Dyeing Properties of Cotton Fabrics using Extracted Dyes from Ethiopian and Congolese Coffee

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***ABSTRACT* -** A natural dye on textiles has gain significant importance for the reason that it was eco-friendly non-toxic, and increased environmental cognizance in order to avoid some dangerous synthetic dyes. In this study, the natural coloring substances such as Ethiopian and Congolese coffee (Coffee Arabica L) were useful to cotton fabrics, dyeing to both functionalize in addition to color the fabrics. The dyeing solution was prepared by extracting coffee grounds collected from Wad Medani Market-Sudan, they were extracted in hot water at 90 °C (raw coffee, medium-roasted and dark-roasted coffee). Also, the caffeine is separated and dyed with the coffee residues, the result was similar to the samples that were dyed with coffee. The coffee extract was used to Cotton fabrics using an infrared (IR) dyeing machine by conventional method at pH 8 for 90 min. The dyeing and surface properties of Cotton fabrics were investigated by Spectrophotometry and were characterized by Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR) to understand the performance of these natural dyes, respectively. The colors of the dyed Cotton fabrics obtained were found to be significantly light green for raw coffee, brown in appearance for medium-roasted and dark-roasted coffee, and their color fastness to washing and rubbing of dyed fabric with natural colorant extracts were compared. The results of fastness were found to be in the range of good to excellent. The dyeing properties of the color was found to be significantly reliable on the concentration of extracts and the degree of roasting of coffee.

Keywords: Coffee, Extraction, dye, Cotton, FT-IR.

المستخلص – الأصباغ الطبيعية ذات أهمية كبيرة لأنها غير سامة و صديقة للبيئة على المنسوجات نسبة لزيادة الوعي البيئي من أجل تجنب بعض الأصباغ الاصطناعية الخطرة والمسرطنة. في هذه الدراسة، اجريت صباغة الاقمشة القطنية بأصباغ طبيعية تم استخلاصها من نوعين من البن الإثيوبي والكنغولي الذى تم جمعه من سوق مدينة ود مدنى – السودان ، تم تحضير محلول الصباغة عن طريق استخلاص البن المطحون ، وتم استخلاصه في الماء الساخن عند 90 درجة مئوية (بن خام ، بن متوسطة التحميص وبن داكن التحميص) وايضا تم فصل الكافيين والصباغة بمخلفات البن وكانت النتيجة شبية بالعينات التى تمت صباغتها بالبن. تم وضع وغمر الأقمشة القطنية المبيضة في مستخلص البن في ماكينة صباغة بالأشعة تحت الحمراء(IR) باستخدام الطريقة التقليدية عند درجة الحموضة8 لمدة 90 دقيقة. تم فحص خصائص اللون والسطح للأقمشة القطنية باستخدام مقياس الطيف الضوئي، وتم تمييزها باستخدام مطياف الانعكاس الكلي المخفف لتحويل طيف الأشعة تحت الحمراء (ATR– FTIR)لمعرفة أداء وخصائص هذه الأصباغ الطبيعية. اظهرت الأقمشة القطنية المصبوغة اللون الاخضر الفاتح بشكل ملحوظ للبن غير المحمص، واظهرت النتائج اللون البني عند استخدام البن متوسطة التحميص واللون البنى الغامق للبن داكن التحميص، كما تم قياس ثبات اللون للغسيل والاحتكاك للاقمشة المصبوغة بمستخلصات البن المختلفة، وتم الحصول على نتائج ثبات اللون في المدى من جيد إلى ممتاز. وقد وجد أن هذه الخصائص للصباغة تعتمد بشكل كبير على تركيز المستخلصات ودرجة التحميص.

INTRODUCTION

Natural dyes recognized and used to as colorant a leather, a food substrate, a wood as well as natural fibers as main areas of application since ancient times. Natural dyes were not the same range of shades, and can be found from various amounts of plants including fruit, flowers, leaves, bark, and roots. In 1856 the beginning of widely available and inexpensive synthetic dyes having modest to excellent color fastness properties, there is a great interest in the use of natural dyes in textile coloration all over the world [1]. Several countries concerned of the toxic and reactions of synthetic dyes due to the imposed environmental standards. Also, natural dyes are eco-friendly in nature, smooth shades, not unsafe, attractive, creating highly and biodegradable. The fabrics dyed with natural dyes generally have weak fastness properties and can remain enhanced by applying metal salts as mordant [2].

Excellent color fastness identified via the metal ions usage a complex by the dye molecule; which is insoluble in water [3]. On the other hand, in recent times there has been revival of the growing interest on the application of natural dyes on natural fibers owing to worldwide environmental awareness. The natural dyes taking place of textiles eco-friendly and non-toxic has come to be a significance for the reason that of the increased environmental consciousness to avoid more hazardous synthetic dyes. On the other hand, the use of the natural dyes for the dyeing of textiles has generally be presented, a small scale of the dyes and the printers as well as small scale producers and exporters production by means of high valued eco-friendly textile production and sales [1].

Some studied using a natural dye for ordered basis dyeing and printing of textiles have started observing at the possibilities to overcome environmental pollution caused by the synthetic dyes [4].

Samanta, et al., [1] studied the Coffee is the second greatest traded commodity using volume after petroleum, indicative of the economic impact of coffee is substantial and it is just having about 7.4 million tons of which is produced for each year, is the maximum consumed affluence table beverage in the world. Coffee is one of the greatest popular beverages worldwide because is desirable taste and smell, in addition to its desirable properties, such as immune stimulation. Besides, coffee is the most important agricultural product in some countries in addition to the normal coffee beans created by the conventional approach. [5]

Some researcher studied extraction of Coffee are largely considered by different extraction methods and process. One more representative difference completed in Western societies is between an espresso and a lungo. Espresso using forcing hot water at high pressure, a lungo like espresso, extracted under high pressure using pouring hot water on ground coffee and after that filtering and using hot of water and coffee together for a period of time [6]. General studies of this type were carried out by Lo´pez-Galilea et al. [7, 8] and Peters [9].

The coffee was studied to take extended knowledge of the chemical, health and sensory related to coffee along the all-value chain. Moreover, to polyphenols, other natural substances such as caffeine, trigonelline and α-dicarbonyl compounds found in coffee and show antibacterial activity against S. mutans. The results including caffeine are controversial [10].

Lee KO et al. [11] studied the dyeing and finishing of wool fabric by extracted the spent coffee grounds prepared via the extraction method. A tannin was used as the natural of mordant in the fabrics to increase its functional group, such as antibacterial activity on the way to Gram-negative bacteria, and the coloring influence on the wool fabric.

This study, will focus on the use of coffee for dyeing fabrics using the extracted of two kind of coffee (Ethiopian and Congolese) prepared by the extraction method which extracted the caffeine from raw coffee, medium-roasted coffee and dark-roasted coffee using hot water, the structure of caffeine is shown as in Figure1. In particular, the coffees extract showed very good color fastness in the fabrics. The strong color appearance indicates that (Ethiopian and Congolese) coffee may be an effective material for fabrics dyeing. Lee et al (2013) [11] used FTIR technique to analyze natural dye using a minimum amount of sample.

The result of FTIR identifies the different fiber constituents and as well as the organic and inorganic dyes present in the fiber. The compound can be identified via comparing with the library of known material. [12] The interaction between fibers and dyestuff plays important role on stability of dyed fibers. The molecular structure change induced by Coffee natural dye can be identified by FTIR [13, 14].

This study also identifying and characterizing the peaks changes of cotton fabrics dyed with natural dyes by using FTIR.

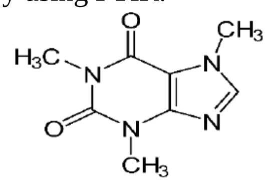

Figure 1: Structure of caffeine

MATERIALS AND METHODS

Materials and Equipment

Scoured and bleaching cotton fabric (weight 5 g/m^2, thickness 0.37 mm) for each sample, from Sur Military and Civil Clothing Factory. Green (raw) beans of Ethiopian and Congolese coffee were collected from Wad Medani Market. They were washed, dried under sunlight and all green coffee samples were roasted under different conditions. The sample was weighed up to 30 g and roasted as long as 20 and 30 mines with a temperature of 200 and 240 °C to medium-roasted and dark-roasted coffee respectively, until the volatile compounds inside the beans were evaporated.

The beans were prepared and ground separately into powder form. These powders were passed through a sieve to mesh size to obtain uniform particle size of the materials. These powder particles were used 25g to extract the corresponding dyes. For extracting of coffee dye three samples of commercial (Ethiopian and Congolese) raw coffee, medium-roasted and dark-roasted coffee were used.

Extraction method

The caffeine is first extracted from coffee using hot water by heating at 80 °C. This will take 30 minutes at a concentration of (30 g/L), cooling to room temperature, then it was filtered with filter paper. This aqueous solution also contains tannins and other water-soluble materials. The pH of each sample was measured at 20 °C. A total of 50 ml of coffee was titrated with 0.05 M NaOH.

The raw coffee solution was transferred to a separating funnel. 20 ml of auxiliary chemicals such as chloroform from Sd-Fine-Chem Limited (SDFCL) India were added, the separating funnel was gently shaken and opened every 5 seconds interval without pouring the solution only to release build up pressure produced by the gas released. The separating funnel was placed at rest for 1 minute to allow the density gradient of coffee solution and chloroform to be formed. The opening bottom of the separating funnel was opened to flow out the chloroform without allowing any coffee solution to be poured out into the conical flask.

The separated chloroform was then boiled in the conical flask to evaporate the solution until the precipitate is formed. The precipitate was put out on a piece of filter paper and weighted. The whole process was repeated using different materials of coffee. The chloroform solution was then dried, the solvent was evaporated, and the crude caffeine obtained was further being purified by careful recrystallization. The extraction method was prepared form Chemical Engineering and Chemical Technology Department Laboratory University of Gezira, Sudan.

Dyeing process

Dyeing was carried out in a dye bath containing 25 g of (Ethiopian and Congolese) raw coffee, medium-roasted and dark-roasted coffee in distilled water without any auxiliary.

The cotton fabrics were dyed using conventional method at pH 8, and the liquor ratio with the stock solution of the coffee extract are 1:10. Cotton fabric samples were cut into 15 cm x 15 cm pieces, each piece was immersed in the stock solution of the coffee extract, in a laboratory automatic dyeing machine (model 4-12; Tokyo, Japan). Fabrics were first immersed in the dye bath at room temperature and the temperature was then increased up from room temperature to 40 °C with a gradient of 2 °C/min which followed by increasing of the temperature up and held constant to 90 °C with a gradient of 1.25 °C/min for 60 min, and then cooled down to 30 °C with a gradient of 3 °C/min.

The dyeing profile is shown as in Figure 2. At the end of the dyeing process, the dyed samples were rinsed with water, fully washed and squeezed using padding rollers to obtain a wet pick-up rate of (80 wt %) and dried. The solution was used to determine the dye uptake. The dyeing process were prepared form Textile Engineering Department Laboratory University of Gezira, Sudan.

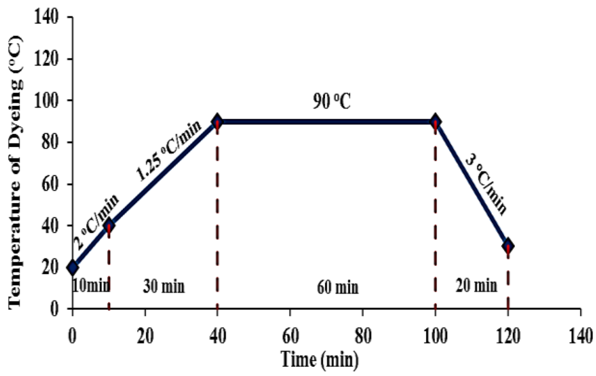

Figure 2: Profile of the dyeing process

Dyeability analysis

To determine the dye uptake used Spectrum lab. 725 S, visible spectrophotometer product standard No: QSEEK2. The wavelength at maximum absorbance (260 nm) for the dyes from (Ethiopian and Congolese coffee) raw coffee, medium-roasted and dark-roasted coffee respectively then measured the absorbance of the dyed solution. 50 ml dye solution was taken out from the dye baths and 5 ml was used for every time measurement. The %dye uptake (E) of dye solution was calculated by the equation below:

$$\mathbf{E} = \frac{\mathbf{A_o} - \mathbf{A_t}}{\mathbf{A_o}} \times \mathbf{100} \quad (1)$$

Where A_o and A_t are the absorbance of the dye solution at the starting and the finished of dyeing time.

Color fastness testing

Washing fastness*:* The washing color fastness was determined according to ISO 105-C01:1989 test method [15]. The washing fastness test was conducted in a laboratory launder tester (model DA-8 Tokyo, Japan) using 5 g/L nonionic detergent at 50 °C for 30 min, the liquor ratio 1:50. Then, removed the compound specimen by wetted with running tap water, squeezed, and then dried. It included the test specimen and the two adjacent fabrics in contact to the main sample. Gray scale was used to assess the color change of the dyed sample and the staining of the two adjacent undyed fabrics (cotton and polyester).

Rubbing fastness*:* The rubbing color fastness was determined according to ISO 105-X12: 1987 with a crock-meter under conditions for determination of dry and wet fastness [16]. Laboratory rubbing tester Marubeni Corporation. Textile machinery DEPT. model LA-375. Tokyo; Japan (Uenoyama Kiko CO. LTD). The washing and rubbing color fastness were prepared form Textile Engineering Department Laboratory University of Gezira, Sudan.

Fabric surface characterization

Fourier transforms infrared spectroscopy analysis

The Fourier transforms infrared spectro-photo-meter (FTIR) were recorded using IR Tracer-100 (EN230V) analytical FT-IR Spectrometer, SHIMADZU Company, Japan. The total reflectance of spectra was taken with a resolution of 4 cm^{-1} and accumulation of 32 scans for each undyed and dyed Cotton sample with a range of 500–4000 cm^{-1}, for consistency and reproducibility all spectra were normalized against the C-O-C asymmetric stretching vibration. From Mohamed Abied center Lab University of Gezira, Sudan.

RESULTS AND DISCUSSIONS

From the extraction test, the amount of caffeine extracted from (Ethiopian and Congolese) coffee powder was weighted, the raw coffee 0.34 g and 0.27 g, medium-roasted coffee 0.32 g and 0.33 g, dark-roasted coffee 0.26 g and 0.29 g respectively.

This indicates that there are some differences between coffee powder samples. Besides that, this experiment was carried out using the same number of grams of coffee powder to carry out a fair and equal result. As can be seen from the results of this experiment, 30 g of coffee contained 0.3 g of caffeine which has a higher caffeine content than 30g of roasted coffee which only contains 0.2 g of caffeine. From the beginning, it was aimed to compare the caffeine contents in different coffee types.

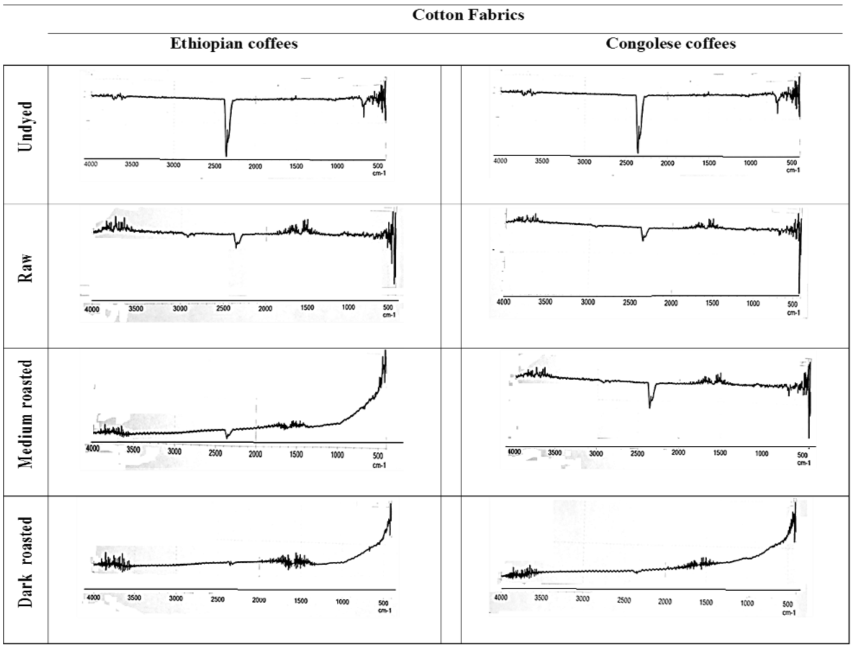

Figure 3. FTIR spectra of dyed Cotton fabric samples: undyed and dyed fabrics using Ethiopian and Congolese coffee extract.

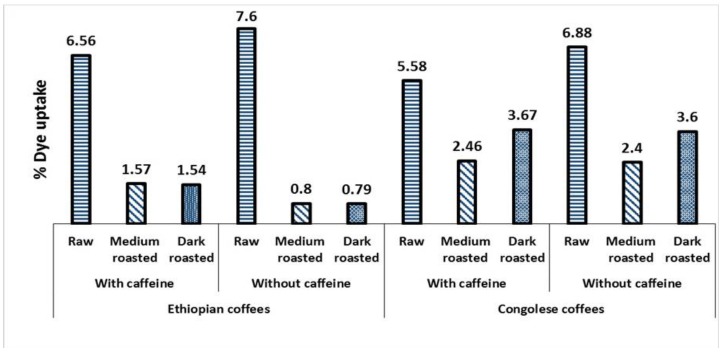

Figure 4: % Dye uptake rate of dyed cotton fabrics using Ethiopian and Congolese coffee extract.

ATR-FTIR spectral analysis

The (FTIR) spectra of the plain undyed cotton fabric, and fabrics dyed with (Ethiopian and Congolese) raw coffee, medium-roasted and dark-roasted coffee extracts are presented on Figure 3. From the figure it can be observed that the undyed cotton fabric presents a spectrum which is a typical cellulose structure. The cellulose spectral features are discussed somewhere else [17].

The comparison was done in the 4000cm^{-1} to 500cm-^{1} wave numbers because this is the spectrum where most of the chemical changes take place during modification of cellulose polymer. Cotton fabrics are made from cellulose fibers containing various functional groups, such as carboxyl (–COOH)) and hydroxyl (–OH) groups.

The FT– IR spectra of the cotton fabrics dyed with the coffee extract displayed similar absorption bands at 3500-3900 cm^{-1} peaks corresponding to the OH stretch. However, new peaks at 1100 cm^{-1} and 1500-1800 cm^{-1} were observed in the spectra of the cotton fabrics dyed with the coffee extract. This feature is unique and does not appear in the plain, un-dyed cotton fabrics. The peak at 2358 cm-1 on the undyed cotton fabric has weaker intensity after the dyeing process and disappeared after dyeing of the cotton fabrics.

From the spectrum, it can be speculated that the chemical functional bonding of the dye and cellulose has taken place. Further analysis is required to elucidate the functional groups involved in the chemical interaction between the dye and the cellulose.

Dyeing of Cotton fabrics with Ethiopian and Congolese Coffee

The wavelength for the control and the coffee dyed fabrics solution are changed from 260-400 nm. The slight change of wavelength indicates that there is a small shift in color for the dyed fabrics with coffee. Figure 4 shows the variation of color yield with varying different coffee before and after extraction with hot water for dyeing at 90 °C. The results show that the dye uptake for both Ethiopian and Congolese roasted coffee is very low, in the other hand the dye uptake of raw coffee is a slightly higher than that of all the roasted coffee. That means the roasted coffee acquire a good tint shade in the cotton fabrics.

Color fastness properties

Color fastness (Washing and Rubbing fastness) of fabrics dyed with (Ethiopian and Congolese) raw coffee, medium-roasted and dark-roasted coffee extracts are shown in Table 1 and Table 2 respectively**.** Color fastness was in the range of 3 – 5 ratings with the exception of fabrics dyed with coffee. The washing fastness of the cotton fabrics (control) and the dyed fabrics for both Ethiopian and Congolese coffee have shown no significant changes and they have shown good to excellent results, while the rubbing fastness obtained is moderate and good for dry rub fastness, but the wet rub fastness is found to be medium.

From Figure 4 through the results showed that the Ethiopian coffee dye uptake percentage and facial color depth of the cotton fabrics are higher than the Congolese coffee. However, as shown in Figure 5 and comparing the dye properties of cotton fabrics using Ethiopian and Congolese coffee extract dyeing also, the caffeine is separated and dyed with the coffee residues, the result was similar to the samples that were dyed with coffee. The amount of coffee that should brew depends on the degree of roasting. Stronger coffee will achieve a darker tint with the more coffee or a dark/very strong roast. A light or medium roast or less coffee showed a lighter tint. The coffee extract significantly colored cotton fabrics to greenish, brown and dark brown colors respectively via the dyeing process.bleta

CONCLUSION

Cotton fabrics were dyed with (Ethiopian and Congolese) raw coffee, medium-roasted and dark-roasted coffee extract. The caffeine is separated and dyed with the coffee residues; the result was similar to the samples that were dyed with coffee. The coffee extract was simply prepared and significantly colored cotton fabrics to greenish, brown and dark brown shades respectively via the dyeing process. Effect of color was enhanced by increasing the dyeing time and temperature. On the other hand, the rub fastness of the dyed cotton fabrics indicated that this natural dye is more stable to dry rubbing than wet rubbing conditions. FTIR analysis could not establish the dye chemical groups involved in the interaction with cellulose but it indicates the unique feature appears wave numbers of the FTIR spectrum of cotton fabrics dyed with the Ethiopian and Congolese coffee. More studies are required to investigate the nature of the dyes and their interaction with cellulose in order to optimize the dyeing conditions and performance of the dyes.

TABLE 1: RESULTS OF COLOR FASTNESS TO WASHING OF THE DYED COTTON FABRICS USING ETHIOPIAN AND CONGOLESE COFFEE EXTRACT.

Coffee Dyeing	With caffeine					Without caffeine			
	Color Change		White Fabrics Staining			Color Change		White Fabrics Staining	
			Cotton	Polyester				Cotton	Polyester
Ethiopian coffee									
Raw	5		5	5		4-5		4-5	5
Medium roasted	4-5		4-5	5		4		4	5
Dark roasted	4-5		4-5	5		3-4		3-4	5
Congolese coffee									
Raw	5		5	5		4-5		4-5	5
Medium roasted	5		5	5		4		4	5
Dark roasted	4-5		4-5	5		3-4		4	5

TABLE 2: RESULTS OF COLOR FASTNESS TO RUBBING OF DYED COTTON FABRICS USING ETHIOPIAN AND CONGOLESE COFFEE EXTRACT.

Coffee Dyeing	With Caffeine						Without Caffeine				
	Color Change			White Fabrics Staining			Color Change			White Fabrics Staining	
	Wet	Dry		Wet	Dry		Wet	Dry		Wet	Dry
Ethiopian coffee											
Raw	4-5	5		4-5	5		4-5	5		4-5	5
Medium roasted	4	5		4	5		3-4	5		3-4	5
Dark roasted	3-4	5		3-4	5		3	5		3	5
Congolese coffee											
Raw	4-5	5		4-5	5		4	5		4	5
Medium roasted	4	5		4	5		3-4	5		3-4	5
Dark roasted	4	5		4	5		3	5		3	5

Note: Washing fastness and rubbing fastness a staining on Polyester and Cotton:
5 refer to Excellent, 4 refer to Very good, 3 refer to Good, 2 refer to Fair, 1 refer to Poor

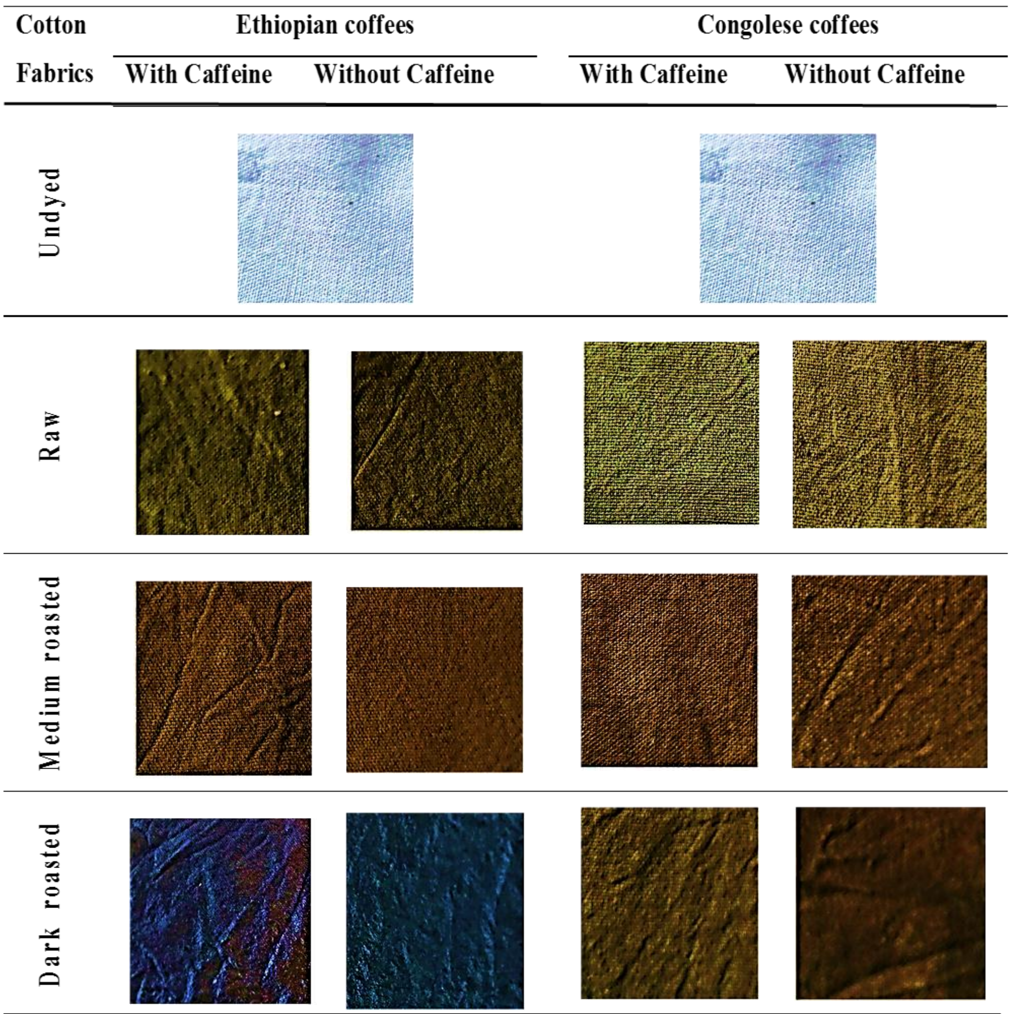

Figure 5. Photographs of dyed Cotton fabric samples: undyed and dyed fabrics using Ethiopian and Congolese coffee extract.

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