



Synthesis and Application of Dye Derived from *Caesalpinia Coriaria* (DIVI-DIVI) and Carbon Steel Waste Extract on Cotton

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ABSTRACT

Dye substances produced from the reaction of *Caesalpinia coriaria* (Divi-divi) and carbon steel waste extract, were assessed using FTIR, UV-Visible spectra, and fastness test on wash fastness, light fastness, rub fastness and fastness to ironing. Characteristic group absorption within 3200 cm⁻¹ – 3400 cm⁻¹ indicate hydrogen bond OH absorption usually broad while others in region 2900 cm⁻¹ – 2000 cm⁻¹ are attributed to CH₃, CH₂, CH, C=C stretch, and 1640 cm⁻¹ – 1707 cm⁻¹, 1440-1640 cm⁻¹ are attributed to C=O stretch and C=C stretch respectively. The dye bath containing formic acid has a wavelength of 599 nm and an absorbance of 1.447 while the wavelength of *Caesalpinia coriaria* in an aqueous medium is 450nm and absorbance of 1.113. The wash fastness is 5, light fastness is 5, rub fastness for both wet and dry is 5, and fastness to ironing for wet and dry is 5. The following numbers represent the following: 5 for excellent, 4 for very good, 3 for good, 2 for fair and 1 for poor according to the grey scale for change of colour. The above result shows that the dye has a good fastness property and that it is suitable for application on cotton.

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INTRODUCTION

Dye is a substance that is used to change the colour of things such as cloth, hair or leather. It could be hair dye, black dye, natural dye or vegetable dye. Dyes possess colour because they absorb light in the visible spectrum (400 – 700nm), have at least one chromophore (colour bearing group) also have a conjugated system i.e a structure with alternating double and single bonds, and exhibit resonance of electrons, which is a stabilizing force in organic compounds (Abrahart,1977). When any one of these features is lacking from the molecular structure the colour is lost. In addition to chromophores, most dyes also contain groups known as auxochromes (colour helpers) examples of which are carboxylic acid, sulfonic acid, amino and hydroxyl groups. Dyes are soluble in water and or an organic solvent, it is used to colour substrates to which they have an affinity (Aspland, 1997).

Dyes could be either obtained from natural or synthetic sources. They are produced either chemically or from plants, unlike paints; they do not build up on the surface of the fibre or hide but are absorbed into the pores of the materials. This is becoming possible because the size of the dye molecules is smaller than the

size of the pores in the fibre. The dye molecules have a shape like arrow strips of paper. This planar shape assists them to slip into the polymer system when the fibre yarn or fabric is introduced into the dye bath (Choudhary, 2006). Research has shown that dyes can be obtained from either natural or synthetic sources they are produced either chemically in the laboratory or from a plant which is present in many types of plant material (Brewster 1948).

MATERIALS AND METHOD

Collections of Samples

The carbon steel waste was collected from Mechanical Engineering Department Ahmadu Bello University Zaria. The *Caesalpinia coriaria* (Divi-divi) was collected from a Tannery in Nigerian Institute of Leather and Science Technology and was crushed using a mortar and a pestle and a sieve was used to filter the crushed *Caesalpinia coriaria*.

Soaking of the Carbon Steel Waste

289g of carbon steel waste was soaked in 900ml of water and 100ml of tetraoxosulphate (vi) acid was also added to it, for a period of two weeks to hasten the rusting process, after which the carbon steel waste was



removed from the solution. The same procedures were used to soak the carbon steel waste using 900ml of distilled water and 100ml of formic acid.

Dyeing Trial using together mordanting

2.5g of Cotton fabrics were cut using succour and placed into a dyebath containing 10g tannic acid and 50ml of the mordant of carbon steel waste containing, the tetraoxosulphate (vii) acid cotton was agitated in the dyebath for 1 hour, after which the dyed fabric was fixed using a diluted formic acid of 0.05M. The fabric was then removed from the dyebath, rinsed with cool water and dried under a shade. The same procedure was used for seven different cotton samples. The volume of the solution varied using 60, 70, 80, 90, 100, 110, and 120ml respectively. The above procedure was used to dye the cotton, using the mordant containing carbon steel waste and formic acid.

Wash Fastness Test

The dyed sample was subjected to a wash fastness test via the following procedures: the specimens were prepared by cutting the dyed fabric into 4cm for both length and breadth; it was then made into composites by stitching the tested specimen made of the dyed sample placed in between white cotton at a dimension of 10cm for length and 4cm for breadth. The composite was agitated into the solution made up of the following: Soap Solution 5g, Sodium Carbonate 2g, Liquor ratio 50:1. The washing was maintained at 50°C for 45 minutes with continuous agitation. At the end of the washing test, the composite specimen was removed, rinsed in running tap water and the components were separated and dried at room temperature. The change in colour of the dyed sample and the staining of adjacent and dyed cloths were assessed using the appropriate grey scale.

Light Fastness Test

The dyed sample was exposed facing due south where the exposure is being made. Adequate ventilation of the sample during exposure was ensured. The partly covered sample was exposed to UV- radiation. Exposure proceeded for 74 hours, then the sample under test was examined at the interval and the change in colour of the sample was compared visually with the changes that occur in the

standard. The light fastness of the sample is the number of the standard that showed a similar visual contrast between the exposed and unexposed parts of the sample. The exposure was terminated when the exposed and unexposed test sample is equivalent to the grade on the grey scale.

Rub Fastness Test for wet and dry

The standard pad (felt) was immersed in solving water and allowed to cool. The water was squeezed out and cold water was quickly added to felt pads before seeing used for the test. The dyed sample and the felt pad were placed in the machine accordingly and the machine was switched on after some revolutions of the felt pad on dyed cotton, the machine was switched off and the felt pad was removed and replaced with a new one. The dyed cotton sample was also removed and a new sample was replaced for the test. The test was carried out and the sample was assessed after the 1000 revolution. The specimen was allowed to dry and change in colour of the dyed fabric cotton and the staining of the felt pad was not immersed in water, it was used dry.

Fastness to Ironing for Wet and Dry

For the wet fastness to ironing the dyed sample was immersed in cooled water and was ironed using an electrical iron the sample was ironed to dryness. After which a grey scale for colour change rating was used to know the colour change. The above procedure was used for the dry sample.

Ultra Violet- Visible Spectroscopic Analysis

1g of the dyed sample was weighed using an analytical weighing balance and dissolved in 10cm³ distilled water in a test tube. Each of the dye solutions was diluted in the following order; 1cm of the dyed sample was measured in each case into a different test tube and 9cm of distilled water was added to each test tube. UV-Visible spectrophotometer (Jenway 6305 spectrophotometer) was used to determine the different absorbance of the different dye samples at different wavelengths from 400-800nm at an interval of 10 minutes. The tested fabric was placed between two specified pieces of undyed cloth measuring 5cm by 4cm, and the three-piece was held together by stitching around the edge and was soaked in water for 30 minutes. The change in colour of

the tested specimen and the staining of the adjacent undyed clothes will be assessed.

RESULTS

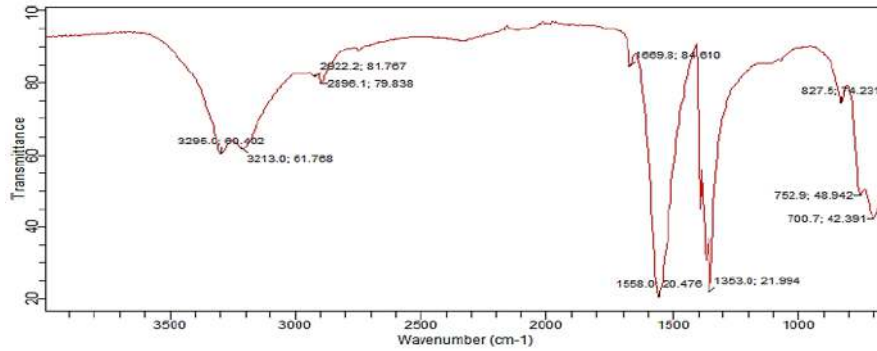


Figure 1: FTIR on Pure Carbon Steel Waste Extract in Aqueous Medium

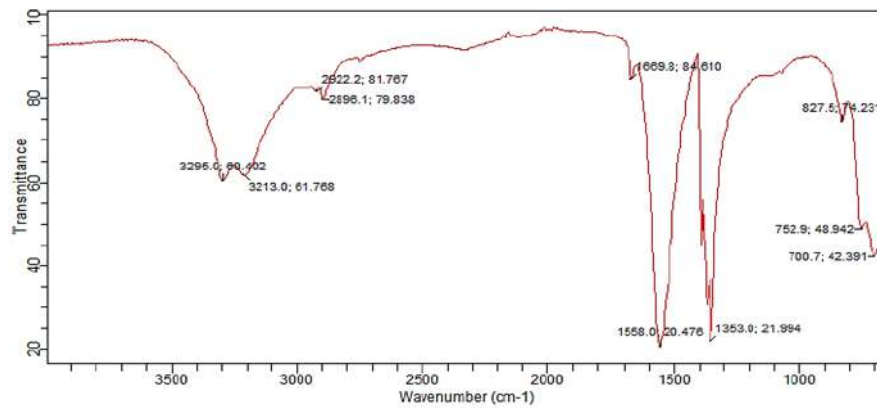


Figure 2: FTIR of Pure Carbon Steel Waste Extract Containing Formic Acid

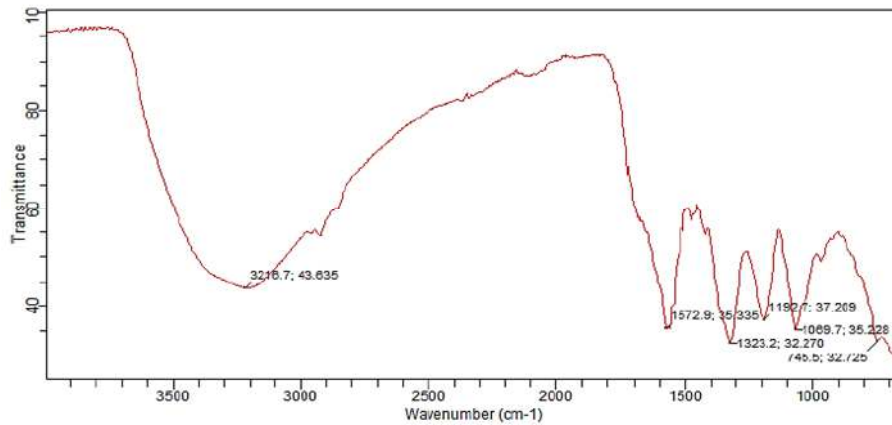
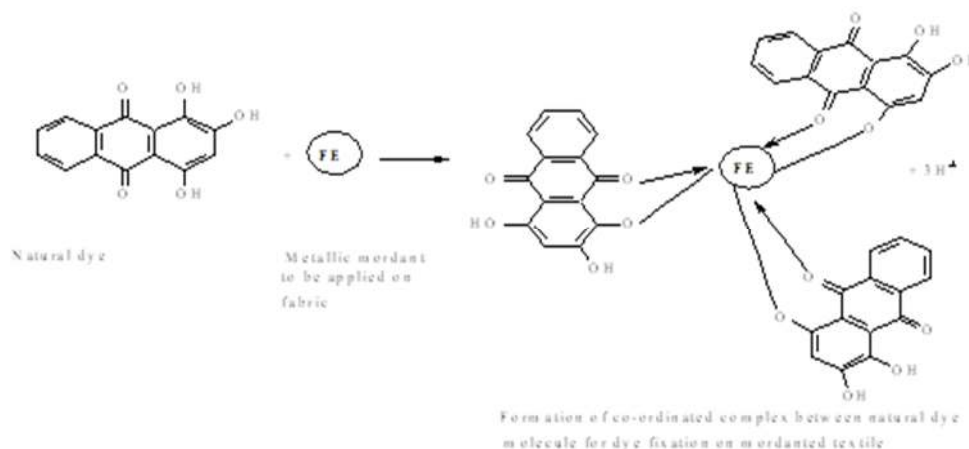


Figure 3: FTIR of Carbon Steel Waste Extract, Natural Acid Dye and Formic acid



Scheme 1: Equation for the Reaction between Natural dye and Metallic Mordant

DISCUSSION

The infrared spectrum of the pure carbon steel waste extract in an aqueous medium shows the presence of OH stretch which can be seen to be at 3280-1cm-1 typical of phenolic compounds, while a subsequent peak at 2922.2-2855.1cm-1 can be assigned to C-H stretch and peak at 2105.9 can be assigned to C≡C stretch which is an alkyne. The weak peak observed at peak 1576-7 cm-1 can be assigned to C=C stretch while the peaks at 1457.4-1375.5 cm-1, and 931.8 cm-1 are attributed to C-H deformation in CH2 respectively have shown the presence of chromophores which are C≡C, C=C and the auxochrome which is the O-H. From the infrared spectrum result on carbon steel waste extract with formic acid, the OH stretch can be seen to be at 32950-3213 cm-1 which is a phenolic compound, while 2922.2 cm-1 and 2896.1cm-1 is attributed to CH3, CH2, C-H stretch and 2896.1cm-1 is attributed to -C-H stretch which is an aldehyde.

The weak peak observed at 1669.8 is attributed to carbonyl C=O and the peak at 1558.0cm-1 is attributed to C-C stretch, 1353.0 cm-1 can be observed at O-H deformation. The peak observed at 827.5 cm-1, 700.7-752.9 cm-1 is attributed to C-H deformation respectively has shown the presence of the carbonyl group and the auxochromes. The infrared spectra of a dye substance produced from the reaction of natural dye, carbon steel waste extract and formic acid, the broadband at 3216.7 cm-1 is an OH stretch. The other peaks at 1572.9 cm-1, 1323.2 cm-1, 1192.7 cm-1, 1069.7cm-1 and

745.5 cm-1 are observed to be carbonyl C=O stretch and C-H deformation respectively. From the FTIR spectrum of the reaction of tannic acid, carbon steel waste extract and teraaxosulphate (vii) acid, the O-H stretch can be seen at peak 3213.1cm-1 which is a phenolic compound, the weak peak at 1610.2-1610.2 cm-1 are assigned to C=O stretch and C-H deformation respectively. The presence of the carbonyl group and the auxochromes whose function is to intensify colour.

From the infrared spectrum of a solution of carbon steel waste with teraaxosulphate (vii) acid, the O-H stretch can be observed at 3201.8 cm-1 and 2922.2 is assigned to the C-H stretch, the peak at 2102.2 cm-1 is assigned to C≡C stretch. The weak peaks observed at 1461.1cm-1, 1375.4 cm-1 and 1073.5cm-1 are attributed to C-H deformation, O-H- deformation and C=O stretch respectively have shown the presence of the chromophores which gives the dye a potential colour, the auxochrome which intensifies colour and the carbonyl group. From the infrared spectrum of a *Caesalpinia coriaria* (Divi-divi) in an aqueous medium, the O-H stretch can be observed at 3183.9 cm-1 and 1708.4 cm-1 is assigned to the C-H stretch, the peak at 1623.5 cm-1 is assigned to C=O stretch.

The weak peaks observed at 1448.2 cm-1, 1312.0 cm-1 and 1080.9 cm-1 are attributed to O-H deformation and C=O stretch respectively have shown the presence of the chromophores which gives the dye a potential colour, the auxochrome which intensifies colour and the carbonyl group. The UV-visible spectra



indicate the wavelength of maximum absorption (λ -max) of the dye substance produced from the reaction of carbon steel waste extract, natural dye and the application on fabric. The results show that the dye produced from the reaction of natural dye, carbon steel waste extract and formic acid has a λ -max of 599nm with absorbance at 1.447 showing that there is a hypsochromic shift which is a change to a shorter wavelength (higher frequency) and the visible colour is green-blue.

Table 1 shows the result of fastness to light wash wet and dry, rub wet and dry, ironing wet and dry properties of the dye substance produced from the reaction of natural dye, carbon steel waste and formic acid and application of fabric. The wash fastness properties of the dyed cotton for both wet and dry show that the dyed fabric has very good wash fastness properties according to the rating which is 3 for both wet and dry. The transfer of colour to the felt for drying is 1/2 and for wet, the transfer of colour to the felt is 1/2 according to the grey scale rating for transfer of colour. The light fastness properties of the dyed fabric after exposing the dyed cotton to light for 72hours, the result shows that the dye has very good light fastness properties according to the rating using a grey scale which is 4.

The rub fastness properties of the dyed fabric for wet is 3 which indicates that the dyed cotton has a very good rub fastness according to the grey scale rating, and the transfer of colour to be felt is 1 according to the great scale rating for transfer of colour. The rub fastness properties of the dyed fabric for dry is 5 which indicates that the dye has excellent rub fastness properties and for transfer of colour to be felt is 1/2.

The fastness to ironing properties of the dyed fabric for wet is 5 according to the grey scale rating which indicates that the dye has excellent fastness to iron properties, while that of the dry according to the grey scale rating is also 5 which also indicates that the dye has excellent fastness iron properties. Fastness properties of the dye substance produced from the reaction of natural dye, carbon steel waste extract containing teraoxosulphate (vii) acid. The wash fastness properties of the dyed fabric show that the dye has very good wash fastness properties according to the grey scale rating which is 4, and for transfer of colour to be felt is 1 according to the grey scale for transfer of colour to be felt for both dry and wet, which indicates that the dye has a very good wash fastness property.

The light fastness properties of the dyed fabric after exposing the dyed fabric to sunlight for 72hours the result shows that the dye has very good light fastness properties according to the grey scale rating which is 4. The rub fastness properties of the dyed fabric for both wet and dry according to the grey scale rating which is 5, indicates that the dye has an excellent rub fastness property for both wet and dry. And for transfer of colour to be felt for wet is 1 and that of dry is 1/2 according to the grey scale rating for transfer of colour to be felt. The fastness to ironing properties for the dyed fabric for both wet and dry according to the grey scale rating is 4 indicates that dye has an excellent fastness ironing property for both wet and dry samples.



Table 1 Fastness Properties of the Dye on the Textile Fabric (Cotton)

Dye	Colour	Colour on Substrate	Wash Fastness wet	Wash Fastness Dry	Light Fastness Rating	Rubbing Fastness Wet	Fastness to ironing Wet	Fastness to Ironing Dry	Rub Fastness Dry
Natural dye, Carbon Steel waste extract and Formic acid	Navy Blue	Navy Blue	4	4	4	4	5	5	5
Natural dye, Carbon Steel waste extract and Tetraoxosulphate (vi) acid	Navy Blue	Navy Blue	4	4	4	5	5	5	5

CONCLUSION

In this research, the production of dye substance from the reaction of natural dye with carbon steel waste extract was successfully carried out. The infrared spectra of a dye substance produced from the reaction of natural dye, carbon steel waste extract and formic acid show the presence of chromophores, auxochromes, and phenolic compounds. The FTIR spectrum of the reaction of tannic acid, carbon steel waste extract and tetraoxosulphate (vii) acid also shows the presence of chromophores, auxochromes, and phenolic compounds.

The UV-visible spectra show that the dye produced from the reaction of natural dye, carbon steel waste extract and formic acid has a λ -max of 599nm indicating a hypsochromic shift in the dye and that of *Caesalpinia coriaria* (divi-divi) is 450nm. It can be deduced that the λ -max in each of the dyed substances is a result of the π - π transition in chromophores which occurs within the visible region of 400-800nm, which indicates the degree of conjugation in the

dye molecule. The fastness to light wash wet and dry, rub wet and dry, ironing wet and dry properties of the dye substance produced from the reaction of natural dye, carbon steel waste and formic acid showed that the dye exhibits excellent fastness properties.

The Fastness properties of the dye substance produced from the reaction of natural dye, carbon steel waste extract containing tetraoxosulphate (vii) acid also indicated that the dye has very good fastness properties. Black and blue shades generally obtained from tannin-rich plant natural dyes are substantive towards cellulosic and protein fibre impacts well overall fastness properties. E.g logwood, harda, custard apple (Ashis and Adwaita, 2011). The result of the fastness properties when the two dyes are compared together shows that the dye produced from the reaction of tannic acid, carbon steel waste and tetraoxosulphate (vii) acid has very good fastness properties when compared with that of natural dye, carbon steel waste extract and formic acid.

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