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# Dyeing of Organic Cotton Fabric using Conventional and Ultrasonic Exhaust Dyeing Method

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## ABSTRACT

In this research dyeing behavior of organic cotton woven fabric using ultrasonic technique and conventional dyeing method has been compared. The fabric samples were dyed with reactive dyes Drimarene Red CI-5B and Drimarene Blue CI-BR (0.5% owf) using exhaust dyeing method. The samples were ultrasonically dyed at varied temperature (60, 50 and 40°C) for 60, 50, 40 and 30 minutes and for conventional method at varied temperature but at recommended time, 60 minutes. For optimizing the dyeing behavior, the samples were causticized by pad-batch method and then dyed with ultrasonic technique at varied temperature and time. It has been observed organic cotton fabric dyed using ultrasonic exhaust method at 60°C for 50 minutes gives highest  $(K/S)_{\lambda_{max}}$  value, excellent fastness property, deeper dye diffusion and less surface deterioration compared to the conventional dyeing method. Moreover, causticized and dyed sample with ultrasonic technique at 60°C for 30 minutes gives colour strength value almost equal to the conventional recommended dyeing method. Hence, dyeing of organic material using ultrasonic exhaust method saves energy and time.

**Key Words:** Ultrasonic, Exhaust Dyeing, Organic Cotton Fabric, Temperature, Time.

## 1. INTRODUCTION

Organic cotton fibers are produced from non-genetically modified plants and are grown without synthetic agricultural chemicals [1]. The fibers offer surface protection of groundwater quality and reduce environmental damages. In addition, nowadays along with textile fibers, textile processing sector is focusing on the conservation of energy. This can be achieved by processing the textile materials using various techniques such as infrared heating, ultrasonic dyeing, plasma technique, radiofrequency, electrochemical

dyeing, microwaves etc each of these techniques are applied by different method [2-4]. However, dyeing process effected on the physical and mechanical properties of the cotton fabric [5-6].

Among all, ultrasonic dyeing is a novel technique to save time, cost, energy and provides high value of dye uptake. Ultrasonic waves are high frequency (20-40 KHz) and high energy waves then the sound waves and can be used for a variety of purposes. It speeds up the rate of collision of

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molecules, hence resulting in generation of heat and energy. The use of ultrasonic energy in dyeing optimizes the dyeing process. Moreover, it can be used for dyeing both hydrophilic and hydrophobic fibers. Brauer reported that the ultrasonic dyeing technique saved the dyeing time of cellulosic fabric with vat dyes. Compared to the conventional desizing technique; [7] ultrasonic desizing reduced the fibre degradation while the degree of whiteness and wet ability of the cellulosic fabric remained same. Ultrasonic scouring of wool fibres reduced the fiber damage. Similarly, in bleaching of cotton fiber with hydrogen peroxide using ultrasound technique at the frequency of 20 KHz, increased the bleaching rate and enhanced the degree of whiteness. The mercerization of cotton material using ultrasonic technique swelled 35% fiber diameter [8].

The use of ultrasonic energy in dyeing of cotton fabric samples with monochlorotriazine and vinylsulphone reactive dyes could result in energy saving, less water consumption, increased colour depth and reduced processing time. Linen fabric sample dyed using ultrasonic technique at 50°C were given slightly higher colour strength values than those obtained using conventional dyeing method at 80°C [9].

The colour strength values obtained for the nylon-6 samples dyed using ultrasonic power were higher than those obtained using conventional method. In addition, the effect of alkaline soaping treatment on dye fixation for the dyed fabric samples with different class of reactive dyes, at both acidic and neutral pH, gave good to excellent fastness properties with ultrasonic probe technique than with conventional heating. The utilization of ultrasonic wave for cold pad-batch dyeing decreased the batching time at minimum quantity of alkali concentration [10-11]. Knitted interlock fabric dyed at 600 KHz ultrasonic

frequencies, temperature (30-50°C) and salt concentrations (60g dm<sup>-3</sup>) showed deeper hue in comparison with that achieved by conventional method [12].

Hence, radcure techniques using ultrasonic, minimize the processing time, temperature and energy. Much work has been carried out on pre-treatment and dyeing of cotton and other synthetic material but no work was done on the comparison of dyeing behaviour of organic cotton material using conventional and exhaust deying ultrasonic technique. Therefore, in this research organic cotton material is dyed with reactive dyes; Drimarene Red CI-5B and Drimarene Blue CI-BR (0.5% owf) using conventional and ultrasonic technique at varied temperature and time. For optimizing the dyeing bahviour the fabric samples were causticized and dyed with ultrasonic technique. The dyeing behaviour was then assessed with colour strength values, washing fastness, dye uniformity and surface effect using scanning electron microscopy.

## 2. MATERIALS

100% bleached organic cotton fabric as mentioned in Table 1 was purchased from Yunus Textile Mills, Pakistan. Reactive dyes, Drimarene Red CI-5B and Drimarene Blue CI-BR and other auxiliaries such as detergent, Ladipur RSK and leveling agent, Drimagen E2R were kindly supplied from Clariant, Karachi, Pakistan. Common salt, NaCl and acetic acid were marketed by Merck, Germany.

TABLE 1 FABRIC SPECIFICATION

Weave	Plain
Ends Per Inch	30
Picks Per Inch	30
Warp Count, tex	76
Weft Count, tex	68
Gram Per Square Meter	150

### 3. METHODS

#### 3.1 Conventional Dyeing

Organic cotton fabric sample of weight 3gm was taken and dyed (0.5% owf) on Rapid HT dyeing machine using conventional dyeing method as shown in Fig. 1 [13]. The dyeing process was carried out at liquor to goods ratio of 20:1 at 60°C with dye, Drimagen E2R solution w/v (2 g l<sup>-1</sup>) and salt solution w/v (60 g l<sup>-1</sup>). After exhaustion (30 minutes) alkali solution w/v (15 g l<sup>-1</sup>) was added and then processed for further 45 minutes at the same temperature. After dyeing, fabric sample was washed with cold water for 10 min then warm water at 80°C for 10 minutes. The soaping was done at 90°C for 30 minutes and before dried in an oven it was again warm and cold wash for 10 minutes each. For comparison the conventional dyeing to the ultrasonic dyeing method, it was proceeding at varied temperature such as 60, 50 and 40°C but at recommended time 60 minutes (C60) for exhaustion and fixation phases.

#### 3.2 Ultrasonic Dyeing

Organic cotton sample of weight 3gm was taken and dyed (0.5% owf) on Ultrasonic Cleaner KB200 Series using same recipe and procedure as mention in conventional dyeing

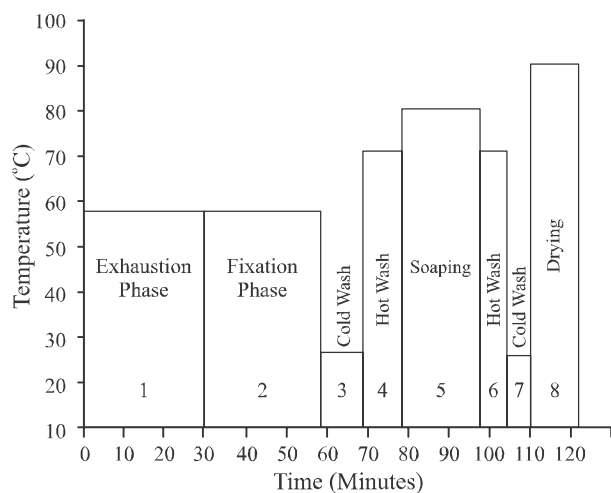


FIG. 1. DYEING PROCEDURE

method (Fig. 1). In addition, the fabric samples were dyed at varied temperatures such as 60, 50 and 40°C and at each temperature 60 minutes (U60), 50 minutes (U50), 40 minutes (U40) and 30 minutes (U30) for exhaustion and fixation phases. However, washing was carried out using recommended method [13].

#### 3.3 Causticisation

For optimizing the organic cotton fabric dyeing behavior, the fabric samples were pre-treated using caustic, sodium hydroxide (2 mol dm<sup>-3</sup>) by pad-batch method for 30 minutes [14]. The fabric samples were then dyed by ultrasonic technique at varied temperature and time as discussed in preceding Section.

#### 3.4 Colour Strength

The spectral reflectance values (400-700nm) were measured using a Data Colour Spectrophotometer. For each sample, readings were taken four times, each at different position. The percentage reflectance values 'R' were converted to K/S values, using the formula shown in Equation (1).

$$K/S = \frac{(100 - R)^2}{200R} \quad (1)$$

#### 3.5 Washing Fastness

Fastness to washing test was performed according to the standard of BS EN ISO 105-CO3: 1998 or BS EN 20105-CO3:1993. After washing, fabric sample and multi fibres stripe were then rated (1-5) using grey scale and staining scale. Where 1 means poor fastness and 5 means excellent fastness.

#### 3.6 Dye Uniformity

Uniformity of dyeing was assessed using Motic video microscope. For preparing the yarn cross-sectional images; 7-10 yarns were taken from the dyed fabric and were laid

parallel [14]. The yarn was then passed from the slide; the extra yarns were then cut using sharp blade. The slide was then placed on a video microscope and at constant magnification yarns cross-sectional images were then captured.

### 3.7 Surface Effects

For assessing the surface distortion of dyed fabric sample with conventional and ultrasonic dyeing method, fabric samples of 1x1cm was taken. The fabric images at 700 magnifications were captured using JEOL JSM 5380 LV. The fabric images were then visually analyzed.

## 4. RESULTS AND DISCUSSION

### 4.1 Effect of $(K/S)_{\lambda_{max}}$ Value at 60, 50 and 40°C Dyeing Temperatures

It has been observed from Figs. 2-3 that the ultrasonic dyed samples with Drimarene Blue Cl-BR and Drimarene Red Cl-5B (0.5% owf) at 60°C dyeing temperature for 50 minutes gives the highest  $(K/S)_{\lambda_{max}}$  values than the conventional dyed samples. However, dyeing time for 40 and 30 minutes, the  $(K/S)_{\lambda_{max}}$  values are decreasing at all dyeing temperature 60, 50 and 40°C. The colour strength value for 50 minutes dyeing time is higher than the 60 minutes dyeing time at all dyeing temperature (60, 50 and 40°C). It is because for 60 minutes dyeing cycle fibre fibrils are propensities on the fabric surface. Hence, hinder the reflectance of light and gives less colour strength value.

When comparing the colour strength values among 60, 50 and 40°C dyeing temperature, dyeing at recommended temperature (60°C) gives highest colour strength value than the 50 and 40°C both by ultrasonic and conventional dyeing methods. Moreover, the colour

strength value dyed with Drimarene Red Cl-5B is higher than the Drimarene Blue Cl-BR at 60°C dyeing temperature for 50 minutes. It is due to the difference in dye molecular size of Drimarene Red Cl-5B and Drimarene Blue Cl-BR dye. The small molecular size of dye molecules make easy for dye molecules to penetrate into the amorphous regain of polymer of fibre filaments at low temperature and less time.

The percent  $(K/S)_{\lambda_{max}}$  value relative to conventional dyeing method for 60 minutes (C60) to ultrasonic dyed samples at various temperature and time are shown in Figs. 4-5. It has been observed that the  $(K/S)_{\lambda_{max}}$  value of conventional dyed samples is lower to all ultrasonic dyeing method except dyeing for 40 and 30 minutes at 60 and 50°C for exhaustion and fixation phases. Ultrasonic dyeing at 40°C gives the lowest  $(K/S)_{\lambda_{max}}$  value on the contrary, it shows much higher  $(K/S)_{\lambda_{max}}$  values relative to C60. It is because

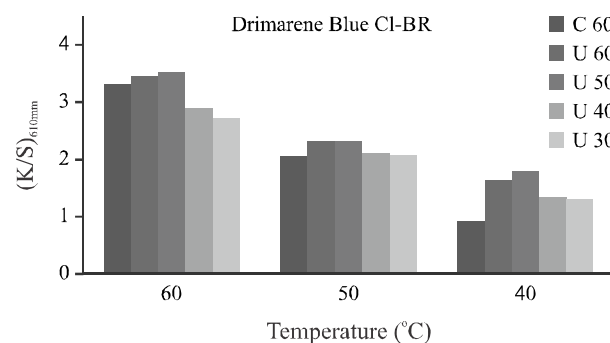


FIG. 2.  $(K/S)_{610nm}$  VALUES OF DRIMARENE BLUE CL-BR DYED SAMPLES

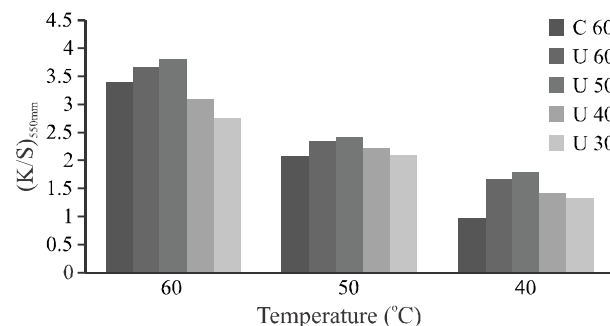


FIG. 3.  $(K/S)_{550nm}$  VALUES OF DRIMARENE RED CL-5B DYED SAMPLES

radcure technique using ultrasonic waves provides much higher energy to dye molecules thus increases the rate of diffusion and exhaustion as compared to conventional dyeing method.

The ultrasonic dyeing of organic cotton woven fabric shows less than to 120% improvement when dyeing at 60 and 50°C for both 60 and 50 minutes dyeing cycle compared to conventional dyeing.

#### 4.2 $(K/S)_{\lambda_{max}}$ Values of Causticized Dyed Samples

Figs. 6-7 shows that pre-treatment using sodium hydroxide improves the  $(K/S)_{\lambda_{max}}$  value. The trend of  $(K/S)_{\lambda_{max}}$  value is almost same for the Drimarene Red Cl-5B and Drimarene Blue Cl-BR (0.5% owf). It is observed that the  $(K/S)_{\lambda_{max}}$

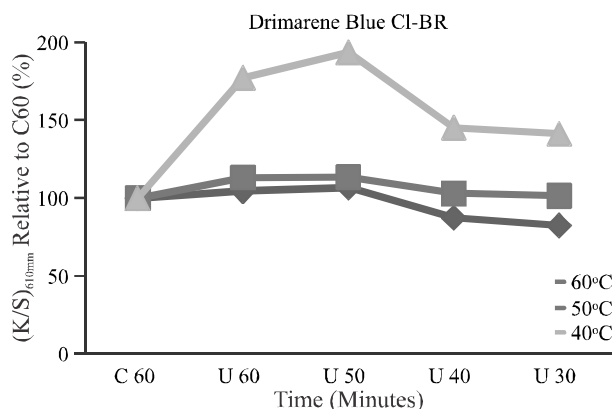


FIG. 4.  $(K/S)_{610nm}$  RELATIVE TO C60 IN %, DYED WITH DRIMARENE BLUE CL-BR 0.5% OWF

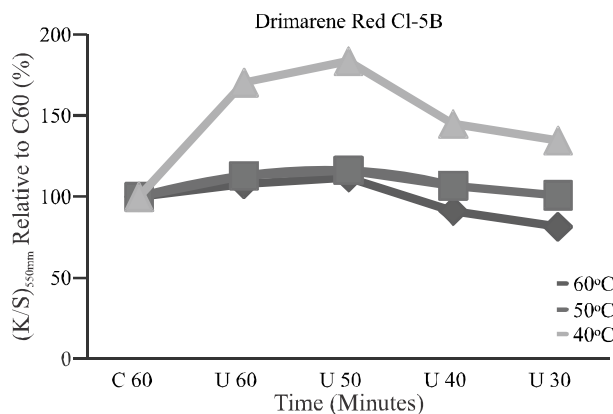


FIG. 5.  $(K/S)_{550nm}$  RELATIVE TO C60 IN %, DYED WITH DRIMARENE RED CL-5B 0.5% OWF

value of pre-treated and ultrasonic dyed sample is almost double to the conventional dyed sample. It is because the caustic soda swells up cellulosic fibers, hence allow the dye molecules to penetrate in to the fibre filaments. Similarly, ultrasonic cavitation assists the dye molecules to exhaust towards the fibre filaments. Thence, improves the  $(K/S)_{\lambda_{max}}$  values, decreasing nearly 40-50% dyeing cycle.

The dyeing at 60°C for 60 minutes using ultrasonic technique (U60) gives highest  $(K/S)_{\lambda_{max}}$  values in both Drimarene Red Cl-5B and Drimarene Blue Cl-BR (0.5% owf). The ultrasonic dyeing with caustic pre-treatment at recommended temperature and half dyeing time (30 minutes) gives colour strength value as with C60. Hence, organic cotton fabric can be dyed at reduce dyeing time at 60°C dyeing temperature. On the contrary causticization process is required an additional cost.

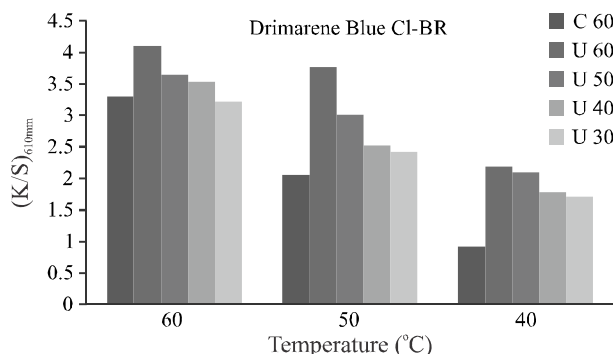


FIG. 6.  $(K/S)_{610nm}$  VALUES OF CAUSTICIZED AND THEN DRIMARENE BLUE CL-BR DYED SAMPLES

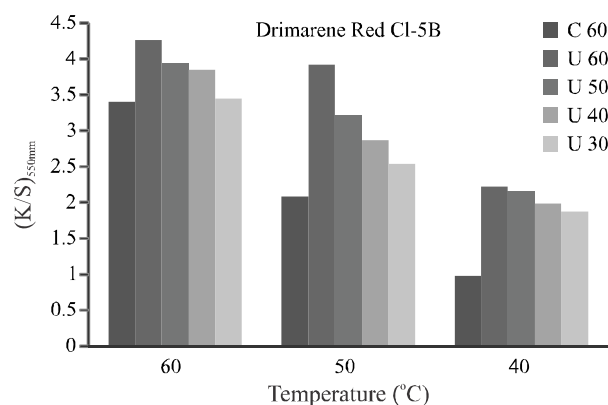


FIG. 7.  $(K/S)_{550nm}$  VALUES OF CAUSTICIZED AND DRIMARENE RED CL-5B DYED SAMPLES

Similar to Figs. 4-5, Figs. 8-9 shows the same trend of colour strength value. The pre-treated and then dyed sample by ultrasonic technique gives 130-240% high colour strength value than the sample dyed by conventional dyeing technique (C60) at all dyeing temperature and time for exhaustion and fixation phase.

### 4.3 Washing Fastness

Organic cotton fabric when dyed with reactive dyes shows inconsiderable change in shade and staining rating. Hence, fastness to washing is more depended on the dye type rather than dyeing methods. Reactive dye

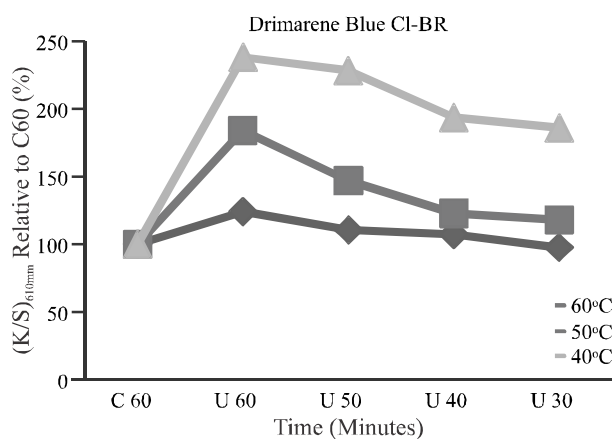


FIG. 8.  $(K/S)_{610nm}$  RELATIVE TO C60 IN %, CAUSTICIZED AND DYED WITH DRIMARENE BLUE CL-BR 0.5% OWF

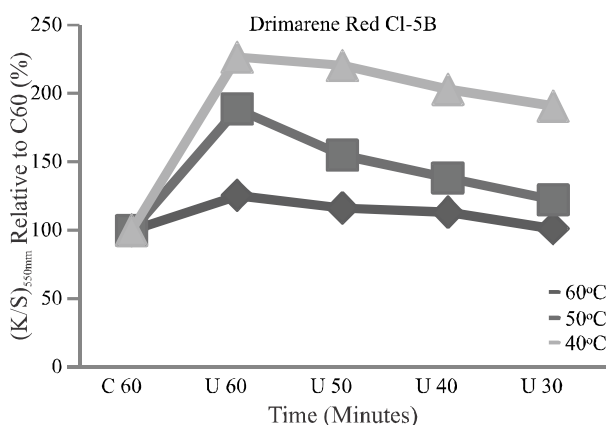


FIG. 9.  $(K/S)_{550nm}$  RELATIVE TO C60 IN %, CAUSTICIZED AND DYED WITH DRIMARENE RED CL-5B 0.5% OWF

molecules formed a strong covalent bond with cellulosic fabric and gives very good to excellent washing fastness properties. It is observed Drimarene Red Cl-5B gives the highest  $(K/S)_{\lambda_{max}}$  values and shows no staining and colour change when dyed by conventional and ultrasonic dyeing method at 60°C dyeing temperature. While fabric dyed with Drimarene Blue Cl-BR shows slight change in shade when dyed by conventional dyeing method (C60 at 60°C) and ultrasonic dyed at 60°C dyeing temperature for 30 minutes.

The staining rating (4-5) on cotton fabric is observed when Drimarene Blue Cl-BR and Drimarene Red Cl-5B is dyed at 50 and 40°C for 40 and 30 minutes dyeing cycle by ultrasonic technique. Pre-treatment using sodium hydroxide improves the washing fastness rating at 60°C dyeing temperature and 40 and 30 min dyeing time with both Drimarene Blue Cl-BR and Drimarene Red Cl-5B dye. However, staining rating (4-5) on cotton fabric has been observed when dyeing was performed at 50°C for 40 and 30 minutes using both dyes by ultrasonic dyeing method.

### 4.4 Dye Uniformity

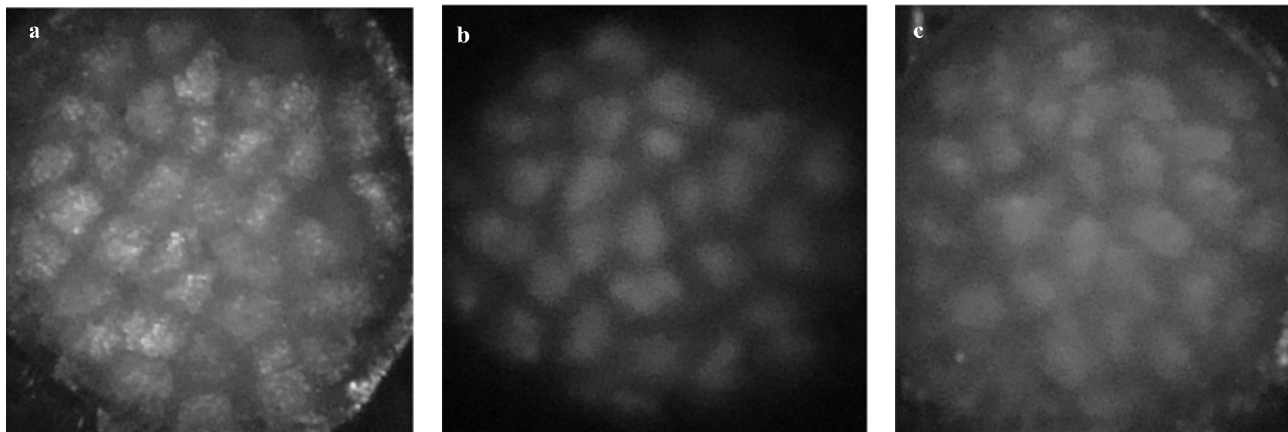
The uniformity of dye molecules in to the fibres filament of the yarns of the fabric was assessed using optical microscope. The images after capturing were assessed manually. It has been observed that the fabric sample dyed by conventional dyeing methods shows less diffusion of dye molecules both with Drimarene Blue Cl-BR and Drimarene Red Cl-5B as compared to ultrasonic dyeing technique as shown in Figs. 10-11.

In case of ultrasonic dyeing method, sample dyed at 60°C for 60 and 50 minutes shows much deeper diffusion of dye

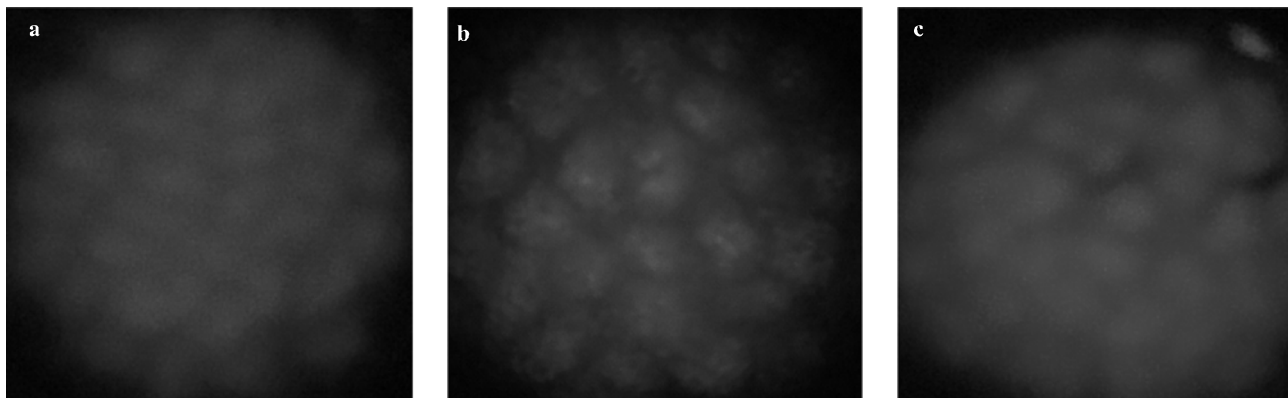


molecules, hence, high dye uptake values and dye uniformity. The fabric samples dyed after causticisation

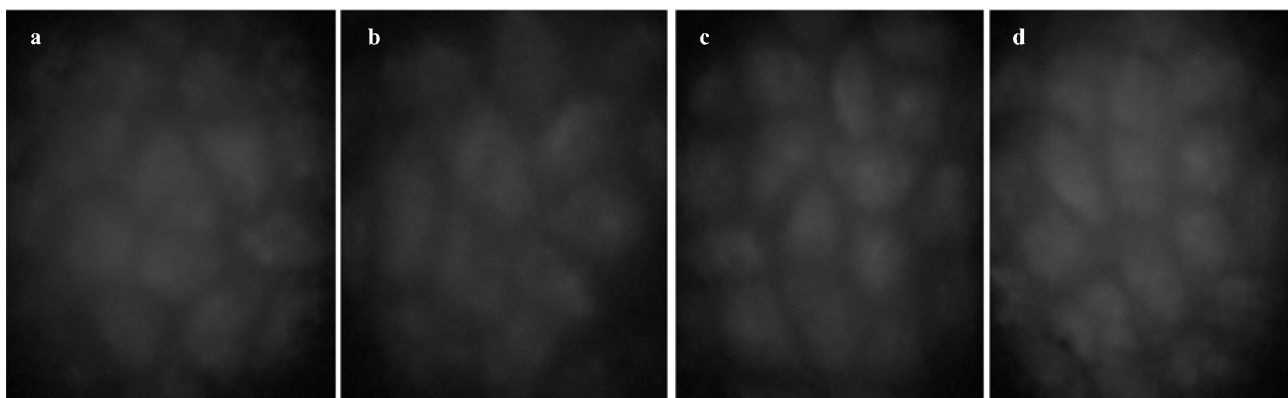
also shows uniform dyeing and deeper diffusion of dye molecules as shown in Fig. 12.



*FIG. 10(a) DYED SAMPLE WITH DRIMARENE BLUE CL-BR BY CONVENTIONAL DYEING METHOD AT 60°C FOR 60 MINUTES (b) DYED WITH DRIMARENE BLUE CL-BR BY ULTRASONIC TECHNIQUE AT 60°C FOR 60 MINUTES (c) DYED WITH DRIMARENE BLUE CL-BR BY ULTRASONIC TECHNIQUE AT 60°C FOR 50 MINUTES*



*FIG. 11(a). DYED WITH DRIMARENE RED CL-5B BY CONVENTIONAL DYEING METHOD AT 60°C FOR 60 MINUTES (b) DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 60 MINUTES (c) DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 50 MINUTES*



*FIG. 12(a)CAUSTICISED AND DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 60 MINUTES, (b) CAUSTICISED AND DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 50 MINUTES (c) CAUSTICISED AND DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 40 MINUTES (d) CAUSTICISED AND DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 30 MINUTES*

#### 4.5 Surface Effect

The SEM images of fabric sample dyed by conventional dyeing technique are shown in Fig. 13(a) gives lower  $(K/S)_{\lambda_{\max}}$  values and surface deterioration as compared to the sample dyed by ultrasonic technique as shown in Fig. 13(b). The sample dyed by ultrasonic technique at 60°C for 60 minutes shows that surface of sample is deteriorated by ultrasonic waves, making its fibrils protruding, hence gives lower  $(K/S)_{\lambda_{\max}}$  value as compared to sample dyed for 50 minutes. Less deterioration of fabric surface gives regular reflection of light hence high  $(K/S)_{\lambda_{\max}}$  values.

Pre-treated and dyed sample has an insignificant surface deterioration due to the combined action of causticization and ultrasonic waves during fabric processing. Few fibre fibrils are protruding on the fabric surface thus gives quite high colour strength values as shown in Fig. 14.

#### 5. CONCLUSION

Radcure using ultrasonic technique is an environmental friendly and sustainable dyeing method saves dyeing assistant, time and temperature. In this research bleached organic cotton fabric samples were dyed with ultrasonic and conventional dyeing method at varied

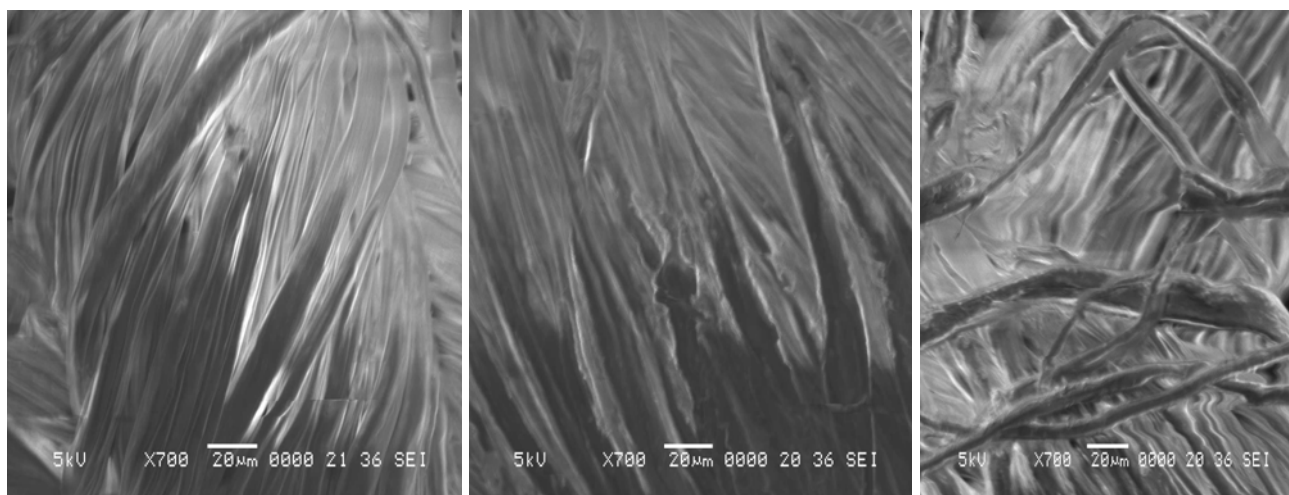


FIG. 13(a). DYED WITH DRIMARENE RED CL-5B BY CONVENTIONAL DYEING METHOD AT 60°C FOR 60 MINUTES (b) DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 60 MINUTES (c) DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 50 MINUTES

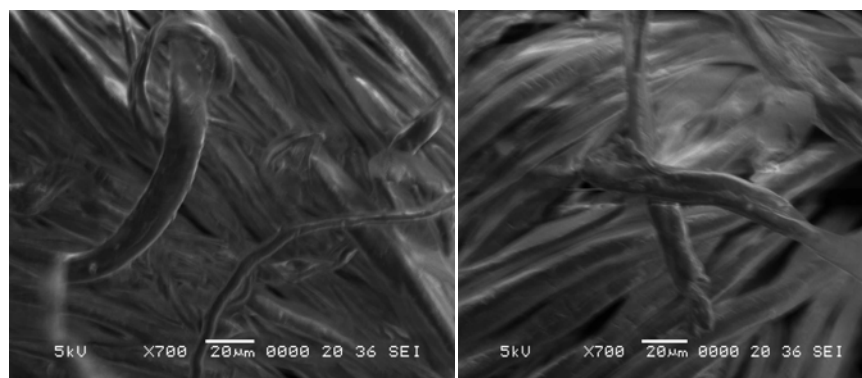


FIG. 14(a). CAUSTICISED AND DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 60 MINUTES (b). CAUSTICISED AND DYED WITH DRIMARENE RED CL-5B BY ULTRASONIC TECHNIQUE AT 60°C FOR 50 MINUTES



temperature and time. It is observed that the ultrasound waves improves 25% colour strength values with Drimarene Red CI-5B (0.5% owf) and 8% colour strength values with Drimarene Blue CI-BR (0.5% owf). Among temperature and time variations, sample dyed using ultrasonic technique at 60°C for 50 minutes gives highest  $(K/S)_{\lambda_{max}}$  values both Drimarene Red CI-5B and Drimarene Blue CL-BR dyes. It is because for 50 minutes dyeing cycle, less deterioration on fabric surface and deeper diffusion of dye molecules is observed as compared to dyeing for 60 minutes dyeing cycle at 60°C dyeing temperature. However, combine action of caustic and ultrasonic waves swell up the fibre filaments cause much deeper diffusion of dye molecules and highest colour strength value of organic cotton fabric. Furthermore, insignificant change in fabric surface is observed. The causticization and dyeing with ultrasonic technique almost half the dyeing time compared to conventional dyeing methods, hence save energy and cost.

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