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Natural Dye from Eucalyptus Leaves and Application for Wool Fabric Dyeing by Using Padding Techniques

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1. Introduction

Natural dyes are known for their use in colouring of food substrate, leather, wood as well as natural fibers like wool, silk, cotton and flax as major areas of application since ancient times. Natural dyes have a wide range of shades that can be obtained from various parts of plants, including roots, bark, leaves, flowers and fruits (Allen, 1971). 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 (Samanta & Agarwal, 2009). Although this ancient art of dyeing with natural dyeing with natural dyes withstood the ravages of time, a rapid decline in natural dyeing continued due to the wide available of synthetic dyes at an economical price. However, even after a century, the use of natural dyes never erodes completely and they are still being used. Thus, natural dyeing of different textiles and leathers has been continued mainly in the decentralized sector for specialty products along with the use of synthetic dyes in the large scale sector for general textiles owing to the specific advantages and limitations of both natural dyes and synthetic dyes. The use of non-toxic and ecofriendly natural dyes on textiles has become a matter of significant importance because of the increased environmental awareness in order to avoid some hazardous synthetic dyes. However, worldwide the use of natural dyes for the colouration of textiles has mainly been confined to craftsman, small scale dyers and printers as well as small scale exporters and producers dealing with high valued ecofriendly textile production and sales (Samanta & Agarwal, 2009; Bechtold & Mussak, 2009; Vankar, 2007). 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 (Glover & Pierce, 1993). Natural dyes produce very uncommon, soothing and soft shades as compared to synthetic dyes. On the other hand, synthetic dyes are widely available at an economical price and produce a wide variety of colours; these dyes however produce skin allergy, toxic wastes and other harmfulness to human body. There are a small number of companies that are known to produce natural dyes commercially. For example, de la Robbia, which began in 1992 in Milan, produces water extracts of natural dyes such as weld, chlorophyll, logwood, and cochineal under the Eco-Text certifying system, and supplies the textile industry. In USA, Allegro Natural Dyes produces natural dyes under the Ecolour label for textile industry (Hwang et al., 2008). Aware of the Toxic Substance Act and the Environmental Protection Agency, they claim to have developed a mordant using a non-toxic aluminium formulation and biodegradable auxiliary substance. In Germany, Livos Pflanzenchemie Forschungs and Entwicklungs GmbH marketed numerous natural products. In France, Bleu de Pastel sold an extract of woad leaves. Rubia Pigmenta Naturalia is The Netherlands company, which manufactures and sells vegetable dyes. There are several small textile companies using natural dyes. India is still a major producer of most natural dyed textiles (Vankar, 2007). Production of synthetic dyes is dependent on petrochemical source, and some of synthetic dyes contain toxic or carcinogenic amines which are not ecofriendly (Hunger, 2003). Moreover, the global consumption of textiles is estimated at around 30 million tonnes, which is expected to grow at the rate of 3% per annum. The colouration of this huge quantity of textiles needs around 700,000 tonnes of dyes which causes release of a vast amount of unused and unfixed synthetic colourants into the environment (Samanta & Agarwal, 2009). This practice cannot be stopped, because consumers always demand coloured textiles for eye-appeal, decoration and even for aesthetic purposes. Moreover, such a huge amount of required textiles materials cannot be dyed with natural dyes alone. Hence, the use of eco-safe synthetic dyes is also essential. But a certain portion of coloured textiles can always be supplemented and managed by eco-safe natural dyes (Samanta & Agarwal, 2009; Vankar, 2007). However, all natural dyes are not ecofriendly. There may be presence of heavy metals or some other form of toxicity in natural dye. So, the natural dyes also need to be tested for toxicity before their use (Vankar, 2007).

2. Natural organic dyes from eucalyptus

Eucalyptus is a members of evergreen hardwood genus, endemic to Australian. There are approximately nine hundred species and sub-species. Eucalyptus has also been successfully grown in many parts of the world, including southern Europe, Asia and the west coast of the United States (Flint, 2007). Eucalyptus is one of the most important sources of natural dye that gives yellowish-brown colourants. The colouring substance of eucalyptus has ample natural tannins and polyphenols varying from 10% to 12% (Ali et al., 2007). The major colouring component of eucalyptus bark is quercetin, which is also an antioxidant. It has been used as a food dye with high antioxidant properties (Vankar et al., 2006). Eucalyptus leaves contain up to 11% of the major components of tannin (gallic acid [3,4,5 - trihydroxybenzoic acid], with ellagic acid [2,3,7,8-tetrahydroxy (1) benzopyrano (5,4,3-cde) (1) benzopyran-5,10-dione]) and flavonoids (quercetin [3,3',4',5,7-pentahydroxylflavone] and rutin [3,3',4',5,7-pentahydroxylflavone-3-rhamnoglucoside]) as the minor components (Chapuis-Lardy et al., 2002; Conde et al., 1997). The structures of the colouring components found in eucalyptus leaves are given in Fig. 1. Tannins and flavonoids are considered very useful substances during the dyeing process because of their ability to fix dyes within

fabrics. Silk dyed with an aqueous extract of eucalyptus leaves and bark possessing a mordant compound displays a yellowish-brown colour. An exception was when the fabric was dyed with ferrous mordant, resulting in a shade of dark brownish-grey. Colour fastness to water, washing, and perspiration was at good to very good levels, whereas colour fastness to light and rubbing exhibited fair to good levels (Mongkhorrattanasit et al., 2007; Mongkhorrattanasit et al., 2010; Mongkhorrattanasit et al., 2011).

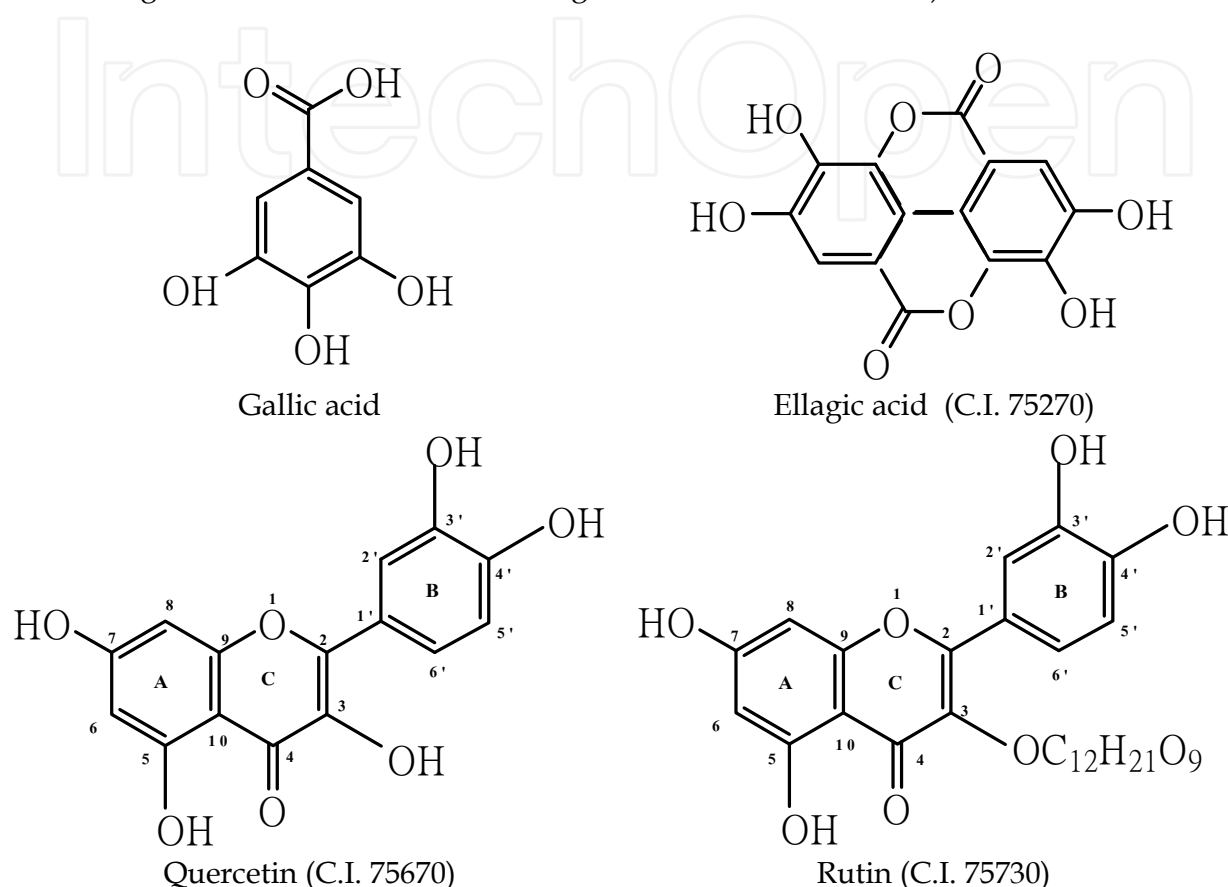


Fig. 1. Colour composition of eucalyptus leaf extract dye

3. Using of natural dyes

Currently, application of natural dye incorporates new technology not only to exploit traditional techniques but also to improve the rate, cost and consistency production. It therefore, requires some special measurement to ensure evenness in dyeing. The processes of natural dyes for textile dyeing are as follows:

3.1 Extraction

Efficient extraction of the dyes from plant material is very important for standardization and optimization of vegetable dyes, utilizing a) soxhlet b) supercritical fluid extraction c) subcritical water extraction and d) sonicator method.

3.2 Dyeing

Normally, one technique used for dyeing with natural dye; exhaustion dyeing (conventional dyeing, sonicator dyeing and microwave dyeing). Exhaustion dyeing is using lot of water as

shown in “Liquor Ratio (ratio between water and goods)”. Producers immerse the goods in dye for extended periods for complete penetrate. This produces excessive waste water compared to a continuous process. The techniques used for dyeing of natural dyes, such as

1. Conventional dyeing : conventional dyeing is carried out by boiling the fabric in dye bath for 4-hours and often the dye uptake is still not completed. Enormous amount of heat is consumed in terms of heating the dye bath (Vankar, 2007).
2. Sonicator dyeing: utilization of ultrasound energy to aid wet processing of fabrics. The process of increasing dye transfer from the dye-bath to fabric using ultrasound energy is a function of the acoustic impedance characteristics of the fabrics (Vankar, 2007 ; Vankar et al., 2009 ; Tiwari & Vankar, 2001).
3. Microwave dyeing : microwave dyeing take into account only the dielectric and the thermal properties. The dielectric property refers to the intrinsic electrical properties which affect dyeing by dyeing by dipolar rotation of the dye and the influence of microwave field upon dipoles. The aqueous solution of dye has two components, which are polar. In the high frequency microwave field, oscillating at 2450 MHz; it influences the vibrational energy in the water molecule and the dye molecules (Tiwari & Vankar, 2001).

3.3 Mordanting

In the actual dyeing process, there are four ways of using mordant (Bechtold & Mussak, 2009; Moeyes, 1993) as follows:

- a. Mordanting before dyeing, or pre-mordanting;
- b. Mordanting and dyeing at the same time, called stuffing or simultaneous;
- c. Mordanting after dyeing, or after-mordanting or post-mordanting;
- d. A combination of pre-mordanting and after-mordanting.

4. Theoretical presuppositions of natural dyes to dyeing

Achieving a good, or at least a relatively good, water solubility using natural dyes is rather exceptional. No chemical group is capable of electrolytic dissociation or ionization in a molecule; an interesting and important exception is the anthocyanins, for example, pelargonidine, cyanidine, and betanidine are slightly cationic dyes and, therefore, also have relatively good solubility in water (Mongkhorrattanasit et al., 2009). The “conditional solubility” of indigoid natural dyes, which in their original form are entirely insoluble, presents a quite special principle. In fact, indigo has been imitated to a great extent; synthetic indigo and their derivatives were produced on an industrial scale at the end of the nineteenth century as a forerunner of the latter large group of vat dyestuffs. The alkali reductive conversion of this fully insoluble compound in a proper soluble sodium salt of leucocompound with affinity to fibers and their oxidation after dyeing with the primary insoluble vat dye, which is finely dispersed in the fiber, is well known. What do the majority of natural dyes have in common? The chemical constitution (and corresponding physical properties) of indigo and other anthocyanin dyes has remarkable similarity with the modern synthetic disperse dyes: the solubility of more or less elongated molecules of chromogen is due to the presence of several polar groups (mainly -OH) on aromatic rings. No groups are capable of electrolytic ionization (with the exception of the anthocyanin and betanin). From this follows that they only have low solubility in water. Empirically, it is known that it is impossible to strengthen dyeing of cotton with natural dyes, but it can be done by adding

neutral electrolytes (sodium chloride or sulfate) as substantive dyes. And bath acidifying, while having a significant effect on the so-called acid dyes (coloured sodium salts of sulfonic acids), has a negligible effect on the natural dyes. The structure of the flavonoid-colouring components of eucalyptus leaves and tannin (Fig. 1) is compared with the typical azo and anthraquinone disperse dye (Fig. 2).

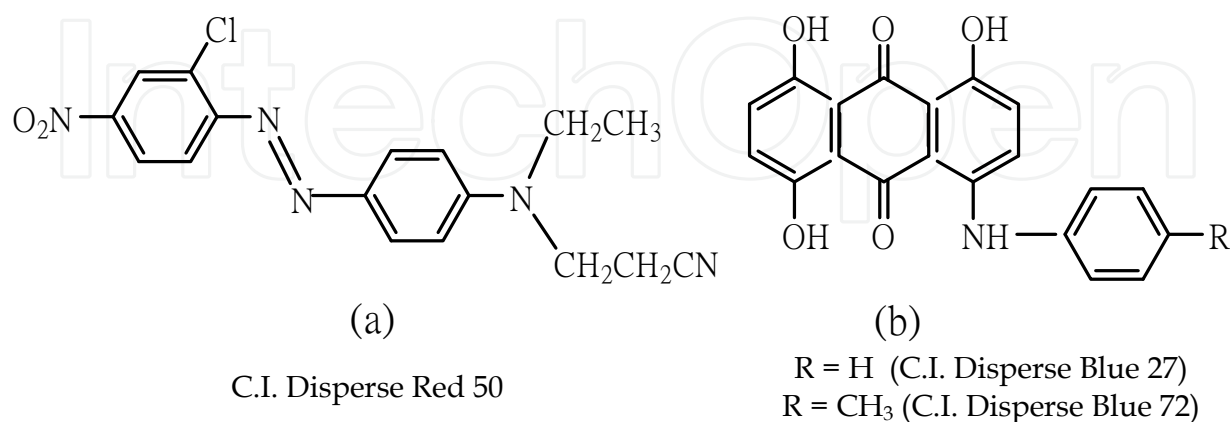


Fig. 2. Chemical constitution of typical disperse dyes. (a) Azo dye and (b) anthraquinone dyes

Assume that most natural dyes are, on the basis of modern dyeing science, the disperse dyes. But what are the dyes for wool, silk, cotton, and flax? Consider that each fiber type in dyeing has already been studied, and it has become apparent that the disperse dyes are not good dyes for the aforementioned fibers. On the contrary, the synthetic disperse dyestuffs were developed for dyeing acetyl cellulose and synthetic fibers (i.e., hydrophobic fibers), and they have a low affinity for wool, silk, cotton, and other such fibers that are mainly hydrophilic. Though low, the indispensable affinity of disperse dyes makes them very undesirable for the staining of wool or cotton component by the dyeing of fiber mixtures, namely with polyester fiber (which is dyeable only in disperse dyes). This imperfect colouration-staining must be rather difficult to remove from wool or cotton component after dyeing because of its poor wet fastness and mostly unpleasant shade, which can be different from the shade of the same dye on polyester. However, the above-mentioned majority of natural dyes are providing only inexpressive wet fastness on wool and cotton fibers, and the mordanting by salts of suitable metals is also needed to improve wet fastness (not only to deepen but also to intensify the colour). A lower affinity results in the low dye exhaustion after the dye bath on the fiber. This can also be observed in the dyeing of natural fibers with natural dyes, such as the indigoid and anthocyanin dyes.

5. Ecological and economical aspects of dyeing with natural dyes

If we carry out the dyeing process with natural dyes in a slightly large manufacturing unit or a factory rather than in a household unit, we can surpass the limits of historic methods of dyeing and material pretreatments, which are lengthy and uneconomical procedures. The old methods (likely transmitted without facing critical evaluation), consist of various actions that do not address modern requirements, and do not take into account the new possibilities offered by the modern textile chemistry. The number and duration of baths seem to be too high (at least for European standards and customs) and are non-productive. For example,

the required 3–5 hours wetting of material with water before dyeing could be greatly reduced by wetting in a bath by specially made wetting agent, and this or another agent could also be added into the dyeing bath. The ineffective use of natural dyes was already discussed above. The majority of dyes ceases as effluents in sewer. The mordanting salts do not have affinity to the fibers and therefore only a small part of them is bounded with fibers, and after dyeing and final rinsing all the remnants are carried off by water. What about the idea of storing the mordanting baths for future use? While logical, the number and volume of stock reservoirs (and place in dye house) make it an unpractical possibility. Naturally, serious conception-questions follow from this. Should “natural dyeing” remain as something principally untouchable whose traditional originality must be safe-guarded at any costs, or are we going to consider this natural raw-material source as an ecologically favorable supplement to synthetic colourants? or, can we synthesize the methodologies of “natural dyeing” with the research and application processes of modern dyeing technology? Nevertheless, both natural dyeing and modern dyeing technology can coexist. In any case, we are trying to explore the second of the following:

- the consequent minimization of concentration of natural dyes and mordants,
- the shortening of operating times, i.e., to save energy and productivity, and
- the maximal efficient use of dye and mordanting baths.

All these can be assured by the padding (pad) technologies, in which the liquor ratio (weight of textiles: bath) is about one order lower ($\leq 1:1$) than the common exhaustion (bath or batch) dyeing methods. The padding technologies are particularly advantageous to dyeing with the low-affinity products, because the dye affinity to fiber by padding is unnecessary (in phase of the dye deposition on the fabric). The dye bath is cloth “padded”: mechanically applied by the rapid passage through the small padding trough, the intensive squeezing between expression rollers follows immediately. The process of padding is continuous and very rapid. It depends on the arrangement of the following dye fixation if the total procedure is continuous or semi continuous. The dye bath by padding is about one order higher than by the common dyeing from the “long bath” (the so-called exhaustion methods), in which the dyestuff exhausts on the fiber in consequence to its affinity to the fiber. The higher padding bath concentration results in more rapid dye diffusion in fiber during the next fixation operation. Much smaller bath volume (related to the fiber unit) causes the higher dye exploitation. In the case of natural dyes, the dye fixation is based on the reaction (see also Agarwal and Patel) (Agarwal & Patel, 2001) with the salts of complex-forming metals-mordants in the same or next bath-or the textile can be pre-metalized with mordant (this pre-mordanting is carried out from the long bath-the large non-effectiveness is mentioned above. Therefore, we also experimented with pad-dry and pad-batch principle at this operation). In semi continuous dyeing technology, several methods of dye fixation are known. The following two principles are important for our purpose:

- a. fixation by drying, the so-called pad-dry method, the process is rapid but requires a reliably functional drying device (an excellently even -drying effect breadth-ways and cross-ways in the fabric is necessary, otherwise it may result in colour depreciation and unevenness),
- b. fixation by batching of the padded goods at room or slightly increased temperature, now known as the pad-batch method. The padded and rolled goods are wrapped up in an airtight plastic sheet so that no selvedge drying occurs during storage, which lasts 8–24 hours.

After both dye fixation methods water rinsing follows repeatedly.

6. Experimental

The research focused on the properties of pad-dyeing techniques, we investigated the dyeing and ultraviolet (UV) protection properties of wool fabric using an aqueous extract of eucalyptus leaves as the natural dye. Different factors affecting dyeing ability were also thoroughly investigated.

The following laboratory-grade mordants were used: aluminium potassium sulfate dodecahydrate ($\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$), ferrous (II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), copper (II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) and stannous chloride pentahydrate ($\text{SnCl}_2 \cdot 5\text{H}_2\text{O}$). The anionic wetting agent, Altaran S8 (sodium alkylsulfate), and soaping agent, Syntapon ABA, were supplied by Chemotex Decin, Czech Republic.

The mordanting and dyeing processes were carried out in a two-bowl padding mangle machine (Mathis, Typ-Nr. HVF.69805). A drying machine (Mathis Labdryer, Typ-Nr. LTE-2992) was used for the drying of the dyed fabrics. A GBC UV/VIS 916 (Australia) spectrophotometer and a Datacolor 3890 were employed for the absorbance and colour strength measurements, respectively. The transmittance and ultraviolet protection factor (UPF) values were measured by a Shimadzu UV3101 PC UV-VIS-NIR scanning spectrophotometer in the 190 nm to 2100 nm range.

Fresh eucalyptus leaves (*E. Camaldulensis*) were dried in sunlight for one month and crumbled using a blender, and then were used as the raw material for dye extraction, which was achieved by the reflux technique: 70 g of crumbled eucalyptus leaves was mixed with one liter of distilled water and refluxed for one hour. The dye solution was filtered, evaporated, and dried under reduced pressure using a rotary evaporator. The crude dye extract of the eucalyptus leaves was then crumbled with a blender and used for obtaining the standard calibration curve. The dilution of the eucalyptus leaf extract gives a relatively clear solution with a linear dependence on the concentration absorbance, an absorption peak (λ_{max}) at 262 nm (Yarosh et al., 2001). The concentration of 20 g/l was calculated from a standard curve between concentrations of eucalyptus leaf dye solutions versus absorbance at the wavelength mentioned.

The pre-mordanting methods, wool fabrics were immersed in each mordant solution with anionic wetting agent and padded on a two-bowl padding mangle at 80% pick up. Next, the mordanted sample was impregnated in each eucalyptus dye concentration. After padding for 2 seconds the samples were dried at 90°C for 5 minutes for a pad-dry technique. Under the cold pad-batch dyeing technique, the padded fabric was rolled on a glass rod with a plastic sheet wrapped around the rolled fabric. Then it was kept at room temperature for 24 hours. After the dyeing step, the samples were washed in 1 g/l of a soaping agent, Syntapon ABA, at 80°C for 5 minutes, then air dried at room temperature. For the simultaneous mordanting (meta-mordanting) method (i.e. dyeing in the presence of mordants), the fabrics were immersed in a bath containing a mordant and the dye extract at room temperature and padded on a two-bowl padding mangle at 80% pick up. The processing of pad-dry, pad-batch and soaping were the same as above mention. In the post-mordanting method, the fabrics were immersed in each eucalyptus dye concentration and without mordant, followed by padded on a two-bowl padding mangle at 80% pick up. Then the padded samples were padded by mordanting. Further processing was the same as described in the pre-mordanting method.

The colour strength (K/S) and CIELAB of the dyed samples were evaluated using a spectrophotometer (Datacolor 3890). All measured sample showed the maximum absorption wavelength (λ_{max}) value at 400 nm. The K/S is a function of colour depth and is

calculated by the Kubelka-Munk equation, $K/S = (1-R)^2/2R$, where R is reflectance, K is the sorption coefficient, and S is the scattering coefficient.

6.1 Identification of crude eucalyptus extracted dye

The crude eucalyptus leaf extract dye was characterized by ultraviolet-visible spectroscopy. The crude extraction solution (50 mg/l) was prepared by dissolving in distilled water. The spectrophotometer was scanned from 190 nm to 820 nm to obtain the UV/Visible spectra.

The UV-vis spectrum of the crude eucalyptus leaf extract dye in an aqueous solution is presented in Fig. 3. The characteristic spectrum shows absorptions in the 205–210 nm and 250–270 nm regions. Absorption in the 205–210 nm region may be attributed to various chromophores, including the C=C bond of various compounds, the C=O bond of carbonyl compounds, and the benzene ring (probably from aromatic compounds) (Pretsch et al., 2000). Absorption in the 250–270 nm regions may be attributed to the electronic transitions of benzene and its derivatives, which may include various aromatic compounds such as phenolics (Pretsch et al., 2000). It can be observed from Fig. 3 that the dye can absorb radiations in the UV-C region (200–290 nm), the UV-B region (290–320) and the UV-A region (320–400) (Feng et al., 2007).

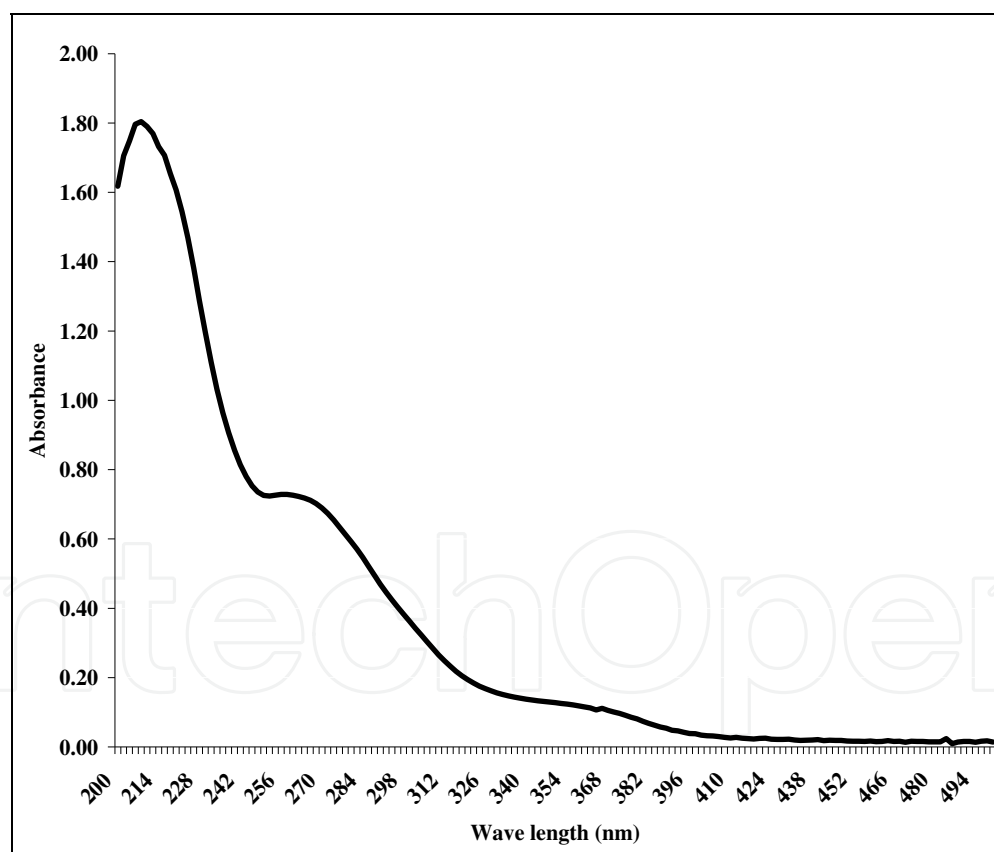


Fig. 3. UV-VIS spectrum of 50 mg/l crude eucalyptus leaf extract dye in distilled water.

6.2 Dyeing property of wool fabric dyed with eucalyptus leaf extract by using padding techniques by varying quantity of dye concentrations

The effect of mordanting methods and padding techniques on dyeing of wool fabric with different mordants are shown in Table 1 to Table 3. All measured sample showed the greatest











λ_{max} value at 400 nm. Table 1 to Table 3 show CIELAB L^* , a^* , b^* values for the wool fabric dyed with different mordants by three mordanting methods (pre-mordanting, simultaneous mordanting and post-mordanting) and using two padding techniques, namely the pad-batch and pad-dry techniques. L^* , a^* , b^* refer to the three axes of the CIELAB system. The L^* value indicates perceived lightness in CIELAB colour space. The L^* scale run from 0 (black) to 100 (white); the higher the L^* reading the lighter colour. The a^* value indicates red ($+a^*$) and green ($-a^*$) while the b^* value indicates yellow ($+b^*$) and blue ($-b^*$) (Sarkar & Seal, 2003 ; Giles,1974; Duff & Sinclair,1989). It can be observed that the K/S values increase with an increase of dye concentration. Little difference between the two padding techniques utilized for the wool fabric dyes by three mordanting methods, except wool fabrics mordanted with copper sulfate whose gave a high K/S values on the pad-batch technique than pad-dry technique. In all cases ferrous sulfate mordant yielded the best dyeing results, and the next good result was obtained in the order of copper sulfate, stannous chloride and alum. As observed from the K/S values, in the case of wool fabrics dyed with alum by using post-mordanting method gave lower colour strength than without mordant.

| Type of mordant | Dye Conc. (g/l) | Pad-batch on wool fabric | | | | | Pad-dry on wool fabric | | | | |
|---|-----------------|--------------------------|-------|-------|-------|--------------------------|------------------------|-------|-------|-------|--------------------------|
| | | K/S | L^* | a^* | b^* | ¹ Dyed sample | K/S | L^* | a^* | b^* | ¹ Dyed sample |
| Without mordant | 5 | 1.50 | 76.1 | 3.5 | 14.2 | | 1.13 | 77.7 | 3.5 | 12.0 | |
| | 10 | 1.60 | 76.0 | 3.5 | 15.0 | | 1.54 | 76.3 | 3.6 | 14.1 | |
| | 20 | 1.86 | 75.4 | 3.5 | 15.8 | | 1.86 | 75.9 | 3.4 | 15.8 | |
| AlK(SO ₄) ₂ (Al) | 5 | 1.44 | 79.0 | 0.4 | 19.1 | | 1.36 | 79.6 | 0.1 | 20.3 | |
| | 10 | 1.70 | 77.6 | 0.1 | 19.3 | | 1.45 | 78.8 | 0.2 | 19.7 | |
| | 20 | 1.75 | 75.2 | 0.8 | 20.4 | | 1.70 | 76.5 | 0.8 | 19.0 | |
| CuSO ₄ (Cu) | 5 | 2.58 | 67.2 | 2.2 | 20.6 | | 2.02 | 70.8 | 2.8 | 20.0 | |
| | 10 | 3.36 | 63.7 | 2.9 | 21.3 | | 2.20 | 69.3 | 3.1 | 19.4 | |
| | 20 | 3.42 | 63.0 | 3.3 | 21.8 | | 2.70 | 67.1 | 3.4 | 20.1 | |
| FeSO ₄ (Fe) | 5 | 2.16 | 50.9 | 1.5 | 0.1 | | 2.66 | 46.8 | 2.3 | 0.4 | |
| | 10 | 2.94 | 45.2 | 1.8 | -0.6 | | 3.52 | 43.1 | 3.1 | 0.6 | |
| | 20 | 4.22 | 40.0 | 2.2 | -0.6 | | 4.34 | 38.6 | 3.4 | 0.7 | |
| SnCl ₂ (Sn) | 5 | 1.86 | 84.5 | -0.4 | 26.2 | | 2.14 | 83.0 | -0.3 | 26.9 | |
| | 10 | 2.28 | 83.7 | -0.3 | 28.4 | | 2.25 | 83.0 | -0.4 | 26.9 | |
| | 20 | 2.98 | 81.5 | 0.5 | 30.8 | | 2.37 | 81.6 | 0.1 | 26.7 | |

Note: ¹ 20g/l dye concentration











Table 1. Colour value of wool fabric dyed with eucalyptus leaf extract by pre-mordanting and padding techniques, with using 10 g/l of metal mordants at different concentration of the dye

Alum and ferrous sulfate were the best mordant during simultaneous mordanting method of dyeing. However, copper sulfate showed the best mordant during simultaneous mordanting and pre-mordanting method of dyeing. For the K/S value on dyed wool fabrics were only little different using stannous chloride as mordant during three mordanting methods. Wool dyed without mordant showed yellowish-brown shade. The samples mordanted with copper sulfate, stannous chloride, and alum produced medium to dark grayish-brown, bright yellow and pale yellow shades, respectively. With ferrous sulfate, the colour was darker and duller. This may be associated with a change of ferrous sulfate into a ferric form by reacting with oxygen in the air. Ferrous and ferric forms coexisted on the fibers and their spectra overlapped, resulting in a shift of λ_{max} and consequent colour change to a darker shade (Hwang, 2008). Additionally, the tannins combined with ferrous salts to form complexes, which also result in a darker shade of fabric (Vankar, 2007). From the results, it can be postulated that wool fabric can be successfully dyed with eucalyptus leaf extract. This may be attributed to the fact that eucalyptus leaves are rich tannin (Conde et al., 1997), which are phenolic compounds that can form hydrogen bonds with carboxyl groups in the protein fibers (Agarwal & Patel, 2001).

| Type of mordant | Dye Conc. (g/l) | Pad-batch on wool fabric | | | | ¹ Dyed sample | Pad-dry on wool fabric | | | | ¹ Dyed sample |
|---|-----------------|--------------------------|-------|-------|-------|---|------------------------|-------|-------|-------|---|
| | | K/S | L^* | a^* | b^* | | K/S | L^* | a^* | b^* | |
| Without mordant | 5 | 1.50 | 76.1 | 3.5 | 14.2 |  | 1.13 | 77.7 | 3.5 | 12.0 |  |
| | 10 | 1.60 | 76.0 | 3.5 | 15.0 | | 1.54 | 76.3 | 3.6 | 14.1 | |
| | 20 | 1.86 | 75.4 | 3.5 | 15.8 | | 1.86 | 75.9 | 3.4 | 15.8 | |
| AlK(SO ₄) ₂ (Al) | 5 | 1.65 | 78.2 | 0.4 | 23.6 |  | 1.65 | 79.4 | 0.1 | 27.2 |  |
| | 10 | 1.91 | 76.8 | 0.1 | 24.0 | | 1.81 | 78.0 | 0.1 | 27.6 | |
| | 20 | 2.55 | 74.9 | 0.7 | 22.9 | | 2.60 | 74.5 | 1.0 | 27.2 | |
| CuSO ₄ (Cu) | 5 | 3.27 | 63.8 | 0.04 | 21.1 |  | 2.32 | 65.5 | 2.0 | 19.6 |  |
| | 10 | 3.44 | 62.6 | 1.0 | 21.2 | | 2.62 | 64.0 | 2.8 | 19.5 | |
| | 20 | 4.12 | 59.6 | 2.2 | 21.1 | | 2.80 | 62.5 | 3.3 | 19.0 | |
| FeSO ₄ (Fe) | 5 | 3.93 | 40.1 | 1.3 | -1.3 |  | 4.54 | 40.6 | 1.3 | -1.0 |  |
| | 10 | 4.23 | 40.0 | 1.1 | -0.9 | | 4.81 | 37.1 | 1.3 | -1.0 | |
| | 20 | 4.62 | 38.5 | 1.2 | -1.1 | | 5.14 | 37.2 | 1.0 | -0.9 | |
| SnCl ₂ (Sn) | 5 | 2.15 | 83.8 | -0.2 | 28.5 |  | 1.58 | 84.7 | -0.9 | 22.5 |  |
| | 10 | 2.38 | 84.1 | -0.7 | 28.3 | | 2.01 | 83.5 | -0.4 | 27.4 | |
| | 20 | 2.67 | 83.4 | -0.8 | 30.4 | | 2.92 | 81.5 | 0.4 | 30.4 | |

Note: ¹ 20g/l dye concentration

Table 2. Colour value of wool fabric dyed with eucalyptus leaf extract by simultaneous mordanting and padding techniques, with using 10 g/l of metal mordants at different concentration of the dye







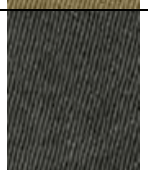
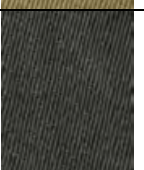
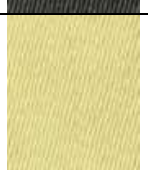
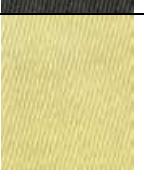
| Type of mordant | Dye Conc. (g/l) | Pad-batch on wool fabric | | | | | Pad-dry on wool fabric | | | | |
|---|-----------------|--------------------------|------|------|------|---|------------------------|------|------|------|---|
| | | K/S | L* | a* | b* | ¹ Dyed sample | K/S | L* | a* | b* | ¹ Dyed sample |
| Without mordant | 5 | 1.50 | 76.1 | 3.5 | 13.7 |  | 1.13 | 77.7 | 3.5 | 12.0 |  |
| | 10 | 1.60 | 76.0 | 3.5 | 15.1 | | 1.54 | 76.3 | 3.6 | 14.1 | |
| | 20 | 1.86 | 75.4 | 3.5 | 15.7 | | 1.86 | 75.9 | 3.4 | 15.8 | |
| AlK(SO ₄) ₂ (Al) | 5 | 1.11 | 81.3 | -1.2 | 19.2 |  | 0.93 | 82.1 | 0.6 | 16.9 |  |
| | 10 | 1.23 | 80.2 | -1.0 | 20.1 | | 1.10 | 80.8 | 0.4 | 18.5 | |
| | 20 | 1.39 | 79.3 | -0.8 | 21.4 | | 1.28 | 79.7 | 0.2 | 19.9 | |
| CuSO ₄ (Cu) | 5 | 2.50 | 66.8 | -1.1 | 19.5 |  | 1.84 | 68.5 | 0.4 | 17.3 |  |
| | 10 | 2.81 | 65.4 | -0.1 | 20.3 | | 2.12 | 66.8 | 0.6 | 18.7 | |
| | 20 | 3.06 | 63.7 | 0.2 | 20.2 | | 2.87 | 63.1 | 1.7 | 20.2 | |
| FeSO ₄ (Fe) | 5 | 3.18 | 53.0 | 1.8 | 8.4 |  | 2.28 | 55.8 | 1.5 | 6.1 |  |
| | 10 | 3.35 | 50.2 | 2.0 | 8.5 | | 2.71 | 51.1 | 1.5 | 3.9 | |
| | 20 | 3.86 | 46.7 | 1.5 | 2.7 | | 3.13 | 45.7 | 1.6 | 1.0 | |
| SnCl ₂ (Sn) | 5 | 1.54 | 85.4 | -0.3 | 25.6 |  | 1.52 | 86.6 | -0.3 | 24.8 |  |
| | 10 | 1.90 | 85.0 | -0.2 | 27.8 | | 1.88 | 85.1 | -0.2 | 25.7 | |
| | 20 | 2.05 | 84.0 | -0.1 | 28.1 | | 2.01 | 84.1 | -0.2 | 26.6 | |

Note: ¹ 20g/l dye concentration

Table 3. Colour value of wool fabric dyed with eucalyptus leaf extract by post-mordanting and padding techniques, with using 10 g/l of metal mordants at different concentration of the dye

6.3 Effect of quantity of mordant concentrations, time/ temperature on pad-dry and batching time on pad-batch

Table 4 shows the colour values of wool fabric dyed with eucalyptus leaf extract by varying quantity of mordant concentrations. All measured sample showed the greatest λ_{max} value at 400 nm. It can be seen that the K/S values increase with an increase of mordant concentration. The dyed uptake values were greater at the higher mordant concentration. This could be attributed to the darkening and dulling of shades due to mordant effect. Little different between the two padding techniques utilized for the study is observed. Wool fabric dyed with eucalyptus leaf extract in the absence mordant showed yellowish brown shades. Comparison of four metal mordants showed that the ferrous sulfate metal mordant gave the highest depth of shade on wool fabric. Thus ferrous sulfate was the best mordant during mordanting method of dyeing. This could be attributed to difference in CIELAB values of the dyed samples. The mordant activity of the five sequences was as follows: Fe > Cu > Al > Sn > without mordanted in wool fabric, the absorption of colour by wool fabric was enhanced by using metal mordants.

| Type of mordant | Conc. (g/l) | Pad-batch on wool fabric | | | | | Pad-dry on wool fabric | | | | |
|---|-------------|--------------------------|------|------|------|---|------------------------|------|------|------|---|
| | | K/S | L* | a* | b* | ¹ Dyed sample | K/S | L* | a* | b* | ¹ Dyed sample |
| Without mordant | - | 1.86 | 75.4 | 3.5 | 15.8 |  | 1.86 | 75.9 | 3.4 | 15.8 |  |
| AlK(SO ₄) ₂ (Al) | 5 | 2.18 | 76.2 | 0.5 | 28.2 |  | 2.09 | 77.8 | 0.8 | 28.0 |  |
| | 10 | 2.55 | 74.9 | 0.7 | 29.9 | | 2.60 | 74.5 | 1.0 | 29.1 | |
| | 20 | 3.91 | 72.8 | 1.0 | 32.1 | | 3.97 | 72.0 | 1.2 | 31.6 | |
| CuSO ₄ (Cu) | 5 | 3.84 | 61.3 | 2.4 | 20.9 |  | 3.80 | 62.4 | 2.3 | 20.2 |  |
| | 10 | 4.12 | 59.6 | 2.7 | 21.1 | | 4.08 | 60.0 | 3.3 | 19.0 | |
| | 20 | 5.00 | 54.4 | 2.8 | 23.1 | | 4.87 | 55.7 | 3.2 | 20.1 | |
| FeSO ₄ (Fe) | 5 | 4.81 | 40.0 | 1.0 | -0.9 |  | 4.95 | 39.1 | 1.1 | -0.6 |  |
| | 10 | 4.98 | 38.5 | 1.2 | -1.1 | | 5.14 | 37.0 | 1.0 | -0.9 | |
| | 20 | 7.28 | 36.9 | 0.7 | -1.4 | | 7.60 | 36.0 | 0.9 | -0.7 | |
| SnCl ₂ (Sn) | 5 | 2.64 | 83.3 | -1.6 | 32.2 |  | 2.66 | 83.1 | -2.3 | 31.8 |  |
| | 10 | 2.67 | 83.4 | -1.2 | 30.4 | | 2.71 | 82.5 | -2.4 | 30.4 | |
| | 20 | 3.11 | 81.8 | -2.1 | 35.3 | | 3.13 | 81.4 | -2.7 | 34.6 | |

Note: ¹ 20g/l metal mordants concentration

Table 4. Colour value of wool fabric dyed with eucalyptus leaf extract by simultaneous mordanting and padding techniques, with using 20 g/l of dye concentration at different concentration of the mordant

From the results, it is clear that ferrous sulfate and copper sulfate mordants are well known for their ability to form coordinate complexes and in this experiment both readily chelated with the dye. As the coordination numbers of ferrous sulfate and copper sulfate are 6 and 4 respectively, some co-ordination sites remained unoccupied when they interacted with the fiber. Functional groups such as amino and carboxylic acid groups on the fiber can occupy these sites. Thus this metal can form a ternary complex on one site with the fiber and on the other site with the dye (Bhattacharya & Shah, 2000). Stannous chloride and alum metals formed weak coordination complexes with the dye, they tend to form quite strong bonds with the dye but not with the fiber, so they block the dye and reduce the dye interaction with the fiber (Bhattacharya & Shah, 2000).

The effect of time and temperature on colour strength (K/S) value was evaluated by padding a sample of wool fabric with eucalyptus leaf extract and ferrous sulfate as mordant. The samples were processed only by drying condition were 40°C, 60°C and 90°C for 1, 3, 5 and 10 minutes. The K/S values obtained are shown in Fig. 4. It is clear that the colour strength (K/S) values increase with in crease in the drying time and temperature in wool fabric. A study of Fig. 4 reveals that the high colour strength values (ca. 7.60) was achieved for the

wool fabric on drying at 90°C for 5 minutes. The pad-batch dyeing process was carried out at room temperature with batching times of different lengths to assure an operation as economic as possible. Fig. 5 shows that low colour strength required a period of 1 hour, medium colour strength of 6-12 hours and high colour strength a period of 24 hours. The colour strength obtained was increased as the batching time increased for wool fabrics.

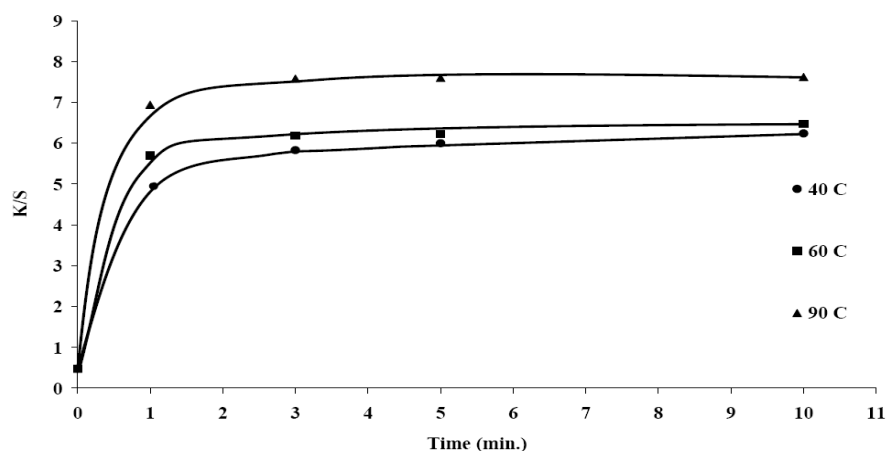


Fig. 4. Effect of drying time and temperature of pad-dry technique on the colour strength (K/S values) of wool fabric dyed with 20 g/l eucalyptus leaf extract and using 20 g/l ferrous sulfate by using simultaneous mordanting

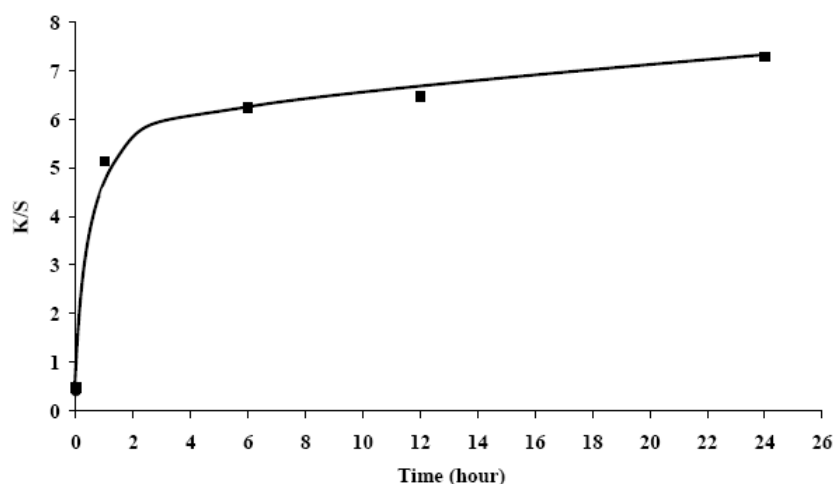


Fig. 5. Effect of batching time of pad-batch technique on the colour strength (K/S values) of wool fabric dyed with 20 g/l eucalyptus leaf extract and using 20 g/l ferrous sulfate by using simultaneous mordanting

6.4 The percentage yield (exploitation) of wool fabric dyed with eucalyptus leaf extract by simultaneous pad-dyeing

It was estimated that the best shades (deep and colour fastness) are obtained when mordanting with ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and, therefore, this mordant was used for the experiments. The following concentration range of eucalyptus leaf extract and mordant $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in the same concentration was used: 1, 5, 10, 20, 30, and 40 g/l, and in all cases anionic wetting agent in the concentration of 1 g/l was added to the padding bath. Glacial

acetic acid was added to maintain the pH of the liquid at 4. The simultaneous padding was carried out at room temperature in a two-bowl padding mangle using 80% pick up. After padding (2 seconds), the samples were dried at 90°C for 5 minutes and after 1 hour, all samples were repeatedly rinsed in warm water at 60°C until the rinsing water remained colourless. The rinsed water was collected with the rest of dyeing bath in the volumetric flask and filled up to the defined volume for absorbance measurement by UV-vis spectrophotometer (at the wavelength of 270 nm at which the maximum absorbance was recorded). The concentration of eucalyptus leaf extract fixed in the fiber and percentage of its use (percentage of yield) from bath on fiber were calculated from the absorbance of the rinsing water by using the standard graph. Relationship between bath concentration and padding condition were calculated from Eq. (1) to Eq. (6) (Mongkhorrattanasit et al., 2009). We assume when the initial dye concentration in the pad bath is C_0 (g/l). The quantity of dye transported by fabric is C_{pi} (mg/g)

$$C_{pi} = \frac{\% \text{ pickup}}{100} \cdot C_0 \quad (1)$$

The concentration of dye in conjoined-water after rinsing can be expressed as:

$$C_r = \frac{\text{Absorbance}}{\varepsilon \cdot l} \quad (2)$$

where C_r = the concentration of dye in conjoined-water (mg/l), ε = absorption coefficient (l/mole.cm) and l = layer of solution (cm). Then the concentration of dye, which was stripped from material, is C_w (mg/g)

$$C_w = \frac{C_r \cdot V}{1,000 \text{ g}} \quad (3)$$

where V = total volume after rinsing (ml) and g = weight of material (g). The concentration of dye absorbed on material, C_s (mg/g) was calculated as:

$$C_s = C_{pi} - C_w \quad (4)$$

The percentage of dye which stripped from the material can be shown as Eq. (5)

$$W = \frac{C_w \cdot 100}{C_{pi}} \quad (5)$$

where W = the percentage of dye which stripped from the material (%). And the percentage of exploitation of dye (yield), E (%) can be calculated as:

$$E = 100 - W \quad (6)$$

Wool fabric dyed with the water extract of eucalyptus leaves in the presence of the FeSO_4 mordant in the same padding bath shows a colour range of a brown grey shade to a dark grey shade. In Table 5, the results are presented. The yield (exploitation) of the colouring component of eucalyptus leaf extract in wool fabric is about 68%–52% from the lowest to the highest concentrations, and this corresponds to the medium deep brown-grey shades in the concentrations of more than 20 g/l eucalyptus leaf extract.

| C_0 (g/l) | Percentage of pick up | C_{pi} (mg/g) | C_s (mg/g) | Yield (%) | K/S value (400 nm) |
|----------------|--------------------------|--------------------|-----------------|--------------|-------------------------|
| 1 | 80 | 0.8 | 0.5 | 68.0 | 1.8 |
| 5 | 80 | 4 | 2.5 | 62.8 | 2.8 |
| 10 | 80 | 8 | 4.2 | 53.2 | 3.7 |
| 20 | 80 | 16 | 8.3 | 52.0 | 3.9 |
| 30 | 80 | 24 | 12.6 | 52.6 | 4.0 |
| 40 | 80 | 32 | 16.0 | 52.2 | 4.5 |

Table 5. Percentage yield and K/S values obtained by the simultaneous pad-dyeing/mordant of wool fabric

6.5 UV protection properties of wool fabric dyed with eucalyptus leaf extract

The transmittance and UPF values of the original wool fabric, and fabrics dyed with the eucalyptus leaf extract were measured using Shimadzu UV3101 PC (UV-VIS-NIR Scanning Spectrophotometer) in the range of 190 nm to 2100 nm. The UPF value of the fabric was determined from the total spectral transmittance based on AS/NZ 4399:1996 as follows (Gies et al., 2000)

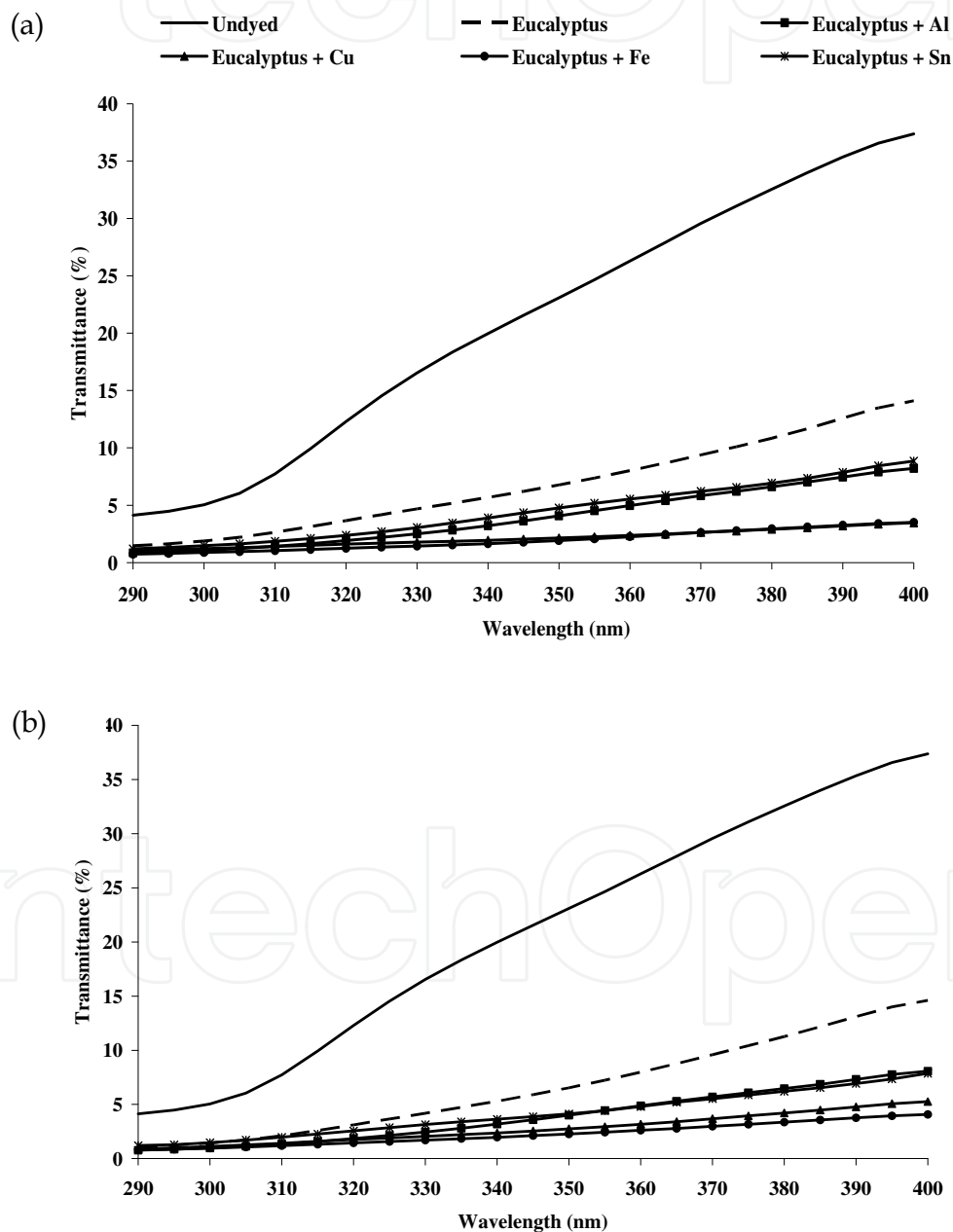
$$UPF = \frac{\sum_{290}^{400} E_{\lambda} S_{\lambda} \Delta_{\lambda}}{\sum_{290}^{400} E_{\lambda} S_{\lambda} T_{\lambda} \Delta_{\lambda}}$$

where E_{λ} is the relative erythemal spectral effectiveness (unitless), S_{λ} is the solar ultraviolet radiation (UVR) spectral irradiance in $W.m^{-2}.nm^{-1}$, T_{λ} is the measured spectral transmission of the fabric, Δ_{λ} is the bandwidth in millimeter, and λ is the wavelength in nanometre. The UVR band consists of three regions: UV-A band (320 nm to - 400 nm), UV-B band (290 nm to 320 nm), and UV-C band (200 nm to 290 nm) (Feng et al., 2007). The highest energy region, the UV-C band, is absorbed completely by oxygen and ozone in the upper atmosphere. Of the solar UV radiation reaching the earth's surface, 6% is in the UV-B region and 94% in the UV-A region (Allen & Bain, 1994). UV-A causes little visible reaction on the skin but has been shown to decrease the immunological response of skin cells (Sarkar, 2003). UV-B is the most responsible for the development of skin cancers (Sarkar, 2003). Therefore, the transmittance of UVR, including UV-A and UV-B, through the fabrics was evaluated in this experiment. Fabrics with a UPF value in the range of 15-24 are defined as providing "good UV protection", 25-39 as "very Good UV protection", and 40 or greater as "excellent UV protection" (Sarkar, 2003). There is no rating assigned if the UPF value is greater than 50.

A commercially produced plain-weave wool fabric (thickness 0.36 mm, weight 193 g/m², fabric count per inch 62 x 54) was used in this experimental. The thread count, fabric thickness, and fabric weight characteristics of the wool fabric was in accordance with ASTM D3775-98, ISO 5084-1996, and ISO 3801-1997, respectively. A pre-mordanting padding process was used in this study.

To investigate the UV-protection property of eucalyptus leaves dye, UV transmittance spectra of the wool fabric with or without dyeing and the dyed wool fabric with mordants were

compared. The percent UV transmittance data of wool fabric dyed with and without a mordanting agent are shown in Fig. 6. The results show significantly different between the dyed and undyed fabrics, which yields a high UV transmittance. The UV transmittance of the undyed wool was in the range of about 4-12% in the UV-B band and about 12-37% in the UV-A band. This indicates that the resistance of undyed fabrics to ultraviolet ray was very poor. While the UV transmittance of wool fabrics dyed by eucalyptus leaf extract appeared to be lower than 5% in the UV-B region. Generally, the UV protection property of fabrics is evaluated as good when the UV transmittance is less than 5% (Feng et al., 2007 ; Teng & Yu, 2003).



Note: Al = $AlK(SO_4)_2$, Cu = $CuSO_4$, Fe = $FeSO_4$, Sn = $SnCl_2$

Fig. 6. UV transmission of wool fabric dyed with 5 g/l eucalyptus leaf extract dye solution, using 10 g/l mordants by (a) pad-batch and (b) pad-dry techniques.

For the samples mordanted with $\text{AlK}(\text{SO}_4)_2$, CuSO_4 , FeSO_4 and SnCl_2 , the percent UV-B transmittance was in the range of 0.8-1.9 %, 1.0-1.6%, 0.7-1.1%, and 1.2-2.7%, respectively for pad-batch and 0.7-1.8 %, 0.9-1.7%, 0.7-1.4%, and 1.2-2.5%, respectively for pad-dry. It is clearly seen that the values of the spectral transmittance are decreased with the mordants such as $\text{AlK}(\text{SO}_4)_2$, CuSO_4 , FeSO_4 , and SnCl_2 and different mordants had different effects on the spectral transmittance of the fabric dyed (Feng et al., 2007). Additionally, the colour and colour depth of the fabric can be related to UV transmittance in which light colours transmit more UV radiation than dark colours (Wilson et al., 2008). Table 6 shows the UPF values and protection class of wool fabric dyed by eucalyptus leaves with and without metal mordants by pad-dry and pad-batch dyeing techniques. Little difference is observed between the two padding techniques utilized for this study. The undyed fabric had a high transmittance and a very low UPF value of 10.8. The dyed samples without metal mordant in both dyeing techniques show UPF values between 32.8 and 35.4, which can be rated as offering Very good UV protection (UPF values between 25 and 39).

From the transmission data and the corresponding UPF values, all metal mordants used in this study caused a reduction in UV radiation transmission through the wool fabric. Wool fabric dyed by the metal mordants at 5 g/l concentrations of dye in the pad-dry and the pad-batch dyeing techniques could be classified as offering Excellent UV protection (UPF values 40 or greater). Wool fabrics, which after dyeing with and without mordant are rated as very good to excellent UV protection because wool fabric have low porosity and high weight and thickness. Therefore, wool fabric gives high UPF and permitting transmission of less UV radiation.

| Mordant | Dye Conc. (g/l) | Pad-batch | | Pad-dry | |
|-----------------------------|-----------------|-----------|----------------------|---------|----------------------|
| | | UPF | UPF Protection class | UPF | UPF Protection class |
| - | Un-dyed | 10.8 | No Class | 10.8 | No Class |
| Without | 5 | 32.8 | Very good | 35.4 | Very good |
| $\text{AlK}(\text{SO}_4)_2$ | 5 | 59.0 | Excellent | 55.1 | Excellent |
| CuSO_4 | 5 | 67.9 | Excellent | 65.0 | Excellent |
| FeSO_4 | 5 | 85.3 | Excellent | 81.8 | Excellent |
| SnCl_2 | 5 | 46.9 | Excellent | 45.5 | Excellent |

Table 6. UPF values, protection class, and K/S values of wool fabric dyed with 5 g/l eucalyptus leaf extract dye solution and using 10 g/l mordants

6.6 Effect of dyeing technique on fastness properties

The colour fastness to washing, light, perspiration, water and rubbing of the dyed samples was determined according to ISO 105-C06 A1S:1994, ISO 105-B02:1994, ISO 105 E04: 1994, ISO 105-E01: 1994 and ISO 105-X12:2001, respectively.

The fastness rating of wool fabric dyed with or without mordants at 20 g/l dye concentration is presented in Tables 7 through 11. When comparing the fastness rating of the samples dyed using the two padding techniques, it can be postulated that the pad-batch technique gives nearly the same fastness properties as the pad-dry technique. Table 7 indicates that the washing fastness ratings of wool fabric dyed with eucalyptus leaves were very good (4-5). However, the light fastness was only fair (3-4), as shown in Table 8. The colour fastness to rubbing is shown to be in range of 4 to 4-5 (good to very good), except for fabrics mordanted with FeSO_4 , whose rating was only 3-4 (fair to good) when subjected to wet rubbing, as shown in Table 9. The rating obtained for colour fastness to water in term of the degree of colour change and colour staining were very good (4 to 4-5), as shown in Table 10. The colour fastness to perspiration in acid and alkaline solution of fabrics dyed with and without metal mordants are shown in range of 4 to 4-5 as seen in Tables 11. The good fastness properties of wool fabric dyed with eucalyptus leaf extract may be attributed to the fact that these dyes contain tannin, which may help covalent bond formation with the fiber, thereby resulting in good fixation on the fibrous material. Again, these tannins, having a phenolic structure, can form metal chelation with different mordants. Hence, after mordanting, these tannins are insoluble in water, ultimately improving washing, water, and perspiration fastness (Agarwal & Patel, 2001).

| Fastness | Pad-batch | | | | | Pad-dry | | | | |
|-----------------|-----------|-----|-----|-----|-----|----------|-----|-----|-----|-----|
| | With-out | Al | Cu | Fe | Sn | With-out | Al | Cu | Fe | Sn |
| Colour change | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Colour staining | | | | | | | | | | |
| -Acetate | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Cotton | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Nylon | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| -Polyester | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Acrylic | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Wool | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 |

Note: Al = $\text{Al}(\text{SO}_4)_2$, Cu = CuSO_4 , Fe = FeSO_4 , Sn = SnCl_2

Table 7. Colour fastness to washing at 40°C (ISO 105-C06 A1S: 1994)

| Pad-batch (Colour change) | | | | | Pad-dry (Colour change) | | | | |
|------------------------------|----|-----|----|----|----------------------------|----|-----|-----|----|
| Without | Al | Cu | Fe | Sn | Without | Al | Cu | Fe | Sn |
| 3 | 3 | 3-4 | 4 | 3 | 3 | 3 | 3-4 | 3-4 | 3 |

Note: Al = $\text{Al}(\text{SO}_4)_2$, Cu = CuSO_4 , Fe = FeSO_4 , Sn = SnCl_2

Table 8. Colour fastness to light (ISO 105-B02: 1994).

| mordant | Colour staining | | | | | | | |
|------------------------------------|-----------------|-----|----------------|-----|----------------|-----|----------------|-----|
| | Pad-batch | | | | Pad-dry | | | |
| | Warp direction | | Weft direction | | Warp direction | | Weft direction | |
| | Dry | Wet | Dry | Wet | Dry | Wet | Dry | Wet |
| without | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| AlK(SO ₄) ₂ | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| CuSO ₄ | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4 | 4-5 | 4 |
| FeSO ₄ | 4 | 4 | 4 | 3-4 | 4 | 3-4 | 4 | 3-4 |
| SnCl ₂ | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |

Table 9. Colour fastness to rubbing (ISO 105-X12: 2001).

| Fastness | Pad-batch | | | | | Pad-dry | | | | |
|-----------------|-----------|-----|-----|-----|-----|---------|-----|-----|-----|-----|
| | Without | Al | Cu | Fe | Sn | Without | Al | Cu | Fe | Sn |
| Colour change | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Colour staining | | | | | | | | | | |
| -Acetate | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Cotton | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 |
| -Nylon | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| -Polyester | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 |
| -Acrylic | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Wool | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4 |

Note: Al = AlK(SO₄)₂, Cu = CuSO₄, Fe = FeSO₄, Sn = SnCl₂

Table 10. Colour fastness to water (ISO 105-E01: 1994)

| Fastness | Pad-batch | | | | | Pad-dry | | | | |
|-----------------|-----------|-----|-----|-----|-----|---------|-----|-----|-----|-----|
| | Without | Al | Cu | Fe | Sn | Without | Al | Cu | Fe | Sn |
| Acid | | | | | | | | | | |
| Colour change | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Colour staining | | | | | | | | | | |
| -Acetate | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Cotton | 4 | 4-5 | 4 | 4 | 4-5 | 4 | 4-5 | 4 | 4 | 4-5 |
| -Nylon | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 |
| -Polyester | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 |
| -Acrylic | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Wool | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 |
| Alkaline | | | | | | | | | | |
| Colour change | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| Colour staining | | | | | | | | | | |
| -Acetate | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Cotton | 4 | 4-5 | 4 | 4 | 4-5 | 4 | 4-5 | 4 | 4 | 4-5 |
| -Nylon | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 |
| -Polyester | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Acrylic | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| -Wool | 4-5 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4 | 4-5 |

Note: Al = AlK(SO₄)₂, Cu = CuSO₄, Fe = FeSO₄, Sn = SnCl₂

Table 11. Colour fastness to perspiration (ISO 105-E04: 1994)

7. Potential of eucalyptus leaves dye

7.1 Potential commercial applications

Natural dyes cannot be used as simple alternatives to synthetic dyes and pigments. They do, however, have the potential for application, in specified areas, to reduce the consumption of some of the more highly polluting synthetic dyes. They also have the potential to replace some of the toxic, sensitizing and carcinogenic dyes and intermediates (Deo & Desai, 1999). Eucalyptus leaves, as natural dye, has greater potential because it is grown already on an industrial scale. It also shows good fastness on wool substrate.

7.2 Potential effluent problems

The effluent problems of synthetic dyes occur not only during their application in the textile industry, but also during their manufacture, and possibly during the synthesis of their intermediates and other raw materials. The application of synthetic dyes also requires metal salts for exhaustion, fixation, etc (Deo & Desai, 1999). Natural dyes, like eucalyptus leaves do not cause damage the environment by their extraction and many could be used satisfactorily without mordants, although it is true that the use of mordant improves the depth of shade for natural dyes. These mordants are normally metal salts and hence damage to the environment is still possible, albeit to a smaller extent than for synthetic dyes in textile applications. The research in this field has already identified a few "natural mordant", such as *Entada spiralis Ridl* (Chairat et al., 2007) and harda (*Chebulic myrabolan*) (Deo & Desai, 1999). The avoidance of metal-based mordants, or their replacement by natural mordants, may assist in the preservation of the environment.

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9. Conclusions

A wool fabric dyed in a solution containing the eucalyptus leaf extract showed a shade of pale yellowish-brown. The exception was when the fabric was dyed with the ferrous sulfate mordant, resulting in a shade of dark greyish-brown. The yield (exploitation) of the coloring component of eucalyptus leaf extract is good in wool fabric (about 68%–52% from the lowest to the highest concentrations). It can be observed that the K/S values increase with an increase of dye concentration. Little difference between the two padding techniques utilized for the wool fabric dyes by three mordanting methods, except wool fabrics mordanted with copper sulfate whose gave a high K/S values on the pad-batch technique than pad-dry technique. In all cases ferrous sulfate mordant yielded the best dyeing results, and the next good result was obtained in the order of copper sulfate, stannous chloride and alum. As observed from the K/S values, in the case of wool fabrics dyed with alum by using post-mordanting method gave lower colour strength than without mordant. Alum and ferrous sulfate were the best mordant during simultaneous mordanting method of dyeing. However, copper sulfate showed the best mordant during simultaneous mordanting and pre-mordanting method of dyeing. For the K/S value on dyed wool fabrics were only little different using stannous chloride as mordant during three mordanting methods. The fastness properties ranged from good to excellent, while light fastness was fair to good. It

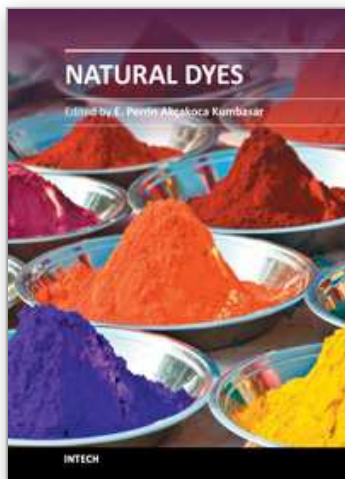
was observed that the ultraviolet (UV) protection factor (UPF) values rated as excellent for the wool fabric. In addition, a darker colour, such as that provided by a ferrous sulfate mordant, gave better protection because of higher UV absorption.

The application of eucalyptus leaves dye on wool fabrics by pad-batch and pad-dry technique of dyeing can be considered as an affective eco-option because it gives extremely good results with substantial minimization of processing cost. In case of pad-dry technique, the average hot air consumption is considerably high whereas no hot air is being consumed in cold pad-batch process which leads to energy conservation. However, the time employed for the fixation of eucalyptus leaves dye is very long in cold pad-batch technique. So, these techniques can be considered as best suitable for small scale industries or cottage dyeing of eucalyptus leave.

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Textile materials without colorants cannot be imagined and according to archaeological evidence dyeing has been widely used for over 5000 years. With the development of chemical industry all finishing processes of textile materials are developing continuously and, ecological and sustainable production methods are very important nowadays. In this book you can find the results about the latest researches on natural dyeing.

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