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Research Article

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Effects of Modification of Natural Dye Extracted from *Cissus populnea* stem Bark on Cotton Fabric

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Abstract Extraction, modification and testing of natural dye from *Cissus populnea* stem bark and its application on cotton fabric was studied. Modification of dye was carried out using 3, 7-bis (dimethylamino) phenothiazine-5-ium chloride and 2-(N, N-dimethyl)-4-aminophenyl. These gave a variety of colours as the pH tends to shift towards the acidic scale. Blue-violet-purple coloration was observed when 3, 7-bis (dimethylamino) phenothiazine-5-ium chloride was used as modifier and violet-red coloration was observed when 2-(N, N-dimethyl)-4-aminophenyl was used as modifier (absorbs at 592 nm to 540 nm). Wash fastness test was carried out with and without a mordant. It was obtained that, without a mordant, there was a fair dyed-cotton fastness but excellent dyed-cotton fastness was achieved using a mordant, i.e. the dye works best with a mordant than without a mordant. This research has create a scientific means of effecting color change on natural dye by either increasing or reducing the dye pH, hence improving the wash fastness of cotton fabric.

Keywords *Cissus populnea* , colour, cotton, mordant, absorption, fastness, textile

Introduction

The application of natural dyes on natural fibres and or fabric has witness growing interest due to environmental pollution [1]. The wide availability of synthetic dyes at low cost has been responsible for the decline in the utilization of natural dyes [2]. Although, natural dyeing of different textiles and leathers continued mainly to arts / crafts, small scale or cottage level dyers and printers as well as to small scale exporters and producers while industrial scale dyeing of specialty products such as in plastics, textile and garment industries, has seen the use of synthetic dyes [3]. Advantages of natural dyes such as its non-allergic, non-toxic, biodegradation and eco-friendly on textiles makes them suitable for consideration [4]. The numerous applications of synthetic dyes has witnessed researches in modification, dyeing process and other physico-chemical analysis. Recently, a number of commercial dyers and small scale textile exporters have started looking at the possibilities of using natural dyes for regular basis dyeing and printing. To obtain newer shades, colour fastness and colour yield, appropriate scientific techniques or procedures on methods, processing variables, kinetics and compatibility of natural dyes are required[5]. The research is aimed at the extraction, modification of natural dye from the stem of *Cissus populnea* plant and used in dyeing of cotton fabric. Effects of some physico-chemical parameters on dyed cotton fabric were investigated.

Materials and Methods

Potential dye-yielding plants namely *Cissus populnea* stem of the family *Amplidaceae* (*Vitaceae*) was collected in the month of August 2015 in Adamawa state in Nigeria. This plant was collected, dried and kept in a properly sealed and well labeled plastic container and was kept in the chemistry laboratory in ModiboAdama University of Technology, Yola.*Cissus populnea* stem bark, distilled water, sodium hydroxide, Liquor, 3,7-bis(dimethylamino) phenol thiazine-5-ium chloride, 2-(N,N-dimethyl)-4-aminophenyl, HCl acid, aluminium sulfat-18-hydrat, cotton fabric.

Preparation of reagents

- A 0.10% 2-(N,N-dimethyl)-4-aminophenyl was prepared by dissolving 0.10 g of the compound in 250 ml beaker containing 100 ml of alcohol (ASTM D1091)
- A 2% 3, 7-bis (dimethylamino) phenol thiazine-5-ium chloride was prepared by dissolving 2.0 g of the compound in 250 ml beaker containing 100 ml of distilled water. (ASTM D3694, 2011 Standard)
- A 10% Sodium hydroxide was prepared by dissolving 50 g of the compound in water, and dilute with water to 500 ml. (ASTM D1696, 2011 standard)

A 10% Hydrochloric acid was prepared by diluting 118 mL of the compound with water to 500 mL

Extraction of *Cissus populnea* stem bark dye

Hot method of extraction was used to extract the dye from the stem. The powdered stem bark of *Cissus populnea* (9 g) was detanned with acetone by percolation. The detanned crude stem extracts from *Cissus populnea* was added to 250ml of distilled water in a 400ml pyrex beaker. The mixture was3 stirred, heated and maintained and boiled for 3 hours, allowed to stand for 20 minutes and then filtered using a 24 cm filter paper. 200 mlof the coloured filtrate was used for dyeing the fabric in the presence of 10% (o.w.f) selected mordant[6].

Modification of dye

5 ml of 2-(N, N-dimethyl)-4-aminophenylsolution was added to 50 ml of extracted dye sample. This mixture was stirred till a homogenous phase is obtained. The pH was tested using a pH meter according to ASTM D 2867. The pH of 10 ml of the modified dye was adjusted using a 10% hydrochloric acid [7]. This was repeated for 3, 7-bis(dimethylamino) phenothiazine-5-ium chloride as modifier.

Measurement of absorption of the dye using UV-Visible spectrophotometer (D4951)

The identification of natural dyes in textiles involves selective extraction of dyes and comparison of each dye by various testing techniques, viz. UV-Visible and IR spectroscopy. The curve that was obtained provides a detailed description of the color properties of dyed samples with mordant was evaluated using standard procedures [7].

Treatment of cotton fabric (ASTM D 2259)

The cotton fabric was treated with sodium hydroxide. In this treatment of cotton fabric, cotton fabric was immerse into 50 ml of 10 % sodium hydroxide solution for 30 minutes and then it was dried set to undergo dyeing process [8].

Dyeing process

1.70 g of cotton fabrics was dyed using the simultaneous mordanting method at boiling for 1 hour with constant stirring using a fixed amount of liquor ratio (1:200). The dyed samples was thoroughly washed with cold and hot water to remove any unfixed dyed material and finally dried in open air [6].

Fixed-dye on cotton fabric

In this dye-fixed test, the treated cotton fabric was weighed *(Wp)*, immerse into 50 ml of the dye bath containing 0.2 g of aluminum sulfate and is heated at 100°C for 1 hour, then is cooled and dried (ISO 105-CO_2). It was wash with cold water then hot water to remove the unfixed dye, this cotton fabric was dried and then weighed *(Wf)*.The purpose and scope of this is designed to measure the amount of fixed-dye on the cotton fabric [9].

$$Weigh\ of\ fixed\ dye = \frac{Wf - Wp}{Wp} \times 100$$

Where W_p= *weight of pure cotton fabric*

W_f= *weight of dyed cotton fabric*

This was process was repeated at different temperature interval of 80, 60, 50 and 40 °C, these values recorded.

Effect of temperature on wash fastness

Wash fastness without mordanting agent was done by immersing 1.70 g (W_p) of the treated cotton fabric into 50 ml of the dye in the absence of a mordant (alum) and was heated to 100 °C for 30 minutes [10]. Then is cooled and dried. It was wash with cold water then hot water to remove the unfixed dye, this cotton fabric was dried and then weighed *(W_f)*.

$Weigh\ of\ fixed\ dye = \frac{Wf - Wp}{Wp} \times 100$Where W_p= *weight of pure cotton fabric*

W_f= *weight of dyed cotton fabric*

This process was repeated at different temperature interval of 80°C, 60°C, 50°C and 40°C values recorded.

Note; this whole process was repeated using aluminum sulfate as a mordant [9] and the values are recorded.

Effect of concentration on wash fastness test

Wash fastness test was carried out by dyeing 1.70 g of cotton fabric in 5/40 ml of the dye bath in the absence of a mordanting agent. The purpose and scope of this is designed to measure the amount of fixed-dye on the cotton fabric.

$Weigh\ of\ fixed\ dye = \frac{Wf - Wpure}{Wpure} \times 100$Where W_{pure}= *weight of pure cotton fabric*

W_f= weight of dyed cotton fabric

This process was repeated at different concentration of 10/40, 20/40, 30/40 and 40/40 ml, these values are tabled [9].

Note; this whole process was repeated using aluminum sulfate as a mordant [9] and the values are recorded.

Results and Discussion

Infra-Red chart for unmodified dye extract of *Cissus populnea* stem bark dye

Figure 2 shows the Infra-Red chart of dye Extract from *Cissus populnea* stem bark. Absorbance at 3268.9 wavenumber (cm-1) shows a strong = C-H of weak-medium intensity of an N-H symmetry and asymmetry stretch. Wavenumber 1636.3 cm^{-1} confirms the presence of Amide group R-NH_2 of a medium-Strong intensity with N-H bend. Stretching frequencies are higher than corresponding bending frequencies (it is easier to bend a bond then to stretch or compress it), this wave number also indicates the presence of an amine group of an N-H bend with a weak-medium intensity. This provides active functional group that react with the O-H functional group on the cotton fabric.

Figure 1: Cissus populnea stem bark powder

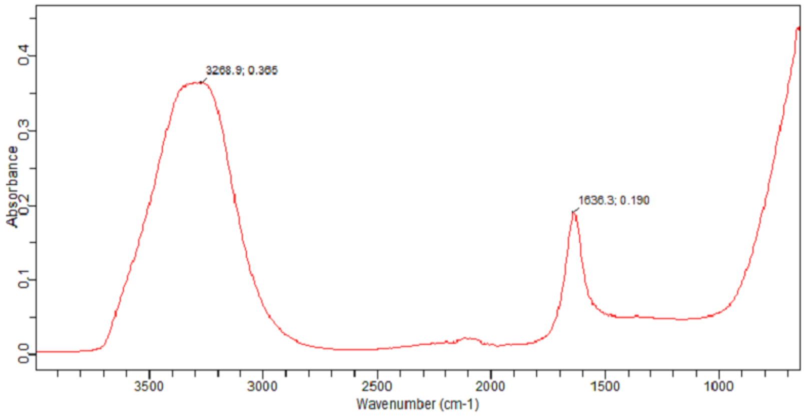

Figure 2: Infra-Red chart for Cissus populnea stem bark dye extracted using hot method of extraction

Effect of increase acid concentration when 3, 7-bis(Dimethylamino) phenothiazine-5-ium chloride as modifier

An increase in concentration produces more collisions. The chance of an effective collision goes up with the increase in concentration. 3, 7-bis (dimethylamino) phenothiazine-5-ium chloride reacts with anthraquinones. Figure 5 shows that as the acid concentration increases there was a shift in UV-Visible spectrophotometer colour absorbance (592 – 579 nm). This can be due to increase in intensity and colour strength [11].

Figure 3: modified dye using 3, 7-bis (dimethylamno) phenothiazine-5-ium chloride

Infra-Red chart for modified dye using 3, 7-bis (dimethylamno) phenothiazine-5-ium chloride

Figure 6 shows four transmittance peaks of modified *Cissus populnea* stem barkusing 3, 7-bis (dimethylamino) phenothiazine-5-ium chloride as modifier. 3268.9 cm^{-1} shows the strong C-H stretch intensity. It also indicates an amide group of weak-medium intensity of an N-H symmetry and asymmetry stretch. 1636.3 cm^{-1} confirms the presence of amide group R-NH_2 of medium-Strong intensity with N-H bend. This provides active functional group the will react with the O-H functional group on the cotton fabric.

Figure 4: Effect of pH on modified dye using 3, 7-bis (dimethylamno) phenothiazine-5-ium chloride

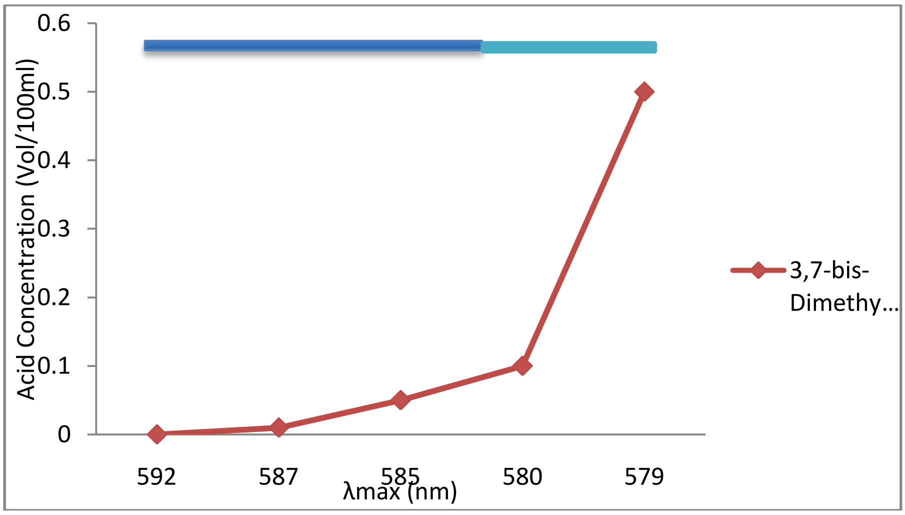

Figure 5: Effect of increase in acidic concentration using 3, 7-bis(dimethylamino) phenothiazine-5-ium chloride as modifier

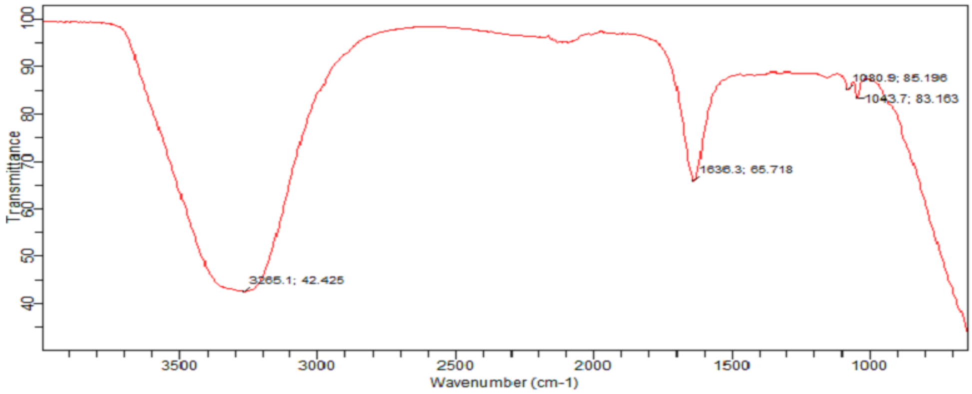

Figure 6: Infra-Red chart, modified dye with3, 7-bis(dimethylamino) phenothiazine-5-ium chloride as modifier

Effect of increase acid concentration using 2-(N, N-Dimethyl)-4-aminophenyl as modifier

The graph in figure 7, shows that as the acid concentration increase there was a decrease in UV-Visible spectrophotometer colour absorbance (560 – 490 nm). This can be due to the fact the intensity of the dye decreases as the pH increases. This indicates that there was a color change as the pH tends to drop towards the acid scale. 495-500 nm shows that Red color was observed, 505-545 nm a purple color was observed and violet.

Infra-Red chart, dye modified with 2-(N, N-Dimethyl)-4-aminophenyl

From figure 8, 3268.9 cm^{-1}shows the presence of alkynes with a strong C-H stretch intensities. It also indicate amide group of weak-medium intensity of an N-H symmetry and asymmetry stretch. 1636.3 cm^{-1} confirms the presence of amide group $R\text{-}NH_2$ of medium-Strong intensity with N-H bend. 1018 cm^{-1} shows the presence of an alkyl halide group of a very strong C-F stretching. This strong absorption intensity indicate that a strong covalent bond between the dye and the fabric was formed

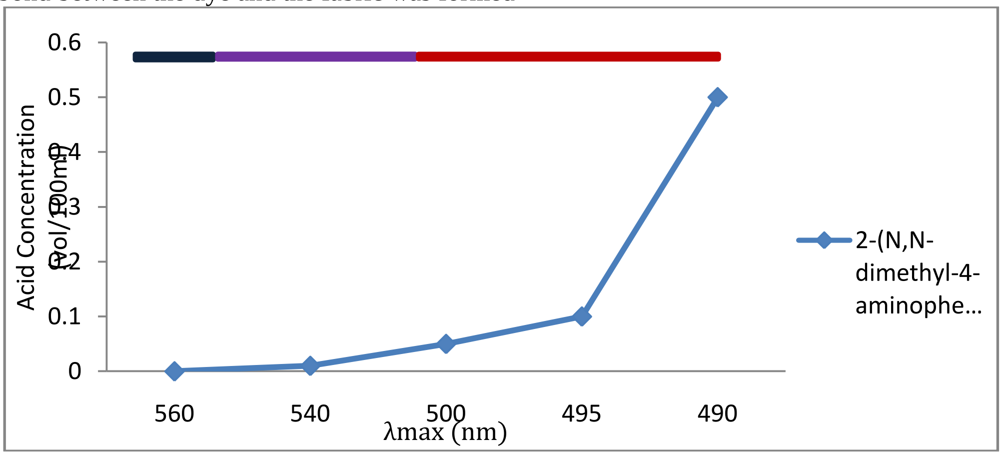

Figure 7: Effect of increase in acid concentration using 2-(N, N-dimethyl)-4-aminophenyl as modifier

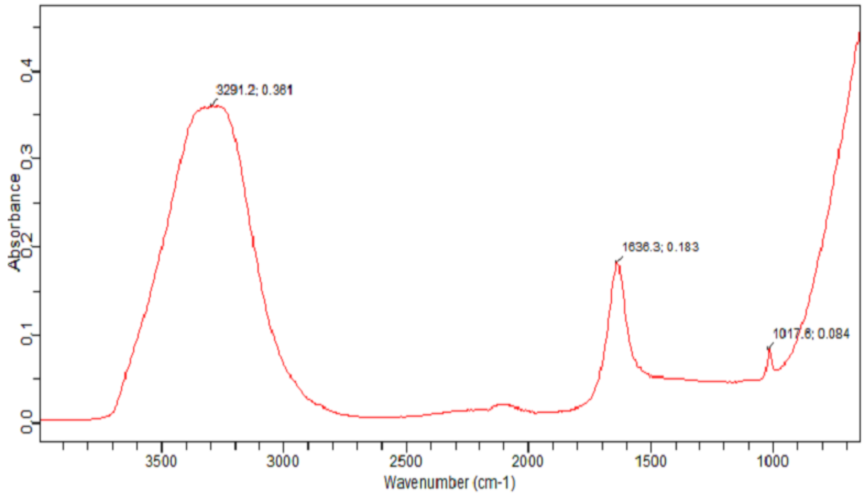

Figure 8: Infra- Red chart of dye Modified with2-(N, N-dimethyl)-4-aminophenyl as a modifier

Wash fastness test for unmodified dye extract and modified dyes on cotton fabric at different temperature

Wash fastness test of unmodified, modified dye using3,7-bis(dimethylamino) pheno thiazine-5-ium chloride and 2-(N,N-dimethyl)-4-aminophenyl at different temperature is shown in figure 10, shows that, dye-fixation of the pure dye extract is less compared with 3,7-bis(dimethylamino) phenothiazine-5-ium chloride and 2-(N,N-dimethyl) -4-amino phenyl. This shows that the unmodified dye has a low affinity for the cotton fabric. 3, 7-bis (dimethylamino) phenothiazine-5-ium chloride achieves the highest dye-fixation compared with others. This may due to more reactive sites on the 3, 7-bis(dimethylamino) phenothiazine-5-ium chloride compared with others and also has high affinity for the fabric. It also shows that as the temperature increases the dye – fixation of each increase , this implies that temperature favors in dye-fixation, this also may due to Collisions between the cotton fabric (Cell-OH) and the dye (Dye-NH_2) was more violent at higher temperatures [12]. The higher temperatures mean higher molecular velocities this means there was less time between collisions. The frequency of collision was increase. The increased number of collisions and the greater violence of collisions results in more effective collisions. The rate for the reaction increases.

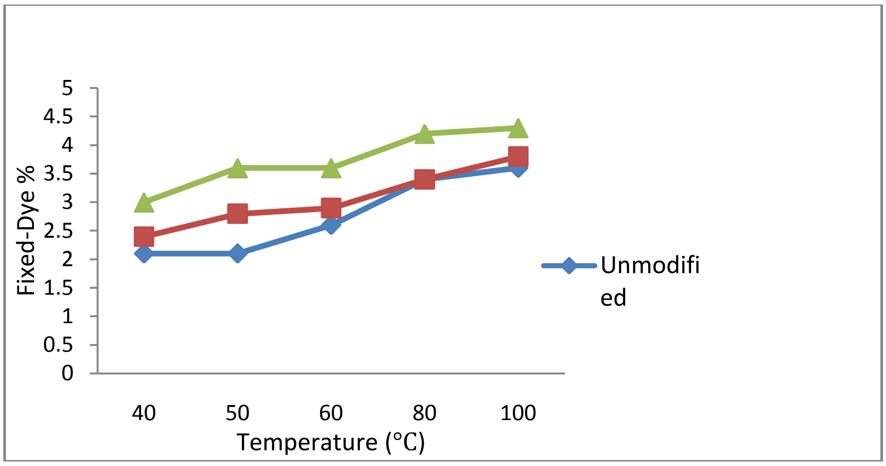

Figure 11: Effect of temperature on fixed-dye "with" a mordant and "without" mordant

Effect of temperature on fixed-dye "with" a mordant and "without" mordant

Figure 11: shows the effect of temperature on dye-fixed with a mordant and without a mordant. Those without mordant (aluminium sulfat-18-hydrate) shows a lower dye-fixation on the cotton fabric compared to the one having aluminium sulfat-18-hydrate. Although there was a bit increase of dye-fixed on the cotton fabric as the temperature increase (1 – 2.2 %). The kinetic energy and molecule velocity increase with temperature. $KE = [1/2][mv^2]$. The mass of the molecules is "m" and the velocity is "v". Covalent bonds are the strongest chemical bonds and are formed by the sharing of a pair of electron. Cotton fabric dyed with a mordant shows a high dye-fixation on the cotton fabric, this may be due to the metallic salt forming complex [13]. This indicates that the dye works better with aluminum sulfat-18-hydrate as mordant.

Effect of concentration on fixed-dye "with" a mordant and "without" a mordant

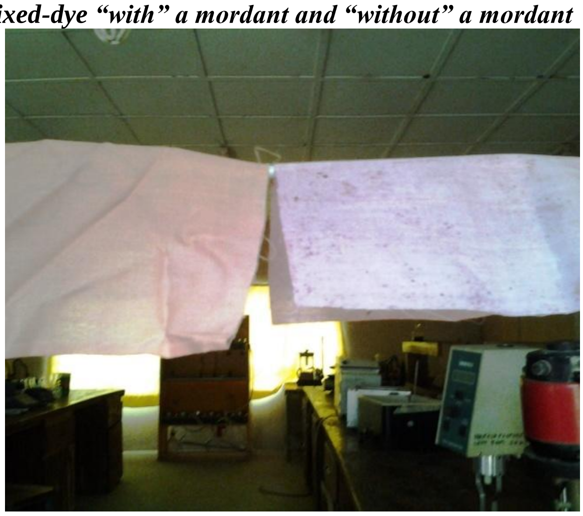

Figure 12: Effect of concentration on fixed-dye "with" a mordant and "without" a mordant at 100^{o}C

Figure 13 shows the effect of concentration on fixed-dye with aluminum sulfat-18-hydrate as mordant and without a mordant. Concentration is the amount of solute per volume of solvent. An increase in concentration produces more collisions. The chances of an effective collision goes up with the increase in concentration [15]. The exact relationship between reaction rate and concentration depends on the reaction mechanism. There was a decrease in dye active bond sites reduces. The effect of concentration on fixed-dye with aluminum sulfat-8-hydrate as mordant shows a higher fixed-dye compared to dye-fixed without mordant. 3, 7-bis(dimethylamino) phenothiazine-5-ium chloride show the highest dye fixed on the cotton fabric at 4.0 %. This implies that more fixed-dye is achieved on the modified dye compare to pure dye extract [16]. Effect of fixed-dye on cotton fabric without aluminum sulfat-8-hydrate as a mordant show a little dye fixation on the cotton fabric. The modified dye shows a better dye uptake than the corresponding pure dye extract [17].

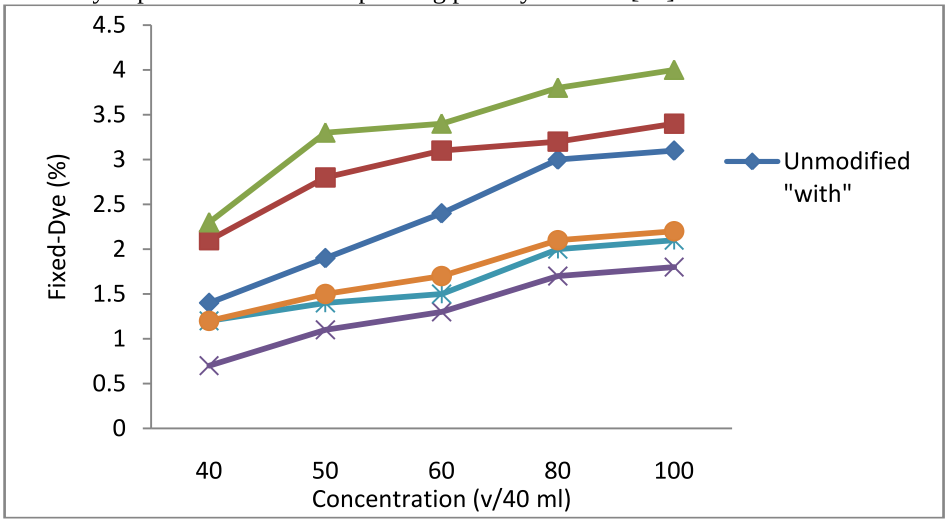

Figure 13: Effect of concentration on fixed-dye "with" a mordant and "without" mordant

Conclusion

The effect of pH on modification of the dye indicates that, the pH of the dye shift towards a lower λ-max if acidic scale is favored. Fixed-dye test was carried on a cotton fabric, this Test reveals that the dye works best with a mordant and this mordant favors the uptake of the modified dyes compared to the unmodified dye extract. The dye obtained displays fairly good saturation on cotton with medium to good fastness properties. This result shows that modification of *Cissus populnea* stem bark dyes can be done using 3,7-bis(dimethylamino) phenothiazine-5-ium chloride and 2-(N,N-dimethyl)-4-aminophenyl which gives a good fixed-dyeing properties these includes; good wash fastness also.

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