

Optimization of Extraction Conditions and Dyeing Standardization of *Eucalyptus Bark* as Natural Dye

Saminu Murtala Yakasai and Abdulkadir Ahmed Isah
Department of Chemistry, Federal University Dutse, Jigawa State

ABSTRACT

This study forms an important basis on which a natural dye was extracted from Eucalyptus bark, obtained at federal university Dutse, Jigawa state at different extraction conditions. The dye was extracted at different temperatures with various stirring time. For the extraction process, the effect of stirring was sporadic and the dye extract obtained at boiling after soaking overnight have the maximum colour strength. The dyeing condition was optimized by dyeing four more samples of cotton fabrics at various temperature viz 40 °C, 60 °C, 80 °C, and 100 °C respectively. Maximum colour strength was obtained at 100 °C which is the optimized dyeing temperature. The optimum sodium sulphate concentration required for best degree of exhaustion was obtained, by dyeing six more samples at the optimized temperature (100 °C) for 60 minutes each with 0 g/L, 20 g/L, 40 g/L, 60 g/L, 80 g/L, 100 g/L sodium sulphate respectively, it was found that the dyeing at 100 g/L yields the maximum colour strength. To obtain the optimum dyeing time, 6 more samples were dyed at the optimized dyeing temperature and electrolyte concentration for 20 minutes, 40 minutes, 60 minutes, 80 minutes, 100 minutes, and 120 minutes, respectively. The dye extract from the optimized conditions was used to dye cotton fabric, by direct dyeing method, in an attempt to attain desirable fastness properties by comparing the fastness properties of dyeing with different dyeing techniques.

Keywords: *Eucalyptus bark, Natural dye, Colour strength, Cotton fiber and Dyeing.*

INTRODUCTION

Natural dyes are colourants gotten from vegetable or animal without undergoing any chemical processing. The international demand in textile materials dyed with natural dyes has increased, this is as result of increasing awareness of the environmental and health risks accompanying the synthesis, processing, and application of synthetic dyes. The textile colouration, cosmetic, pharmaceutical as well as the food industry increased demand for natural colourants during the last decade as displayed the importance of natural dyes in our modern world (Adeel *et al.*, 2017).

Environmental Pollution is one of the major setbacks in synthetic dyes processing industries. In order to process a textile material a large volume of water might be use and as a result the heavy chemicals waste generated by the water during the textile processing will pollute the environment. Therefore, to reduce the environmental pollution, the use of dyes and chemicals that are environmentally friendly, non-toxic and ecofriendly needs to be encouraged (Bectold *et al.*, 2006).

Since the advent of widely available and cheaper synthetic dyes in 1856 having moderate to excellent colour fastness properties, the use of natural dyes having poor to moderate wash and

light fastness has declined to a great extent. However, recently there has been revival of the growing interest on the application of natural dyes on natural fibers due to worldwide environmental consciousness (Bectold *et al.*, 2003).

Recently, a number of commercial dyers and small textile export houses have started looking at the possibilities of using natural dyes for regular basis dyeing and printing of textiles to overcome environmental pollution caused by the synthetic dyes (Bectold and Mussak, 2009).

Extraction of dyes have been engaged by several domestic and foreign institutes from natural origin, even though their various setbacks encountered such as, lack of desirable fastness properties and poor reproducibility of shades (Vankar *et al.*, 2006). This research is about the extraction of dye from Eucalyptus and its application on cotton fabric in an attempt to investigate the optimum conditions for its application to achieve a desirable fastness property.

Eucalyptus grows on valleys, swampland, and mountains. Its trees are characterized by their leathery, whitish leaves with a peculiar aroma. Eucalyptus bark is one of the most important

sources of yellowish brown colourants. The important compounds found in the eucalyptus bark are Eriodictyol, Naringenin, Quercetin, Rhamnazin, Rhamnetin and Toxifolin, apart from tannins of which some are colourants. The

major colouring component of Eucalyptus bark is quercetin, which is also an antioxidant. The structures of two important colouring components of Eucalyptus is show bellow in Figure 1. (Vankar, 2007).

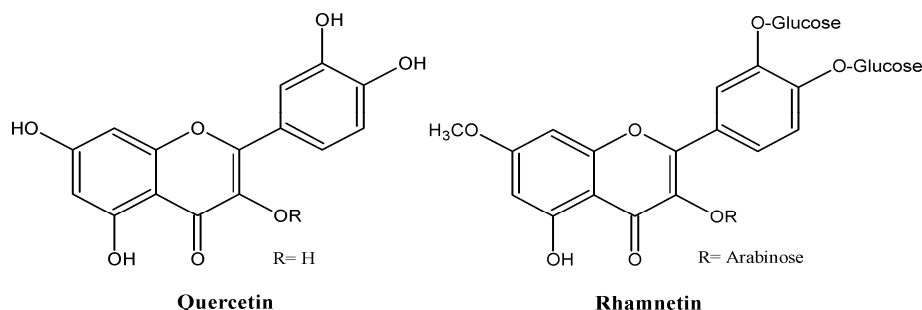


Figure 1: Chemical structures of Quercetin and Rhamnetin.

MATERIALS AND METHODS

Materials and Reagents

Eucalyptus bark, Beakers, Bulb, Conical flask, Cotton material, Filter paper, Mesh, Magnetic stirrer, Crockmeter, for testing rubbing fastness, Spectrophotometer for colour measurement, Sodium sulphate, to promote dye exhaustion.

Methods

The procedures reported was adopted by Ali *et al.*, (2007) with some modifications.

Optimisation of extraction conditions

Dead Eucalyptus bark was collected from the Federal University Dutse, Jigawa state without harming the trees. It was washed thoroughly and dried, then crushed into powder form and sieved to reduce the particle size. In order to find out the optimum extraction conditions, a total number of 25 experiments were carried out at different temperatures with various stirring times as given bellow:

- A set of 5 samples of crushed powder of Eucalyptus bark weighing 20 g, dissolved in 200 mL distilled water in a beaker and subjected to stirring at room temperature for 20, 40, 60, 80 and 100 min, respectively.
- A similar set of 5 samples subjected to stirring at 60°C for 20, 40, 60, 80 and 100 min, respectively.
- Another set of 5 samples subjected to stirring at 100°C for 20, 40, 60, 80 and 100 min, respectively.
- A set of 5 samples of crushed powder of Eucalyptus bark weighing 20 g, soaked for overnight in 200 mL distilled water in a beaker and then subjected to stirring at

room temperature for 20, 40, 60, 80 and 100 min, respectively.

- Another set of 5 samples soaked for overnight in 200 mL distilled water in a beaker and then subjected to stirring at boil for 20, 40, 60, 80 and 100 min, respectively.

25 samples of cotton fabric weighing 4 g each were used to dye in separate baths containing dye extracts obtained from the above stated experiments. Dyeing was carried out at 60 °C, with 15:1 liquor-to-goods ratio for 60 minutes, using 3 g of sodium sulphate in each dye bath to improve exhaustion. The dye extract that gave the maximum exhaustion was then selected for further experimentation in order to find out the optimum dyeing conditions.

Optimization of dyeing conditions

Four samples of cotton fabric, weighing 2 g each, were dyed in 4 separate baths for 60 minutes taking 30 mL of the dye extract (with the maximum exhaustion) in each bath at 40 °C, 60 °C, 80 °C and 100 °C, respectively, to determine the optimum dyeing temperature. All of these dyeings were carried out in the presence of 1.5 g sodium sulphate to promote exhaustion.

The optimum sodium sulphate concentration required for best degree of exhaustion was obtained, 6 more dyeings were carried out at the optimized temperature (100°C) for 60 minutes each with 0 g/L, 20 g/L, 40 g/L, 60 g/L, 80 g/L, 100 g/L sodium sulphate, respectively.

To obtain the optimum dyeing time, 6 more samples were dyed at the optimized dyeing temperature and electrolyte concentration for 20

minutes, 40 minutes, 60 minutes, 80 minutes, 100 minutes, and 120 minutes respectively.

FASTNESS TESTING

Fastness to Light

The samples of the coloured textile obtained under the optimized conditions was stitched to a piece of undyed cloth of the same fiber, to determine the light fastness. The test was carried out by exposing the specimen to a light bulb for about 72 hours. The test required eight standard blue-dyed wool cloths, numbered 1-8. Number 1 represent the very low light fastness and number 8 represent very high light fastness. At the end of the test, the change in colour of the sample was compared with changes which have occurred in the standards. The fastness rating of the specimen is the number of the standard which viewed a similar change in colour.

Fastness to Washing

Assessment of washing fastness was made by series of five washing test varying in severity from number 1 to number 5. The degree of staining was assessed by matching the appropriate undyed piece of fabric to the specimen, it was assessed by the used of grey scale.

Fastness to Rubbing

A machine known as crockmeter is used to test the fastness to rubbing or crocking. A relatively simple test is to rub the dyed fabric with a piece of undyed fabric wrapped round the finger, the first with white dry and then with wet white fabric, which will stain the white fabric and the fastness to rubbing property is determined.

RESULTS AND DISCUSSION

Effect of Extraction Conditions

The relative colour strength of the dye extracts obtained under different extraction conditions is given in Figure 2, From the Figure, colour strength of dye extracts obtained at room temperature was minimum, slightly getting better at 60 °C and at boiling temperature. For extractions process, the effect of stirring was sporadic. However, the colour strength of the dye extract gotten at boiling after soaking overnight was maximum, the colour strength of dye extracts increased by increasing the stirring time up to 80 minutes. Stirring for more than 80 minutes decreased colour strength. This happened because after such a long stirring time, some impurities were also extracted along with the colouring components, thus decreasing the overall colour strength of the dye extract. Ali *et al.*, (2007)

carried out similar research and it was found that, stirring time and temperature is proportional to the colour strength obtained during extraction. The maximum colour strength was obtained at boiling after soaking overnight with stirring duration of 80 minutes.

Effect of Dyeing Temperature on Colour Strength

The effect of dyeing temperature on colour strength is demonstrated in Figure 3. Maximum colour strength was obtained at 100 °C, and also a similar work was done by Ali *et al.*, 2007 and similar result was obtained. High temperature will lead to high value of entropy, increase the kinetic energy of dye molecule as well as increasing the pore size of the fiber, which permits dye molecules to rapidly penetrate into the fabric and distribute themselves very easily all over the available dye sites. The increase in kinetic energy of the molecule lead to the molecule moving faster from the solution in to the fiber, and this bring about reduction in the time of dyeing (Ali *et al.*, 2007).

Effect of Salt Concentration on Colour Strength

The effect of salt concentration on colour strength is shown in Figure 4. The colour strength was increased steadily by increasing the concentration of sodium sulphate from 0 g/L to 100 g/L. It was investigated by Ali *et al.*, 2007 and similar result of close values where obtained, further increase in salt concentration had sporadic effect because of increase in dye aggregation at high salt concentrations.

Effect of Dyeing Time on Colour Strength

Effect of dyeing time on colour strength is shown in Figure 5. The longer the dyeing time, the higher is the colour strength until dye exhaustion attains equilibrium and there is no significant increase in the colour strength after further increase in dyeing time.

Fastness Properties

Table 1 shows the fastness properties of the dyed cotton fabric under optimized conditions. The dyed cotton has fairly good fastness properties when compared with the grey scale standards. Washing and light fastness properties are better than many of the commonly used dyes. Ali *et al.*, 2007 carried out similar research work and found out that, the fastness properties of the dyed material was fairly good. However, which is not be acceptable for high quality material requiring very good to excellent fastness properties. Dry rubbing fastness is very good, while wet rubbing fastness is poor.

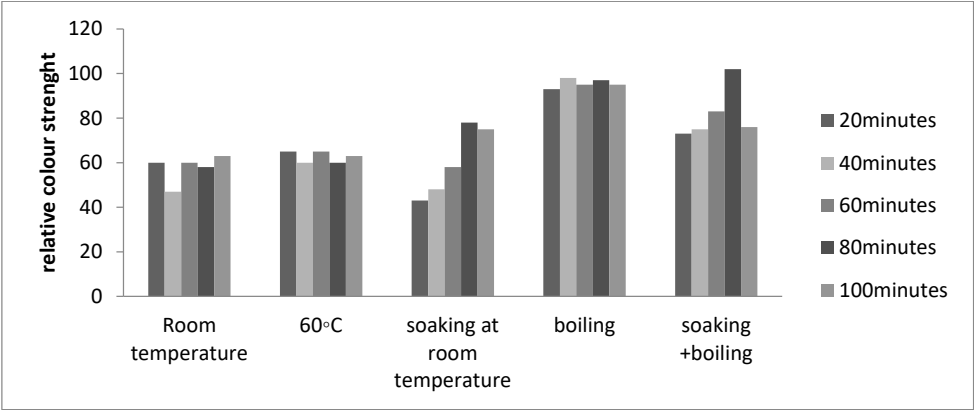


Figure 2: Effect of Extraction Conditions on Colour Strength

EFFECT OF DYEING CONDITIONS



Fabric Dyed at Optimum Salt Concentration



Fabric dyed at optimum dyeing temperature



Bleached treated fabric



Fabric Dyed at Optimum Dyeing Time

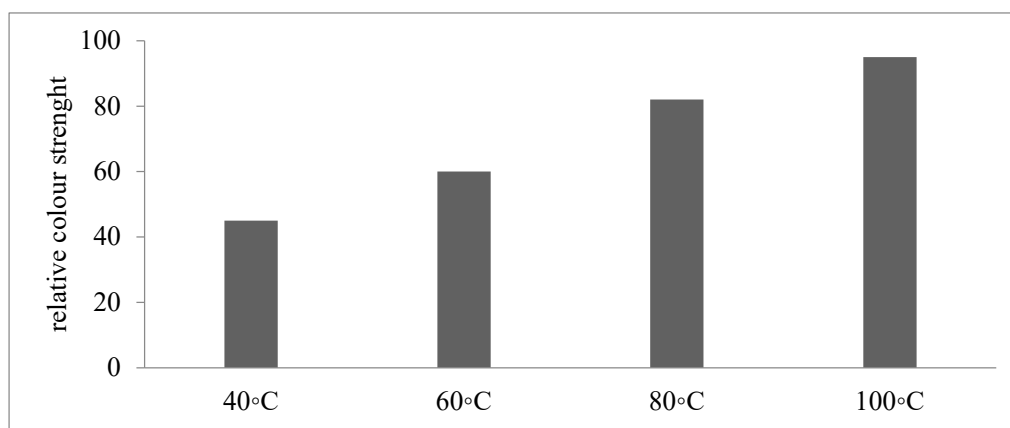


Figure 3: Effect of Dyeing Temperature on Colour Strength

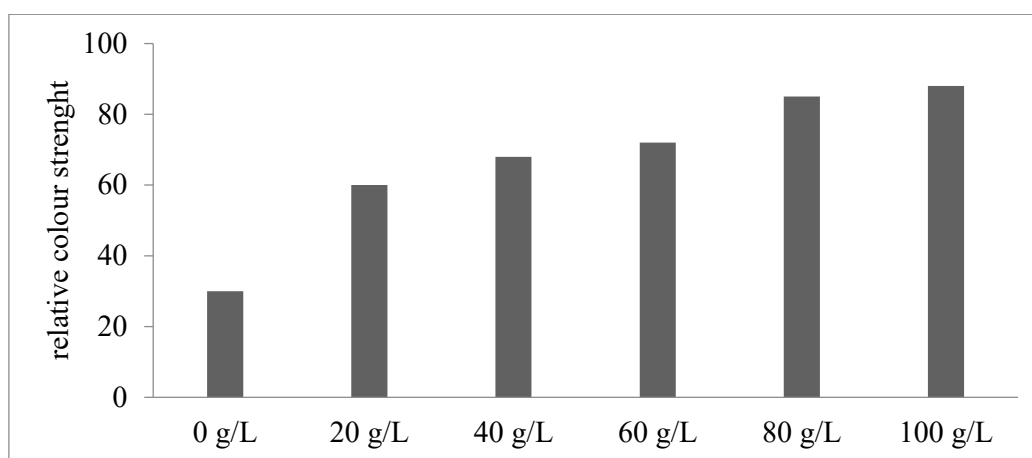


Figure 4: Effect of Salt Concentration on Colour Strength

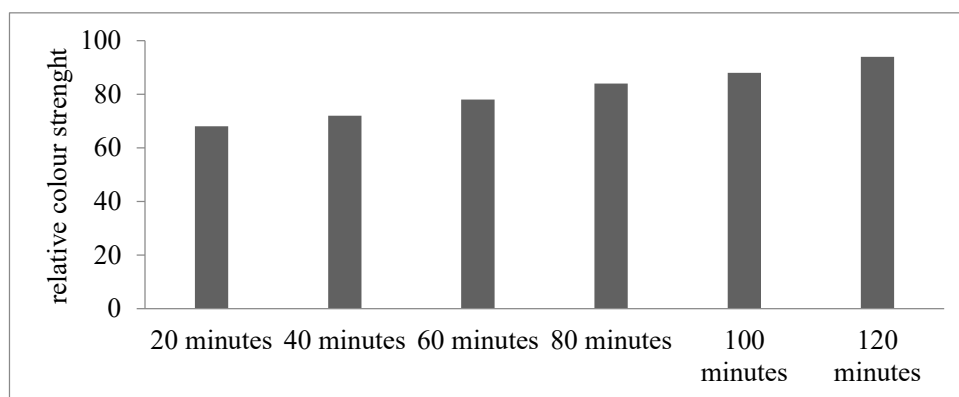


Figure 5: Effect of Dyeing Time on Colour Strength

Table 1: Fastness properties of dyed fabric

Light fastness	washing fastness		Dry rubbing		Wet rubbing	
	Alteration	Staining	Alteration	Staining	Alteration	Staining
4	3	4	4	5	3	4

CONCLUSION

Extraction and dyeing conditions of a natural dye from eucalyptus was optimized, It has been observed that temperature, concentration of salt, and dyeing time plays a vital role in obtaining dye extract with maximum colour strength. The dye obtained displays fairly good saturation on cotton with medium to good fastness properties. The colour strength increases with increased in temperature, high salt concentration and increased dyeing time. The colour strength of the dye extract obtained at boiling after soaking overnight was found to be the best extraction condition, because a maximum yield was obtained. Moreover maximum colour strength was obtained during dyeing at 100 °C. This means that increase in dyeing temperature will result in increase in colour strength. Also, the colour strength was increased by increasing the salt concentration, which result to an increased in dye aggregation. The duration of dyeing has an effect on the colour strength. The longer the dyeing time, the higher the colour strength until dye exhaustion attains equilibrium and no significant increase at that time. The dye cotton fabric exhibit fair to good fastness properties.

REFERENCES

- Adeel, S., Gulzar, T., Azeem, M., Fazal ur, R., Saeed, M., Hanif, I., & Iqbal, N. (2017). *Appraisal of marigold flower based lutein as natural colourant for textile dyeing under the influence of gamma radiations. Radiation Physics and Chemistry, 130*, 35-39. doi:10.1016/j.radphyschem.2016.07.010
- Ali, S.; Nisar, N. & Hussain, T. (2007). *Dyeing properties of natural dyes extracted from eucalyptus. The Journal of The Textile Institute*, Vol. 98, No. 6, pp. 559-562, ISSN1754-2340
- Bechtold, T. & Mussak, R. (2009). *Handbook of natural colorants*, John Wiley & Sons, ISBN978-0-470-51199-2, West Sussex, England
- Bechtold, T., Mussak, R., Mahmud-Ali, A., Ganglberger, E., & Geissler, S. (2006). Extraction of natural dyes for textile dyeing from coloured plant wastes released from the food and beverage industry. *Journal of the Science of Food and Agriculture*, 86(2), 233-242. doi:10.1002/jsfa.2360
- Bechtold, T., Turcanu, A., Ganglberger, E., & Geissler, S. (2003). *Natural dyes in modern textile dyehouses - how to combine experiences of two centuries to meet the demands of the future? Journal of Cleaner Production*, 11(5), 499-509. doi:10.1016/s0959-6526(02)00077-
- Conde, E.; Cadahia, E. & Garcia-Vallejo, M. C. (1997). *Low molecular weight polyphenols in leaves of Eucalyptus camaldulensis. E. globules and E. rudis. Phytochemical Analysis*, Vol. 8, No. 4, pp. 186-193, ISSN 0958-0344
- Gulrajani, M. L., 1992. *Natural Dyes and Their Applications to Textiles*, Indian Institute of Technology, New Delhi, India.
- Hunger, K. (2003). *Industrial dyes: Chemistry, Properties, Applications*, WILEY-VCH Verlag GmbH & Co. KGaA, ISBN 35-27304-26-6, Darmstadt, Germany
- Hwang, E. K.; Lee, Y. H. & Kim, H. D. (2008). Dyeing, fastness, and deodorizing properties of cotton, silk, and wool fabrics dyed with gardenia, coffee sludge, Cassia tora. L., and pomegranate extracts. *Fibers and Polymers*, Vol. 9, No. 3, pp. 334-340, ISSN 1857-0052
- Vankar, P. S. (2007). *Handbook on natural dyes for industrial applications*, National Institute of Industrial Research, ISBN 81-89579-01-0, New Delhi, India
- Vankar, P. S.; Tiwari, V. & Srivastava, J. (2006). Extracts of steam bark of Eucalyptus Globules as food dye with high antioxidant properties. *Electronic Journal of Environmental, Agricultural and Food Chemistry*, Vol. 5, No. 6, pp. 1664-1669, ISSN 1579-4377.