Reactive Dyeing Of Organic Cotton Knitted Fabrics Using Ultrasound Technology

Semra Korkmaz, Gülay Özcan, Pelin Altay

***Abstract*— Ultrasonics holds a promise in applications in the field of textiles. Ultrasonic technology is used for enhancing productivity of pretreatment finishing and dyeing processes in textile industry. We have investigated the possibility of dyeing processes of organic cotton knitted fabrics in ultrasound bath.**

The conventionally bleached organic cotton single jersey and rib fabrics were dyed with reactive dyestuffs by cold pad-batch method. The fabrics were dyed with different class of reactive dyes using the conventional recipe in ultrasound bath. The ultrasonic dyeing processes were applied at three different temperature and time conditions. The aim of this study is to provide environmental improvements by reduced consumption of auxiliary chemicals and energy savings by dyeing at reduced processing times.

***Index Terms*— ultrasound technology, dyeing, reactive dyestuff, organic cotton**

I. INTRODUCTION

Organic cotton is a crop that is grown without the use of synthetic chemicals such as pesticides, herbicides and fertilizers using methods and materials that have a low impact on the environment [1,2]. Cotton was always cultivated organically, like all crops, until the early 20th century, but the demand for 'cosmetically perfect produce' and higher yields gave rise to increase the use of synthetic pesticides and fertilizers, and subsequently to genetically modified cotton [1]. Consequently, this led to the accreditation and certification of organic produce [3].

Reactive dyes are the most commonly used dyes for cotton materials and provide a complete color range and are easily applied [4,5]. Reactive dyes are also the most preferred dyes for organic cotton due to the presence of strong dye-fibre interaction. The parameters affecting reactive dyeing are dye chemistry, substantivity, reactivity, diffusion coefficient and solubility [4,6]. The another most important parameter affecting exhaustion and "fixation" of reactive dyes are temperature, salt concentration, alkali concentration, and liquid ratio. Dyeing conditions, especially the alkali requirement and temperature as well as the use of salt depend on the type of reactive group [5,7,8,9,10]. Reactive dyes are characterized by undesirable hydrolysis in solution, which decreases the dye fixation. An increase in dyeing temperature can improve the swelling of fiber to lead to high dye adsorption during the conventional dyeing process. However, the higher dyeing temperature can lead to hydrolysis of reactive group and reduce the covalent binding of dye molecule to the fiber. Cellulose gets attached with substitutable reactive dyes producing acid. Hydrolysis also occurs in water in the same way producing HCl. Reactive dyes can react with the hydroxyl groups of the water molecule to produce dye molecules with poor sustantivity for the fiber. Fastness of dyeing depends on the extent up to which fully or partially hydrolyzed dyes have been removed by a washing-off process. Ultrasonic energy improves the fastness properties as well as the productivity of dyeing process. The fastness improvement may be a result of better dye penetration and thus better covalent fixation with fiber. Therefore, use of ultrasound in reactive dyeing of cotton provides reduction in after-treatments for removing of hydrolyzed dye, resulting in energy savings and less water consumption [4,6,7,8,9,10,11].

Ultrasound is the science of sound waves above the limits of human audibility. The most common usage area of ultrasonic in industry is cleaning with help of cavitation. Ultrasonic arises as an alternative procedure for cleaning of contaminated machine parts, medical and electronical materials or materials which have small bulges and indents. The ultrasound in textile industry is a new method. It can be used for removing undesirable materials on textiles and improving effectiveness of enzyme molecules [12,13,14].

On the other hand, it has been discovered that ultrasonic energy can be used to enhance removing the stains on the fabrics and the ultrasonically treated fabrics have shown less tenacity loss than the conventional methods [15]. Therefore, the use of ultrasonic energy in wet finishing has potential in decreasing the amount of the process time, energy, chemicals used in and in improving product quality. Ultrasound energy can improve the dyeability of fabric. The effect of ultrasound in dye bath can be explained as dispersion effect, degassing and accelerating the rate of diffusion of the dye or finishing chemicals inside the fiber accelerating the interaction between dye bath and fiber. Less dye and other auxiliaries are needed to obtain the required colour and less effluent is produced. Reactive dyes needed a relatively large amount of salt for exhaustion. Reactive dyes have a low affinity for cotton; therefore, high salt (NaCl or Na_2SO_4) concentrations are added to the dyeing bath to reduce negative surface potential of cotton and to overcome potential barrier which exists in absorption of dyes from solution to the fiber surface [7,8]. Ultrasound can reduce the amount of salt and energy required by dyeing at lower temperatures and reduced process time compared to a conventional process thereby increasing industry competitiveness [16,17,18]. Textile wet processes

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assisted by ultrasound are of high interest for the textile industry for this reason [14,16,17,18,19].

Intensification of mass transfer in the inter-yarn and intra-yarn fabric pores is of great importance in improving the efficiency of wet textile processes. Intensifying mass transfer by conventional methods, such as operation at elevated temperatures, are not always feasible, due to undesired effects such as fabric damage. Usually ultrasound energy in dyeing process provides degassing-expulsion of dissolved or entrapped air entrapped in the inter-yarn and intra-yarn pores and dispersion-breaking up dye aggregate into uniform dyeing bath by ultrasonic cavatiation. Breaking up of micelles and high molecular weight aggregates into uniform dispersions and degassing in the dye bath enhance dye transport to the fiber surface to intensify the adsorption rate. Diffusion-ultrasound energy can penetrate the insulating layer covering the fibers and accelerate the rate of diffusion of dye molecular inside fibers and reaction between fibers and reactive dyes [5,17,19,20,21,22]. Ultrasound energy is also used as an alternative solution for environmental problems. Ultrasonic energy strengthens the adsorption of dyes on fabric, consequently decreasing the dye concentration in wastewater, thus reduce the pollution load. Ultrasound generates free radicals and these radicals attack on the contaminant molecules subsequently with the aim of either, completely mineralizing the contaminants or converting it into less harmful or lower chain compounds which can then be treated biologically [16,23,24,25].

Before the ultrasonic dyeing process, the organic cotton knitted fabrics are bleached conventionally by Net Knit Co.Ltd. The conventionally bleached organic cotton single jersey and rib fabrics are dyed with reactive dyestuff using the dyestuff recipe by cold pad-batch method. The same recipe in cold pad-batch method is used in ultrasonic dyeing of the same samples. The ultrasonic dyeing processes are applied at three different temperature and time conditions.

After the treatments the dyed samples are measured in Datacolour spectrophotometer for CIE Lab (L*,C*,h°) and K/S values. The UV-VIS spectrophotometer is used for measuring the absorbance of the ultrasonic dyeing solutions. In addition, the samples have been tested for rubbing, washing and water fastness properties using relevant ISO standards [33,34,35].

II. Materials and Method

100% organic cotton single jersey and rib structure knitted fabrics were used. The physical properties of samples are shown in Table 1. 100% organic cotton knit fabrics were supplied by Net Örme Co. Ltd and reactive dyes were supplied from Setaş Kimya Co. Ltd. The dyeing recipe recommended by Setaş Kimya for the reactive dyes used in the study is given in Table 2.

Table 1. Physical properties of fabrics were used

Fabric structure	Yarn count	Yarn twist, turns/m	Weight in unit area, g/m^2	Fabric density	Whiteness Index, WI	Absorbency	Rigidity, kgf/cm^2
Single jersey	Ne24/1	860	150	14	21,38	45 s	0,149974
Rib	Ne22/1	750	150	11	23,18	52 s	0,118663

Table 2. The recipe used in pad-batch dyeing method.

Setazol dyestuff	x g/l
Setawet R (wetting agent)	1-4 g/l
Urea	0-100 g/l
Alkali	y g/l
Temperature	20-25 °C
Pick-up	%60-80

This paper proposes a dyeing process at reduced batch time and less amount of chemicals for cotton organic fabric with reactive dyes. Dyeing processes were carried out in ultrasound bath without using urea. In the study, the soda-caustic method was used for the dye fixation. Firstly, 8 samples were dyed by conventional pad-batch method using the recipe given in Table 2. Organic cotton single jersey and rib fabrics were dyed with Yellow GR, Red RBN, Blue BB, Red 3BS reactive dyestuffs using US method. In order to evaluate the effect of the US to the impregnation capacity, the standard conventional pad-batch dyeing processes were applied in the ultrasonic bath by using the same recipe. Only time and temperature conditions were changed and process time was tried to be reduced. The recipe used for US dyeing is given in Table 3.

The pick-up ratio for cold pad batch process is 100% and fabrics were batched in polyethylene bags for the necessary time recommended for these dyestuffs in the recipe given by Setaş Kimya. The longest process time belongs to Blue BB dyestuff and it is 12 hours (Table 4).

Table 3. The recipe used in the study.

Setazol dyestuff amount	2,5 g/l
Setawet R (wetting agent)	1g/l
Soda	30g/l
Caustic soda	4ml/l
Temperature	20-25°C, in the ultrasonic bath 40-60°C
Pick-up	100%

Table 4. Batch times in polyethylene bags for each dyestuff.

Dyestuff	Time
Yellow GR	5 hours
Red RBN	8 hours
Blue BB	12 hours
Red 3BS	8 hours

Dyeing processes were carried out in US bath in three different dyeing conditions given in Table-5. In the ultrasonic bath, temperature increases with the process time. In the US-1 method, dyeing was started at 25°C and finished at 40 ^{0}C. It was observed that starting temperature was reached to 40 ^{0}C in 25 minutes. As a result, it was reported that temperature increases with 0,6°C/min. in the ultrasonic bath. In the US-2 method, cold water was circulated to the ultrasonic bath to hold the temperature constant in the ultrasonic bath. As the first method, the initial temperature was 25°C and cold water was circulated during 30 minutes to hold the temperature between 25-30°C. In the US-3 method, to obtain deeper colors the dyeing time was kept longer. For Yellow GR and Red 3BS dyestuffs, the initial temperature was 25° and the process time was 45 minutes. Using Blue BB and Red RBN dyestuff, the fabrics were dyed with increasing temperature. The starting temperature was 40°C and process time was 30 minutes (Table 5).

Table 5. Ultrasound methods

US method	**Dyeing conditions (temperature and time)**
US-1	Starting temperature: 25°C Final temperature: 40°C Time: 25min. (0,6°C/min. temperature increase)
US-2	Starting temperature: 25°C, by maintaining cold water, temperature is held constant at (27±1)°C. Time: 30 min.
US-3	1) Starting temperature: 25°C, time: 45 min. (0,6°C/min. temperature increase) 2) Starting temperature: 40°C, time: 30 min. (0,6°C/min. temperature increase)

As an after-treatment process, the same washing recipe was applied to single jersey and rib fabrics which were dyed using conventional pad-batch and US method. The washing temperatures recommended by Setaş Kimya are given in Table 6.

Table 6. Washing recipe after the dyeing processes.

Temperature	**Washing**
25°C	Cold washing
50°C	Rinsing with distilled water
95°C	Hot washing
95°C	Hot washing
95°C	Hot washing
80°C	Hot washing
40-50°C	Warm washing

In traditional textile industry, it is recommended always to neutralize in the first rinse when dyestuffs based on VS-groups are used. After dyeing process neutralization is generally done with acetic acid solution which has pH= 5-6. In this study, acetic acid was not used for neutralization, warm rinsing with distilled water was only applied. It was observed that warm rinsing with distilled water has been sufficient to achieve expected fastness properties.
After the treatments the samples were tested in Datacolor spectrophotometer for CIE Lab (L*,C*,h°) and K/S values. For each dyestuff, K/S values were measured at the wavelength that they have the lowest reflectance values (Table 7).

Table 7. The wavelengths used in the color measurements of the samples

dyestuff	**wavelength (λ)**
Blue BB	620
Red RBN	530
Yellow GR	430
Red 3BS	550

The UV-VIS spectrophotometer was used for measuring the absorbance of the ultrasonic dyeing solutions. The dye uptake of samples was assessed by the difference between the concentration of dye bath before and after dyeing process. In order to calculate the concentration of dyeing solutions after dyeing process, calibration curves for each dyestuff were prepared. A series of standard diluted dyeing solutions known concentrations for each dyestuff were prepared and measured using UV-visible spectrophotometer instrument to determine the unknown concentration of the samples. The experimentally determined absorbance values were plotted on a graph against the known concentration of each standard dyeing solution, thus calibration curves were obtained for each dyestuff.

After dyeing processes, absorbance values of dyeing bath samples were measured by using UV-visible spectrophotometer and the concentration of unknown dyeing bath samples were determined using the calibration curve. Wavelengths used in the absorbance measurements of the each dyestuff are shown in Table 8.

Table 8. The wavelengths used in the absorbance measurements of the dye bath samples

Blue BB	580 nm
Red RBN	520 nm
Yellow GR	440 nm
Red 3BS	520 nm

In addition, the dyed samples have been tested for rubbing, washing and water fastness properties using relevant ISO standards, respectively ISO 105 X-12, ISO 105 C-06 and ISO 105 E-01.

III. Results and Discussion

Absorbance values of dyeing solutions before and after US dyeing were measured in UV-vis spectrophotometer. In order to determine the dyeing efficiency, the dyeing solutions known concentrations were treated in the ultrasonic bath until the final temperatures of US1, US2, US3 methods (Table 9). By this way, the absorbance values of the solutions were measured by UV-VIS spectrophotometer. It is observed that the increase in the dyeing temperature causes increase in the absorbance values and the dyestuff concentrations in the dye bath. The change in the concentrations of the four types of dyestuff in the dye bath related to temperature is shown in Fig. 1.

Table 9. Process temperatures in US Methods

Method	Starting temperature (°C)	Final temperature (°C)
US 1	25	40
US 2	25	28
US 3	25	58

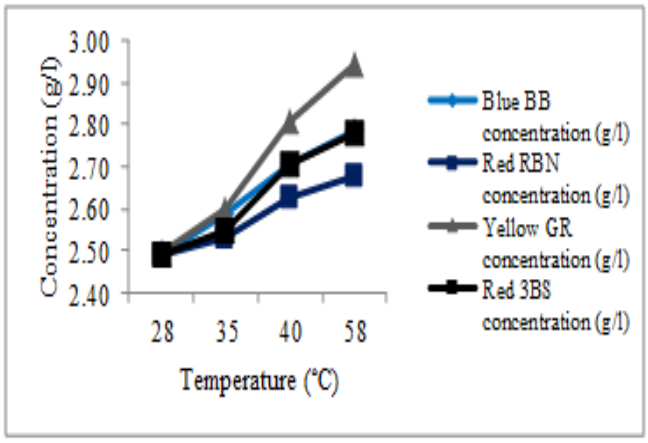

Fig. 1. Concentration changes according to temperatures

The absorbance values measured were substituted in the calibration equations and the dyestuff concentration in the dye bath after all dyeing methods (final concentration) were calculated (Table 10). When it is considered that, the initial concentration is 2,5g/l and there is a change in the dyestuff concentration, for each dyeing method and dyestuff, there is a decrease in the dyestuff concentrations after dyeing processes.

Table 10. Calibration equations for all dyestuffs

Dyestuff	Temp. (°C)	Calibration equations	Absorbance	Concentration (g/l)
Blue BB	28	1,960*abs	1,27	2,49
Blue BB	35	1,960*abs	1,32	2,59
Blue BB	40	1,960*abs	1,38	2,70
Blue BB	58	1,960*abs	1,42	2,78
Red RBN	28	1,346*abs	1,85	2,49
Red RBN	35	1,346*abs	1,88	2,53
Red RBN	40	1,346*abs	1,95	2,62
Red RBN	58	1,346*abs	1,99	2,68
Yellow GR	28	0,751*abs	3,32	2,49
Yellow GR	35	0,751*abs	3,46	2,60
Yellow GR	40	0,751*abs	3,74	2,81
Yellow GR	58	0,751*abs	3,92	2,94
Red 3BS	28	1,447*abs	1,72	2,49
Red 3BS	35	1,447*abs	1,76	2,55
Red 3BS	40	1,447*abs	1,87	2,71
Red 3BS	58	1,447*abs	1,92	2,78

Blue BB, Red RBN and Yellow GR are dyestuffs for cold-dyeing methods, while Red 3BS is suitable for exhaust dyeing at high temperatures. With related to these characterictics of the dyestuffs, the results of US dyeing with cold-pad batch dyestuffs show that in the low temperatures there is a decrease in the dyestuff concentrations. Because, in the dyeing processes with cold pad-batch dyestuff using US 2 (final temperature is 28°C) and US 1 (final temperature is 40°C) methods, the change in concentrations are higher. It is observed that there is a lower concentration change in US 3 (final temperature is 58°C) compared to US 1 and US 2 methods.

The CIE Lab (L*,C*,h°) values of the fabrics dyed with conventional cold-pad batch and US methods were measured with Datacolor 600™ spectrophotometer. According to Fig.

2, for Yellow GR dyestuff, similar results are obtained in both pad-batch and US methods. For Red RBN dyestuff, pad-batch method gives better results while other three US methods give similar results. In Red 3BS dyestuff, results closer to conventional pad-batch method are obtained in US 2 method. For Blue BB method, results closer to cold pad-batch method are obtained in both US 1 and US 2 methods.

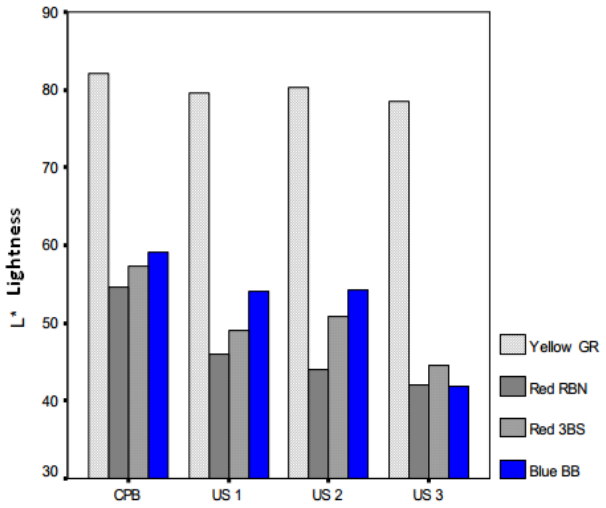

Fig. 2. Changes of L* values according to dyeing methods (CPB: cold pad-batch method).

For each dyestuff type, L* values are the lowest in US 3 method. In temperatures higher than 40°C, deeper colors are obtained. Another result obtained from this figure is that, as temperature increases, L* values decrease for each dyestuff.

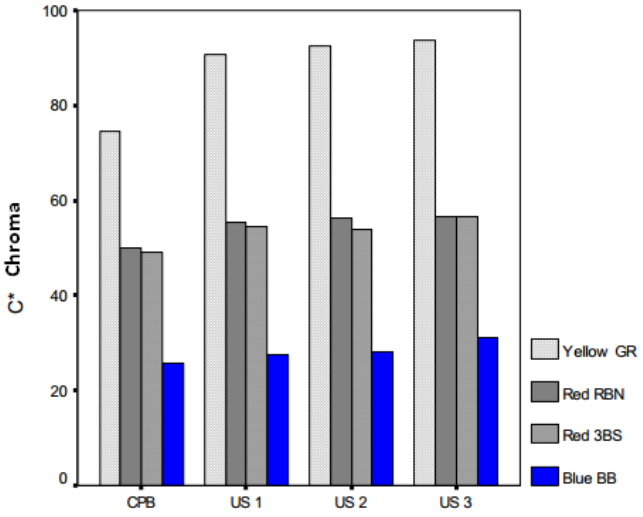

Fig. 3. Changes of C* values according to dyeing methods (CPB: cold pad-batch method).

The relation between dyeing parameters and chroma values (C*) which defines the chromacity of a certain deep color. When the effect of dyeing method-dyestuff interaction to chroma (C*) values are examined, it is seen that the conventional cold pad-batch and US methods give closer results. In addition, when three US methods are compared, it is seen that they give closer results for C* values (Fig. 3). As a result, batching time can be ignored to get better C* values using US method.

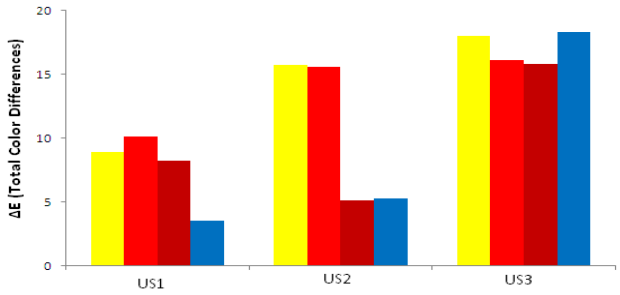

Fig. 4. Changes of ΔE values of samples according to US methods. (US:Ultrasound)

The results of total color differences between the samples dyed by US methods (US1, US2, US3) and cold-pad-batch method are shown in Fig. 4. The total color differences for US 3 method has been found to be higher than that for US 1 and US 2.

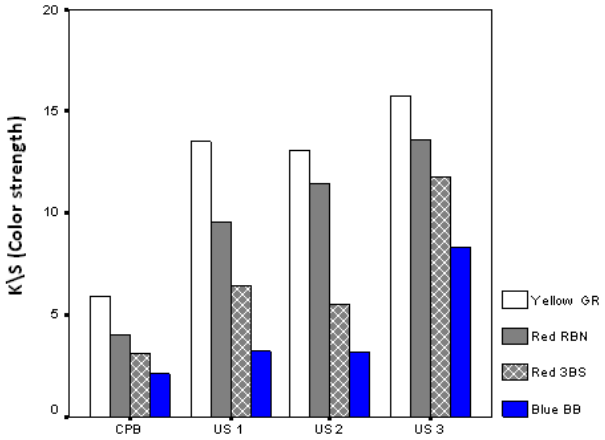

Fig. 5. Changes of K/S values according to dyeing methods (CPB: Cold pad-batch method; US: Ultrasound method).

The correlation analysis showed that there is a positive relation between dyeing methods and K/S (color strength) values. When the K/S values of single jersey and rib fabrics are examined which are dyed with conventional pad-batch method and with US methods using the same recipe, it is seen that the K/S values of the two types of the fabrics are closer in all methods.

Another result from that figure is that the K/S values obtained from the all US methods are better than the conventional pad-batch method. When US methods are compared, it is seen that starting from 25°C and dyeing with increasing temperature for 20 minutes process time (US1 method) give closer K/S results with the method that cold water was maintained over the dyeing bath to hold temperature constant in 25-30°C range. The relationship between dyeing method and K/S values of the samples are shown for each dyestuff in Fig. 5.

After the conventional and US dyeing methods, dry and wet rubbing fastness tests were applied to the samples using ISO 105 X-12 standard. The obtained dry fastness values showed that the US dyeing method results were very similar with conventional pad-batch method for all type dyestuffs. As a result, US method has no negative effect on the dry fastness values.

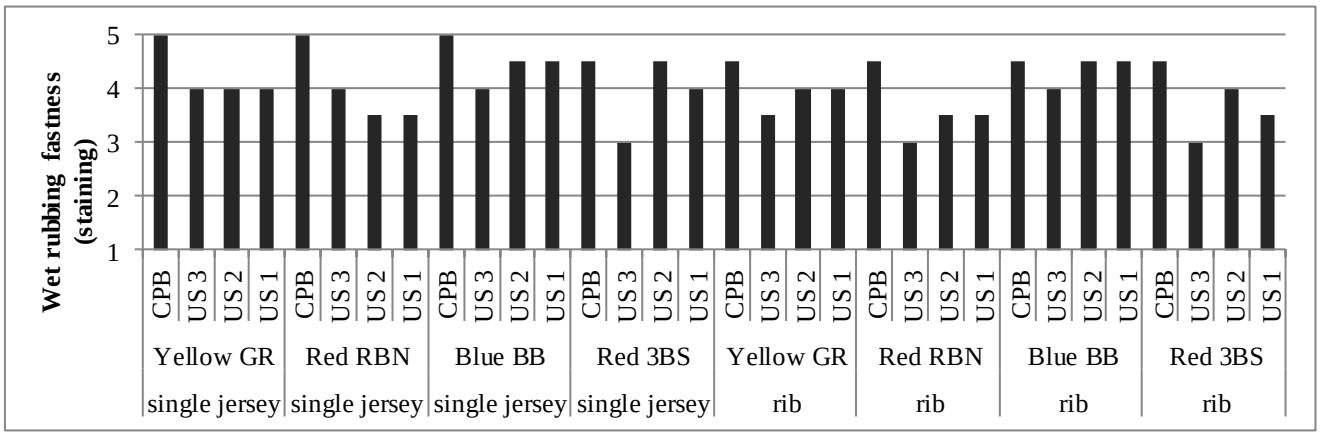

Fig. 6. The wet rubbing fastness (staining) results

It is easily seen from Fig. 6, wet rubbing fastness values change from conventional dyeing method to US methods but it can be obtained tolerable values according to US method. When fabric types are considered, higher wet rubbing fastness values are obtained in single jersey fabrics. While better wet fastness values are obtained in conventional pad-batch method, in US methods for all dyestuff the wet fastness values are better in US1 and US2 methods.

The washing fastness values of the dyed samples are tested with ISO 105 C-06 standard. Color fastness values are shown in Fig. 7. For all dyestuff when cotton staining fastness values are considered, color fastness values are quietly good in US methods except Yellow GR dyestuff. The washing fastness (color change) values are closer in both conventional pad-batch and US methods.

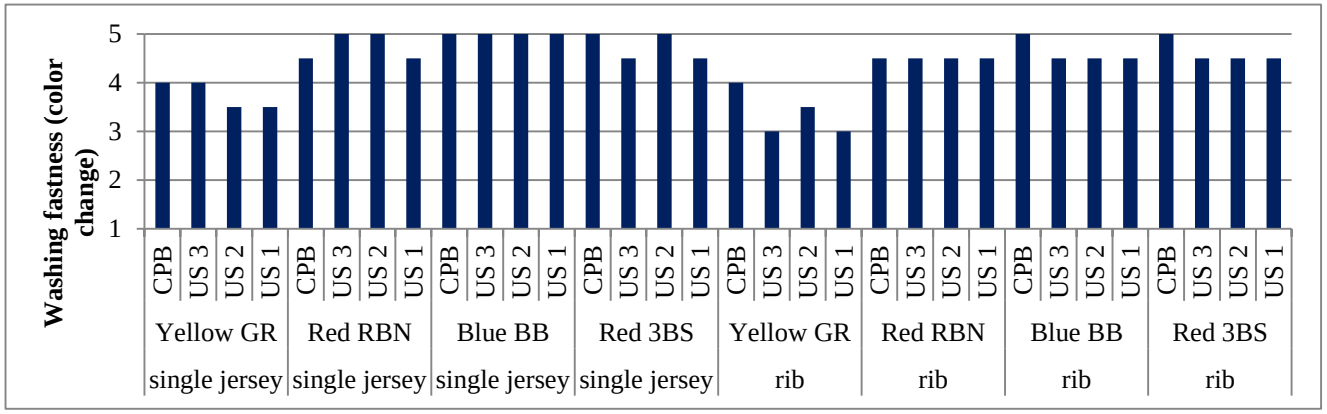

Fig. 7. Washing fastness (color change) results

The water fastness values of the dyed samples are tested with ISO 105 E-01 standard. In both conventional pad-batch and US methods, closer water fastness (color change) values are obtained. This shows that US method has no negative effect on the water fastness color change values. In addition, Fig. 8 shows the change of water fastness (color change) values according to dyestuff type and dyeing methods. For the both dyeing methods the water fastness values are quite good with rating gray scale 4-5.

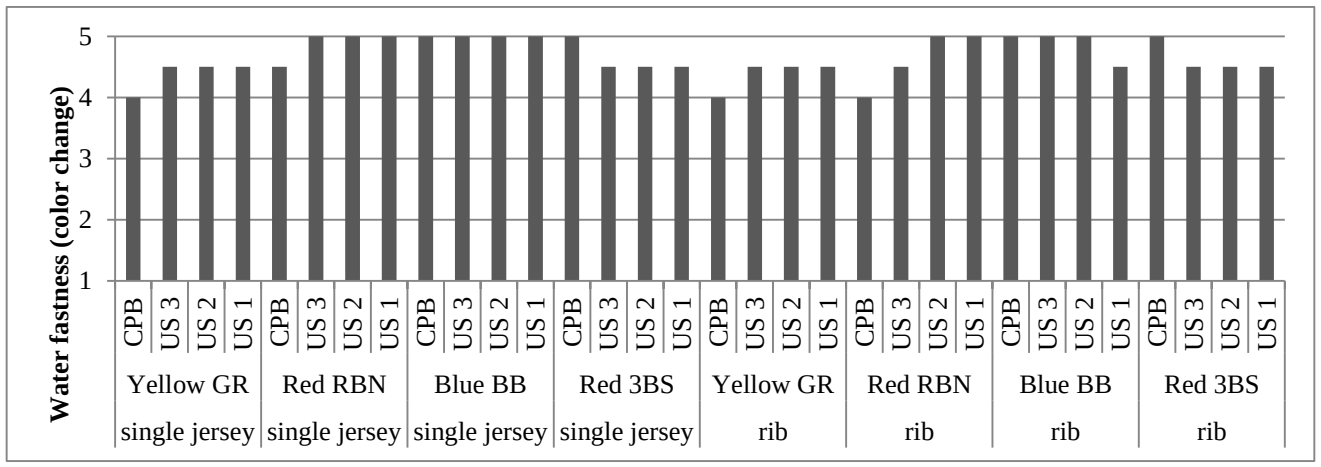

Fig. 8. Water fastness (color change) results

IV. CONCLUSIONS

Reactive dyes are one of the most commonly used application class of dyes for cotton materials. In reactive dyeing process, urea is used to increase the solubility of dye in reaction medium, control the evaporation of water during drying and swelling of cotton, thereby facilitating the dye-fiber reaction. However, the use of urea poses ecological problems associated with the high nitrogen content of the dyeing effluent. In addition, in traditional textile industry acetic acid solution is generally used for neutralization after reactive dyeing process. The aim of this study is to provide environmental improvements by reduced consumption of auxiliary chemicals and energy savings by dyeing at reduced processing times.

In this study, dyeing of cotton fabric with reactive dyestuffs was carried out without using urea in ultrasound bath changing the dyeing temperatures and process times. After dyeing processes, CIE L*a*b* (L*,C*,h°) and K/S values of the dyed samples were measured in Datacolor Spectrophotometer. The UV-VIS spectrophotometer was used for measuring the absorbance of the ultrasonic dyeing solutions. After US dyeing, acetic acid solution was not used for neutralization, warm rinsing with distilled water was only applied to the dyed samples. Additionally, the samples have been tested for rubbing, washing and water fastness properties using relevant ISO standards.

The results show that deepness of color hue (K/S value on λmax) is higher in US dyeing than in a conventional one at reduced process times. It is observed that warm rinsing has been sufficient to achieve expected fastness properties. The dry rubbing fastness values were very close in conventional and US dyeing. Wet rubbing fastness values change from conventional dyeing method to US methods but it can be obtained tolerable values according to US method. The washing and water fastness properties were not affected by US method.

In conclusion, US method can be an alternative method to conventional cold pad-batch method in dyeing processes. The ultrasonic energy show the potential in decreasing the amount of the dyeing time, energy, chemicals used and improving product quality.

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