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EFFECT OF BATCHING AND MICROWAVE HEATING TIME ON COLOR PROPERTIES OF COTTON FABRIC DYED WITH REACTIVE DYES ON LABORATORY SCALE

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Abstract: The fixation using a batching technique on a Cold-Pad-Batch (CPB) reactive dyeing process at room temperature is typically carried out for several hours to get a good color strength on the cotton fabric. The lengthy batching time becomes a problem when undertaking the color matching process because the operator must wait long enough to get the dyeing results evaluated before the production process. In this work, we tried to shorten the batching time by adding a microwave heating step after the batching process (Cold-Pad-Batch-Microwave) and then compared the dyeing results in terms of color strength (K/S) and color difference (ΔE) between both methods. Experiment results revealed that the Cold-Pad-Batch-Microwave dyeing method can be used to shorten the dyeing time to get similar color strength value as opposed to the conventional Cold-Pad-Batch dyeing. The batching time of 1 hour and the microwave heating time 25 minutes are the recommenced condition in the Cold-Pad-Batch-Microwave dyeing method which has comparable K/S values of the conventional CPB method and has good ΔE value.

Keywords: Cold-Pad-Batch dyeing, reactive dyes, color matching, microwave heating

1. INTRODUCTION

Reactive dyestuffs are known for their excellent colorfastness properties due to the covalent bonding between the cellulose fiber and reactive dye. The reactive dye becomes part of the fibers and relatively withstand washing in boiling water in neutral conditions [1].

Since the first discovery of reactive dyes by Rattee and Stephen in 1954, there have been numerous new types of reactive dyes synthesized to date [2]. Reactive dye is a dyestuff predominantly used to dye cellulosic fiber materials in alkaline conditions. However, discoveries in reactive dyes enable us to use some of them for dyeing protein and polyamide fibers in weakly acid conditions [3–5].

Several approaches to dye cellulose fabrics use reactive dyes, namely exhaust, semi-continuous and continuous processes. In the semi-continuous process, the Cold-Pad-Batch method is the most important one because the process can save dyeing costs compared to the other methods [6][7]. The pad batch dyeing machine can save energy usage by up to 80% and water consumption by up to 90% and also reduce the use of dye and salt [8].

In the Cold Pad-Batch method (CPB), the cotton fabric is dipped in a padding liquor containing reactive dyes and then padded through a padder mangle. The next step is batching process, where the padded fabric is batched up to a roll. The roll is covered with a plastic sheet and rotated during the batching process to prevent uneven dyeing. The fixation step using batching technique takes several hours, typically 16-24 hours, to allow dye diffusion [4] and achieve a good color strength result. So, the downside of the CPB method is that it takes a long time to finish the dyeing process.

Dyeing experiments on a laboratory scale (color matching) are essential to be carried out to determine the dyeing recipe that can be used for the dyeing process on a large scale. Color matching in the laboratory is required to meet the color quality following consumer demand. However, if one follows the conventional CPB process, it will take a long time to formulate the recipe for the production process. From this background, there is a need to find ways to shorten the color matching time, one of which is by adding a microwave heating process after a shorter batching process. This work aims at finding the effect of batching time and microwave heating time on color strength (K/S) and color difference (◎E) value. For the first study, it was limited to using only one color of reactive dye.

2. MATERIALS AND METHODS

Ready-to-dye plain-woven cotton fabric was used in this work. There were two different methods of dyeing process i.e., Cold-Pad-Batch and Cold-Pad-Batch Microwave. The padding solution contained reactive dye Remazol Red RGB, Cibaflow PAD (wetting agent), sodium silicate and NaOH (alkali), and urea (humectant).

The Cold-Pad-Batch dyeing process was carried out by impregnating the cotton fabric in a padding solution followed by padding it in a laboratory padding machine, then wrapping it with a plastic sheet and batching it for 12 hours. The Cold-Pad-Batch-Microwave method was done similarly to the Cold-Pad-Batch, but the batching time was shorter (0.5 – 1.5 hours) and followed by heating in a microwave oven (Panasonic NE-1054F) having 1000 W power. After the fixation process (batching and/or microwave heating), the samples were subject to washing in warm water, soaping in a solution containing Sunmorl FL CONC, and then rinsing with cold water.

The recipes and condition used in the experiments are shown below:

a. Padding solution:
- Remazol Red RGB : 3 g/L
- Cibaflow PAD : 1 g/L
- Sodium Silicate 58°Be : 75 g/L
- NaOH 38°Be : 3.3 g/L
- Urea : 40 g/L
- WPU : 70%

b. Batching time : 0.5; 1; 1.5 hour(s)

c. Heating time in microwave oven : 10; 15; 20; 25; 30 minutes

d. Power level of microwave oven : High

e. Soaping:
- Sunmorl FL CONC : 0,5 g/L
- Temperature : 80◎C
- Time : 15 minutes

Finally, color strength (expressed as K/S) and color difference (◎E) of the dyed fabrics were evaluated using Konica Minolta CM 3600d Spectrophotometer with illuminant D65 and a 10° standard observer. The K/S values of the samples were determined using Kubelka-Munk Equation:

$$K/S= 1-R22R \quad (1)$$

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where K is absorption coefficient, S is scattering coefficient, and R is reflectance value at ⚜max. The color differences (◎E) were determined using the CIE L*a*b* 1976 equation:

$$\Delta E= \Delta L*2+\Delta a*2+\Delta b*2 \qquad (2)$$

where L* is lightness, a* is reddish, and b* is yellowish.

The color difference (◎E) was examined by comparing the sample dyed by the Cold-Pad-Batch-Microwave method to the one dyed only by the Cold-Pad-Batch method. The smaller the value of ◎E, the more similar both colors are.

3. RESULTS AND DISCUSSION

1. Color Strength (K/S)

The color strength value (K/S) of the material indicates the amount of dye absorbed into the fibers. The more dyes that are absorbed in the fabric, the greater the value of color aging (K/S) and the darker the color that appears on the fabric.

The result of the color strength (K/S) measurements of dyed samples using the Cold-Pad-Batch method with a batching time of 12 hours was 3.17; while the K/S measurement results of the samples dyed using the Cold-Pad-Batch-Microwave method are shown in Table 1 and Figure 1. It can be seen from Table 1 and Figure 1 that the longer the batching time, the higher the color strength value (K/S). Increasing the heating time in a microwave oven causes an increase in color strength. This occurs in all variations of batching time (0.5; 1.5 and 1.5 hours). Thus, a longer time in microwave heating and batching can cause more reactive dyes to be fixed into the fiber.

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Table 1. Color strength (K/S) of the dyed samples.

Batching time (hours)	Microwave Heating time (minutes)	K/S (✾ 540 nm)	
		Mean	**SD**
0.5	10	2.19	0.05
	15	2.55	0.03
	20	2.75	0.03
	25	3.02	0.04
	30	2.99	0.05
1	10	2.87	0.03
	15	3.14	0.07
	20	3.27	0.04
	25	3.24	0.07
	30	3.19	0.03
1.5	10	3.32	0.03
	15	3.58	0.03
	20	3.53	0.05
	25	3.48	0.05
	30	3.47	0.06

According to El Molla et al.[9], microwave radiation penetrates more deeply into the particles of the dyes causing uniform heating of all particles at the same time with less heat transmission issues. Those characteristics of microwave energy could lead to more reactive dyes being fixed into cotton fibers during the dyeing process and give higher K/S value.

From Figure 1, K/S increases with increasing heating time in the microwave, but between 25 and 30 minutes the K/S value tends looks constant. This could be at high temperatures and a longer time the hydrolysis of reactive dyes was greater.

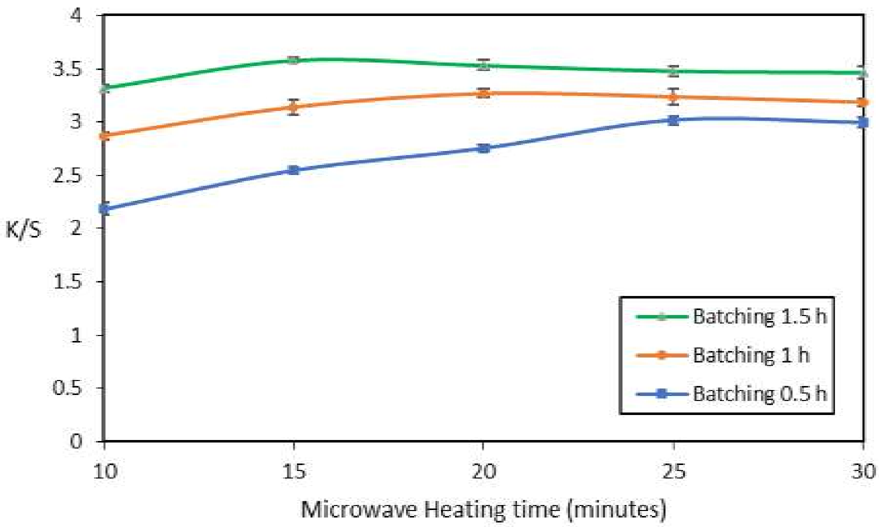

Figure 1. Relationship between variations in batching time and microwave heating time on K/S value.

A two-way ANOVA using Jamovi statistical software was performed to analyze the effect of batching time and microwave heating on color strength (K/S). The p-value of both batching time and microwave heating time on color strength (K/S) was 0.001 indicating that batching time and microwave heating time each influences color strength. The two-way ANOVA also revealed that there was a statistically significant interaction between batching time and microwave heating on color strength ($F (8, 60) = 59.7$, $p<.001$).

A Post Hoc Test was carried out by comparing the Tukey's p-value among the batching time 0.5, 1.0, and 1.5 hours and it showed that there was a statistically significant difference among all combinations of batching time with $p<.001$. Hence, the batching time has impact on the color strength (K/S) of the fabric samples. Microwave heating time did also have a statistically significant effect on color strength ($p<.001$). The Tukey's p-value data for microwave heating time of 20 minutes compared to 30 minutes is 0.392 implying both groups had statistically the same means; the 25 minutes heating time group compared to the 30 minutes has a p-value of 0.428 which also imply that those groups had statistically the same means. However, the 20 minutes

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heating time group compared to the 25 minutes gives a p-value of 0.007 which is statistically different.

Figure 2 presents estimated marginal means graph of batching time and microwave heating time on K/S value. Figure 2a shows that K/S increases with increasing batching time. Figure 2b shows that 25 minutes microwave heating gives the maximum K/S value. These results show that 25 minutes heating in a microwave oven in the Cold-Pad-Batch-Microwave method is sufficient to obtain comparable color strength with the one from conventional Cold-Pad-Batch method.

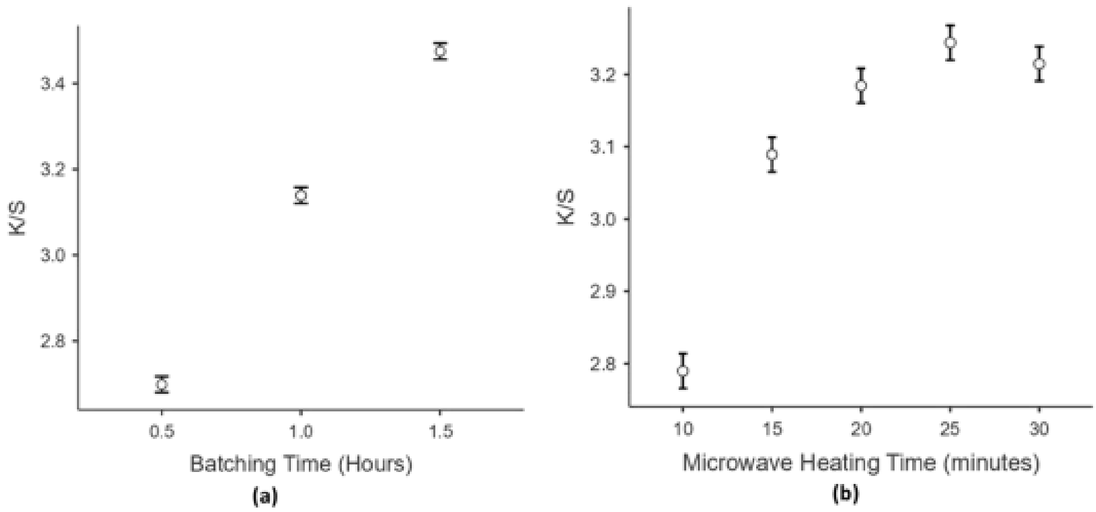

Figure 2. Estimated marginal means graphs of: (a) batching time and (b) microwave heating time on K/S value.

2. Color difference (◎E)

The color difference (◎E) data of the samples dyed using Cold-Pad-Batch-Microwave method can be seen in Table 2 and Figure 3. In this study, the ◎E value of the fabrics dyed by Cold-Pad-Batch-Microwave dyeing method in different batching and microwave heating time were compared to the fabric dyed using conventional Cold-Pad-Batch dyeing method.

Table 2. Color difference (◎E) of the sample dyed using Cold-Pad-Batch-Microwave method compared to the samples dyed using Cold-Pad-Batch method.

Batching time (hours)	Microwave Heating time (minutes)	⚜E	PASS/FAIL
0.5	10	2,13	FAIL
	15	1,84	FAIL
	20	1,62	FAIL
	25	1,16	FAIL
	30	1,23	FAIL
1	10	0,95	PASS
	15	0,32	PASS
	20	0,53	PASS
	25	0,78	PASS
	30	1,12	FAIL
1.5	10	0,75	PASS
	15	1,35	FAIL
	20	1,21	FAIL
	25	1,03	FAIL
	30	0,96	PASS

Note: PASS is ◎E < 1; FAIL is ◎E > 1

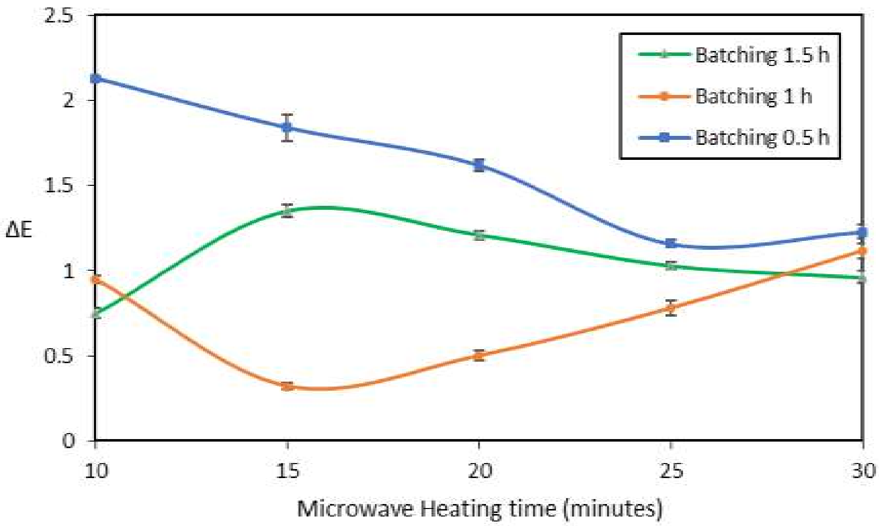

Figure 3. Relationship between batching time and microwave heating time on ◎E value.

In textile companies, color difference (◎E) evaluation is vital to determine whether the dyed fabric is acceptable or not before it goes to the customer. The general standard for determining that the color difference of the tested sample is acceptable (Pass) is less than 1 (◎E<1). The value of ◎E<1 means that the color of the test sample and the standard fabrics are in a very close position in the CIELab coordinate system (not perceptible to human eyes) and can be considered as "Pass", but if the color difference has a value of more than 1, it means that both the test sample and the standard fabrics are considered having different color and will be termed as "Fail".

From the experimental results, all samples dyed with a batching time of 0.5 hours (at all heating times in the microwave oven) produced a ◎E value of more than 1 and some even reached a ◎E close to 2 which indicates that the color characteristics are very far from the color of the reference fabric. So, all those samples are categorized as "Fail". Comparing the K/S values of the samples with the reference fabric, it is obvious that the values are all lower than the reference color. The low K/S value was due to the very short batching time of 0.5 hour, so the amount of fixed dye in the fabric was very low. Fabric samples that were dyed with a batching time of 1.5 hours and heating in a microwave oven for 10 and 30 minutes gave "Pass" results (◎E<1), while samples heated for 15, 20 and 25 minutes were categorized as "Fail" because they had ◎E>1. For samples dyed with a batching time of 1 hour, most of the measured color difference values are less than 1 (except for the 30 minutes microwave heating time with ◎E>1). Those results indicate that the suitable batching time for the Cold-Pad-Batch-Microwave time is 1 hour.

To analyze the effect of batching time and microwave heating on color difference (◎E), a two-way ANOVA was also performed. The statistical test showed that batching time and microwave heating time each influences color difference with p-value less than 0.001. The two-way ANOVA also disclosed that there was a statistically significant interaction between batching time and microwave heating on color difference ($F(8, 60) = 544$, $p<.001$).

A Post Hoc Test for analyzing the effect of microwave heating time on color difference (◎E) showed that there was statistically significant difference for most combinations of batching time groups with $p<.001$, except for the combination between the 20- and 30-minutes microwave heating time with a p-value of 0.568 showing that both microwave heating times have statistically the same means. Again, those results showed that the batching time also influences the color difference of the fabric samples. Post-Hoc Test for the batching time groups showed p-values of all group combinations are less than 0.001, meaning that there was statistically significant difference among all groups.

Estimated marginal means graphs in Figure 4 also confirm that the suitable batching time for the Cold-Pad-Batch-Microwave is 1 hour and the microwave heating time is 25 minutes because these conditions give the smallest estimated marginal means of ◎E.

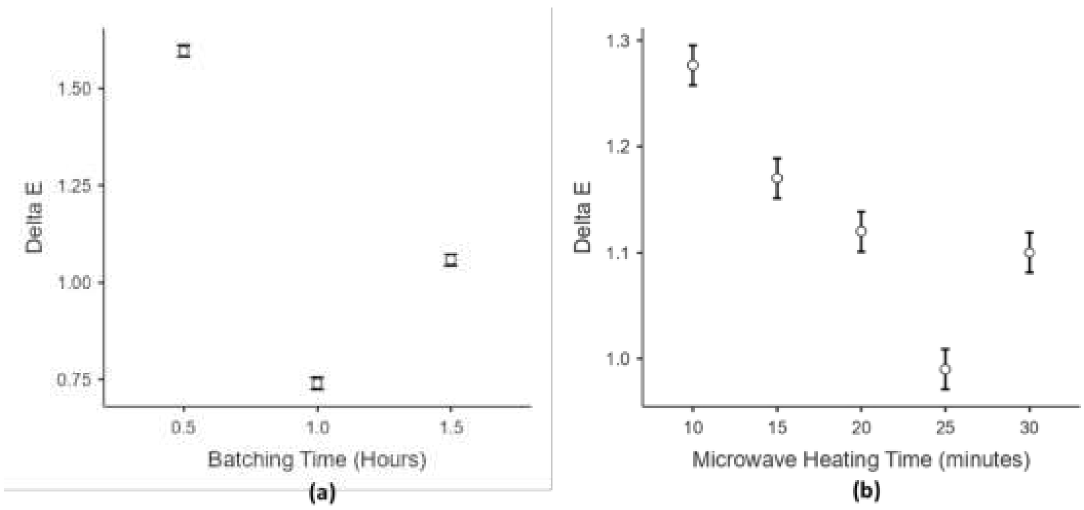

Figure 4. Estimated marginal means graphs of: (a) batching time and (b) microwave heating time on ◎E.

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4. CONCLUSIONS

This study was conducted to compare the dyeing results of the conventional Cold-Pad-Batch and Cold-Pad-Batch-Microwave methods on cotton fabric by analyzing color strength (K/S) and color difference (◎E) value. The conclusions that can be drawn from this study are as follows:

- Batching time and microwave heating time has a significant effect on color strength (K/S) and color difference (ΔE).
- The suitable batching time for the Cold-Pad-Batch-Microwave time is 1 hour and the microwave heating time 25 minutes which is comparable with the reference sample dyed with CPB (batching time of 12 hours).
- Microwave heating can be used to shorten the batching time in the Cold-Pad-Batch-Microwave dyeing method to get similar color strength value as opposed to the conventional Cold-Pad-Batch dyeing.

The results presented in this study used a single color, so further research is still needed using a combination of several colors because most fabrics are dyed using a mixture of several colors in the production process.

Author Contributions: H.H. and T.S. conceived and designed the experiments; H.H., N.C. and T.S. wrote the paper and analyzed the data.

Conflicts of Interest: The authors declare no conflict of interest.

REFERENCES

1. S. M. Shang, "Process control in dyeing of textiles," in *Process Control in Textile Manufacturing*, A. Majumdar, A. Das, R. Alagirusamy, and V. K. Kothari, Eds. Philadelphia: Woodhead Publishing Ltd., 2013.
2. "Reactive dye," *Cameo*. [Online]. Available: https://cameo.mfa.org/wiki/Reactive_dye. [Accessed: 15-May-2022].
3. D. P. Chattopadhyay, "Chemistry of dyeing," in *Handbook of Textile and Industrial Dyeing. Volume 1: Principles, processes and types of dyes*, M. Clark, Ed. Philadelphia: Woodhead Publishing Ltd., 2011, pp. 150–183.
4. Roger H. Wardman, *An Introduction to Textile Coloration: Principles and Practice*. Hoboken, NJ: John Wiley & Sons, 2018.
5. A. K. R. Choudhury, "Dyeing of synthetic fibers," in *Handbook of Textile and Industrial Dyeing. Volume 2: Application of Dyes*, M. Clark, Ed. Philadelphia: Woodhead Publishing Ltd., 2011, pp. 40–128.
6. K. Hunger, Ed., *Industrial Dyeing: Chemistry, Properties, Applications*. Weinheim: WILEY-VCH, 2003.
7. Z. Khatri, M. H. Memon, A. Khatri, and A. Tanwari, "Cold Pad-Batch dyeing method for cotton fabric dyeing with reactive dyes using ultrasonic energy," *Ultrason. Sonochem.*, vol. 18, no. 6, pp. 1301–1307, 2011.
8. S. R. Shukla, "Pollution abatement and waste minimisation in textile dyeing," in *Environmental Aspect of Textile Dyeing*, R. M. Christie, Ed. Woodhead Publishing Limited, 2007, pp. 116–148.
9. M. M. El-Molla, K. Haggag, and Z. M. Mahmoued, "Cold Pad-Batch Dyeing Method for Dyeing Cotton Fabric with Reactive Dye Using Microwave Irradiation Technique," *Int. J. Sci. Res.*, vol. 4, no. 4, pp. 808–815, 2015.