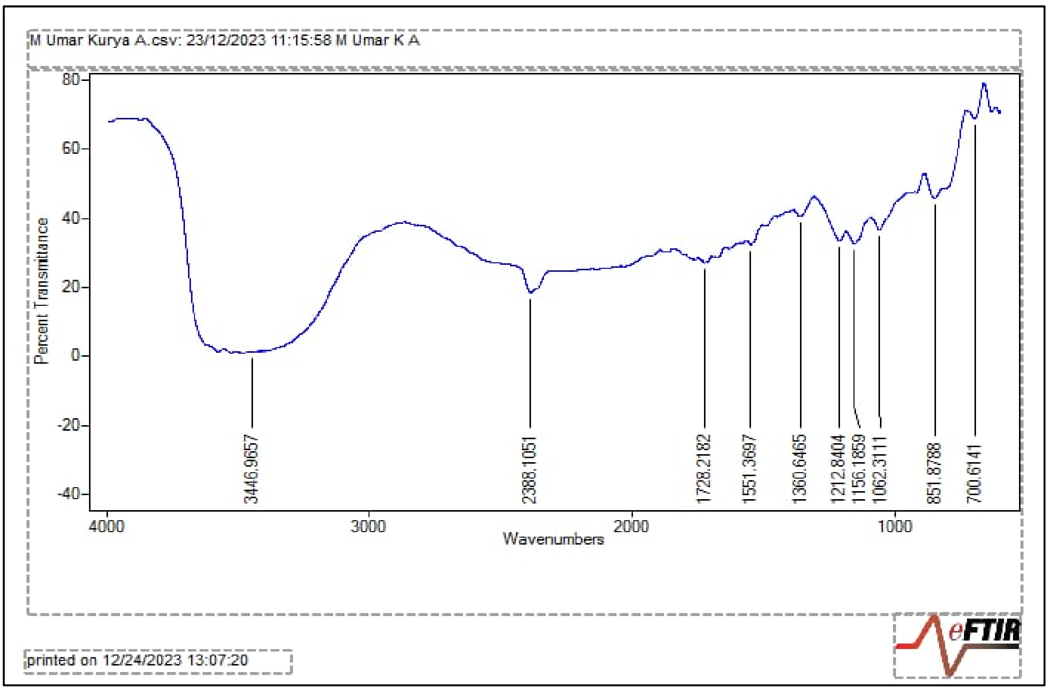

Figure 2 – FTIR spectra of liquid dye extracts

Data in *Table 2* also show that the weight of the fabric after dye application increases with increasing NaOH concentration. For the "pre-mordant dyed cotton" material, fabric weights increased from 0.520-0.530 g at 60°C and from 0.521-0.522 g at 90°C when the mordant/modifier concentration was increased from 0.5% to 1.5%.

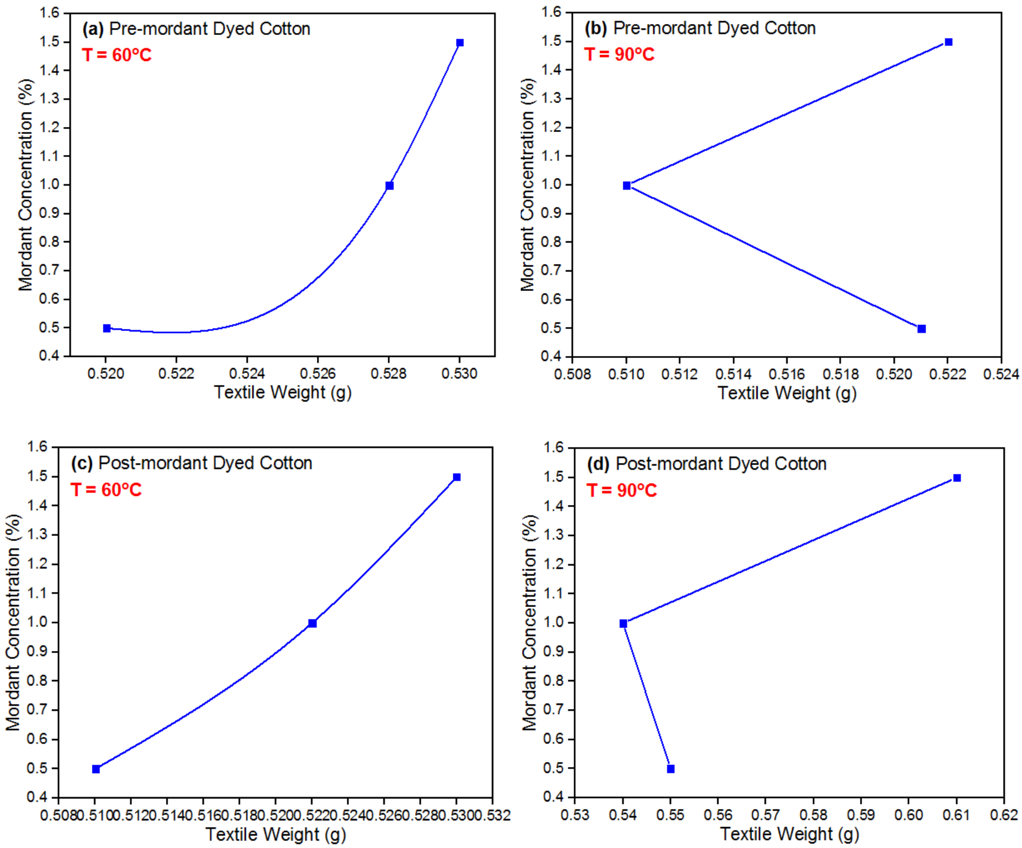

Figure 3 – Modifier concentration vs. fabric weight for pre- & post-mordant dyed cotton

Similarly, for the "post-mordant dyed cotton" material, fabric weights increased from 0.510-0.530 g at 60°C and from 0.550-0.610 g at 90°C when the mordant/modifier concentration was increased from 0.5-1.5%. These results suggest that increasing the NaOH concentration can enhance dye uptake and binding to the fabric, leading to an increase in fabric weight post-dyeing. However, it is important to note that excessive mordant/modifier concentrations can lead to negative effects on the fabric, such as reduced fabric strength and stiffness. Therefore, it is crucial to optimize the mordant concentration to achieve the desired dye uptake and fabric properties while minimizing any negative effects.

Effect of rubbing and sunlight on the dyed fabric

Rubbing did not affect the dyed fabrics in both cases. Islam *et al.* (2020) reported that fastness properties were not affected by the use of orange peel extract (*Citrus aurantium* Dulcis). Washing affected it in both cases in this study. This is because the cotton fabrics turned pale after washing. It was also found that the dyed cotton clothes were not affected by sunlight at all. The pale colour after washing indicates that the dye did not have good wash fastness, which is the ability of the dye to resist fading or washing out during laundering. A good dye should have good wash fastness, which means that it should retain its colour even after repeated washing. Since the study did not extensively evaluate the wash fastness of the extracted dye, it is unclear how well the dye performed in this regard. Given the fact that Kumar and Dhinakaran (2017)'s dye from orange and lemon peel gave better fastness to rubbing and washing, the property demonstrated by orange peel in the present study needs to be improved, as it is mildly satisfactory. It wouldn't be a surprise since Naveed *et al.* (2020) and Katyal *et al.* (2021) affirmed that natural dyes' drawbacks are their poor fastness property and low affinity for textile fibres (caused by weak bonding). In agreement with the current study, Edeen (2015) reported almost zero colour left on a cotton fabric washed at 60°C for 30 min. In this study, the level of adherence of the dye to the cloth, leading to colour changes from plain white, is shown in *Figure 4*.

By mere observation, it is difficult to distinguish the colour intensity of the fabric dyed in *Figure 4*. It can best be differentiated by measuring their colour strength.

Dyeing and Mordanting

The dye and the ingredients used for the dye are chemically bound by the mordant-acting chemical. This might also be the reason for colour alteration. To improve absorption and fixing efficiency and guard against fading from washing and light exposure, a mordanting chemical is utilized.

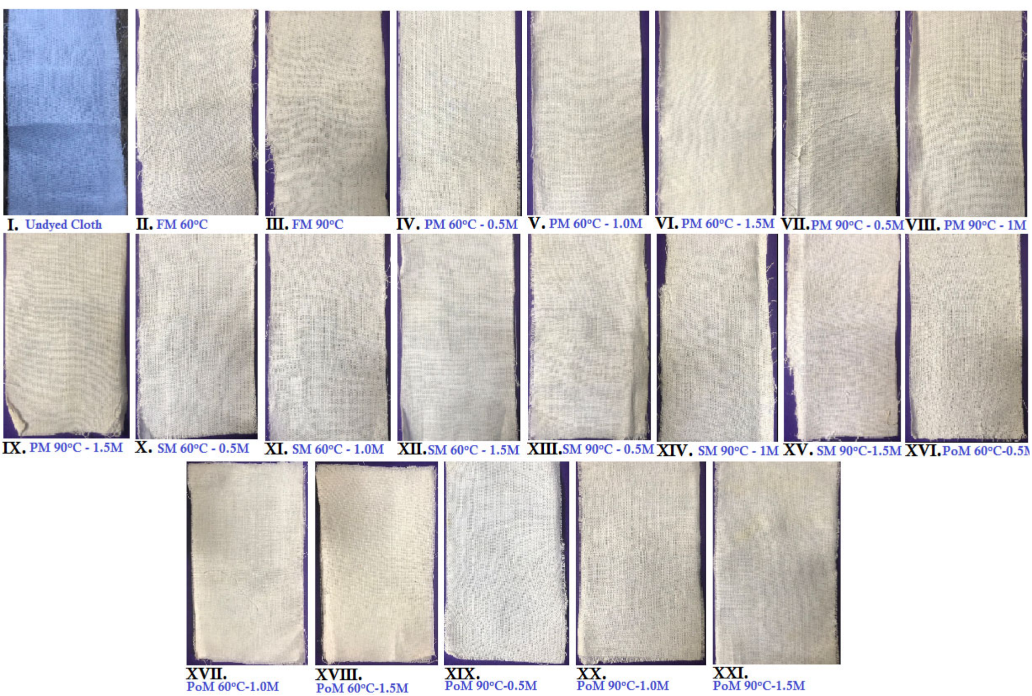

Figure 4 – Shades of colour on the cloth: I. plain cloth; II-III. free mordant; IV-IX. pre-mordant; X-XV. simultaneous mordant and XVI-XXI. post-mordant

Typically, NaOH was used as part of the dyeing ingredients both prior to and following the dyeing process (pre- and post-mordanting), as used on Tencel fabric (Naveed *et al.*, 2020). For instance, a honey-milk colour was observed using NaOH on white cotton material and dyeing it with *Citrus sinensis* peel dye solution. Additionally, cotton white fabric was first dyed with a citrus peel dye solution (post-mordanting) and then NaOH was used as a mordant in a subsequent step. Parikh *et al.* (2019) used NaOH to remove starch and impurities from the cloth while shades of yellow colour were observed by Tejavathi and Niranjan (2018) when different mordants were used on *Celastrus paniculatus* Willd dye extract.

CONCLUSIONS

Primarily, this study successfully extracted a natural dye from orange peel that was found partly effective for dyeing clothes. The extracted dye was characterized using FTIR, its pH was measured and a favourable pH within 3.34-3.36 was discovered. FTIR analysis identified various functional groups within the dye solution, such as N-H stretching vibrations, C≡C stretching vibrations, C=O stretching vibrations, C=C stretching vibrations, N-O bond stretching vibrations and C-H chemical bond bending vibrations in alkenes and aromatics. These functional groups are indicative of the chemical constituents present in the dye solution. Furthermore, the effects of temperature and acting-mordant concentration on cotton fabric weight during the dyeing process were evaluated. The data showed that higher temperatures and NaOH concentrations generally resulted in increased fabric weights after dye application, indicating enhanced dye uptake and binding to the fabric. Sunlight and rubbing effects are not influential in the dyed cloth, but only washing. The modifier-like mordant used (NaOH) proved to be effective at preventing quick fading of the dyed fabric. Other factors should be considered in future findings such as the dye stability, lightfastness and wash fastness. The effects of temperature-time profile, colour strength and actual use of mordant, as well as the liquor ratio were not considered in this study. To determine how environmentally friendly this natural dye is, a life cycle review comparing the effects of utilizing it vs. synthetic alternatives would be necessary.

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