MICROWAVE-ASSISTED ENZYMATIC TREATMENTS OF COTTON KNITS

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ABSTRACT

Microwave energy is a fast, uniform and effective method for pre-treatments of textile materials. Using pectinase, cellulase, laccase, etc. enzymes for treatments of cotton textile material before colouration have many advantages over conventional methods such as reducing the chemical damage of fibers, short process time, energy and water savings. Recently, microwave-assisted enzymatic treatment is a sustainable cleaner production technique for all natural fibers. Microwave-assisted dyeing and finishing methods can be applied successfully in the literature. There are limited works about microwave-assisted enzymatic treatment of cotton. In this work, the effect of microwave-assisted treatments with NaOH, pectinase, cellulase, laccase and combination of pectinase/cellulase, pectinase/laccase, pectinase/cellulase/laccase, pectinase/laccase/cellulase enzymes on conventional reactive dyeability of cotton fabric. Hydrophilicity, contact angle, whiteness/yellowness indexes of conventionally and microwave-assisted treated samples were measured and compared with conventionally treated samples with sodium hydroxide. Whiteness and yellowness results were adequate for dark-coloured samples. The CIELab colour coordinates, colour differences and fastness tests to rubbing/washing results of dyed samples were examined. The pilling test results (4+) and colour fastness to rubbing/washing (4-5 +) were investigated and adequate results were obtained. The time savings was almost 90% after microwave-assisted treatments. Finally, microwave-assisted enzymatic treatments can be an environmental and sustainable method due to short processing times. In addition using microwave-assisted enzymatic treatment processes were contributed to reduction of carbon footprint.

Key Words: microwave energy, enzymatic treatment, enzyme combination, cotton, reactive dyeing

1. INTRODUCTION

Nowadays enzymatic treatments can be applied extensively to cotton materials. Alternative environmentally friendly new methods contribute to clean production in textile wet processes [1,2]. In addition, it provides effects such as tensile strength, wettability and high whiteness in a shorter time [3,4]. Microwave energy is an alternative heating method widely used for pre-tretment, dyeing, finishing, printing, fixing and drying of fabrics. Microwave-assisted treatments are fast, uniform and effective, environmentally friendly methods [5-7]. There are limited works about microwave-assisted enzymatic treatment of cotton.

In this work, the effect of microwave-assisted treatments with sodium hydroxide, pectinase, cellulase, laccase and different enzyme combination on conventional reactive dyeability of cotton fabric. Hydrophilicity, contact angle, whiteness/yellowness indexes of conventionally and microwave-assisted treated samples were measured and compared with conventionally treated samples with sodium hydroxide. Whiteness and yellowness results were adequate for dark-coloured samples. The CIELab colour coordinates, colour differences and fastness tests to washing/ rubbing results of dyed samples were examined.

2. EXPERIMENTAL WORK

Throughout this experimental work, 100 % cotton fabrics knitted by Ne 30/1 yarns was used. The knit structure was single jersey and the fabric weight was 145 g/m^2. Scourzym L [Alfa

Chemistry] is a pectinase enzyme and can be used at pH 8, Cellusoft L [Alfa Chemistry] is a cellulase enzyme and can be used at pH 4.5-5.5. Novalite IIS [Alfa Chemistry] is a laccase enzyme and can be used at pH 4.5-5.5. The chemical structure of the dye was diazo vinylsuphone reactive dye (C.I. Reactive Black 5). The equipment used for the treatments by microwave-assisted process was Arçelik (MD 891 I) microwave oven. The reflectance values of the dyed samples were measured by X-rite Ci6xBT instrument with specular included mode. The colour values of the samples were calculated with D65 illuminant/10° observer values.

Treatments before dyeing process: Conventional and microwave-assisted processes applied to fabrics before reactive dyeing are seen in Table 1.

Table 1. Conventional (C) and microwave-assisted (M) processes applied to fabrics

Methods	Process	Concentrations	Temperature (°C)	Time (min)	pH	Rinsed Process[b]	Dry Process
Method 1	C	0.2 mL/L Wetting agent	95	60	11	Rinsed Type 1	At room
	M	10 g/L Sodium hydroxide		3.5 (P100) + 2.5 (P20)			temperature
Method 2	C	0.2 mL/L Wetting agent	55	30	8.2-8.5	Rinsed Type 2	At room
	M	1.8 % (o.w.f) Pectinase		1.7 (P60) + 1.5 P(20)			temperature
Method 3	C	0.2 mL/L Wetting agent	55	30	4.5-5.5	Rinsed Type 2	At room
	M	1 g/L Cellulase		1.7 (P60) + 1.5 P(20)			temperature
Method 4	C	0.2 mL/L Wetting agent	55	40	4.5-5.5	Rinsed Type 2	At room
	M	1 % (o.w.f) Laccase		1.7 (P60) + 1.5 P(20)			temperature
Method 5	C	Method 2 ➔ Method 3					
	M						
Method 6	C	Method 2 ➔ Method 4					
	M						
Method 7	C	Method 2 ➔ Method 3➔Method 4					
	M						
Method 8	C	Method 2 ➔ Method 4➔Method 3					
	M						

Dyeing Process: Conventionally and microwave-assisted treated fabric samples were conventionally dyed containing 1 % C.I. Reactive Black 5, 65 g/L Na_2SO_4 and 5 g/L Na_2CO_3 at 60 °C and 70 min with a liquor ratio of 40:1. After dyeing process, the bath was then cooled and dyed samples were rinsed six times for 10 min each stage with sequence of a warm rinsing at 70 °C, a second warm rinsing at 70 °C and neutralizing with acetic acid, soaping at the boil with Perlavin OSV [Dr.Petry], a third rinsing at 70 °C, a final rinsing at 50 °C and eventually a cold rinsing.

2. RESULTS

The following results were obtained from the experimental work carried out on conventional and microwave-assisted enzymatic treatments. After the treatments hydrophilicity of the fabric samples was measured by AATCC-79-2014 test method. The hydrophilicity results of Method 1 and 7 were close to each other. In this case the most effective method is combination of pectinase, cellulase and laccase enzymes to achieve high hydrophility.

In order to investigate the surface property of treated fabrics with conventionally and microwave-assisted, water contact angle measurement were performed. The dynamic and static contact angle values of the samples were measured by using PG-X Measuring Head Pocket Goniometer test device as specified in ASTM D5725-99 (2008) method. Each contact angle values and images of water droplets on fabric surfaces are given in Figure 1 and Table 2 respectively.

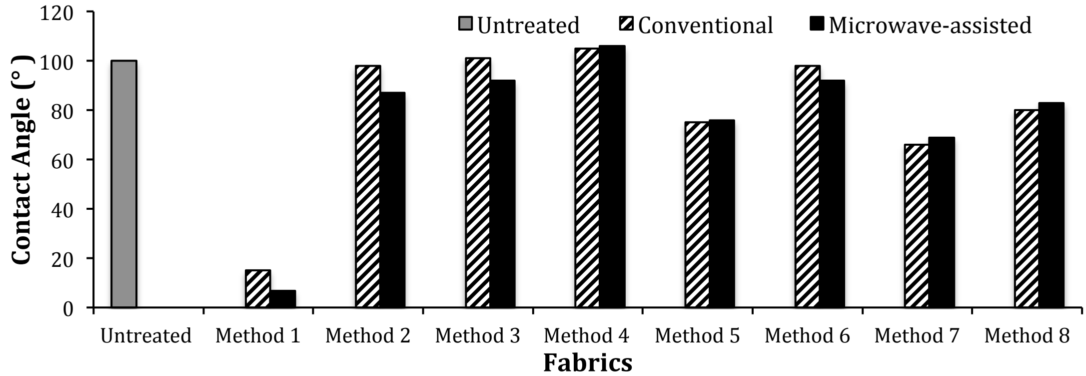
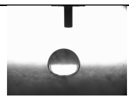
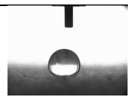
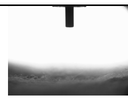
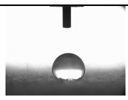
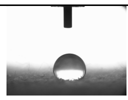
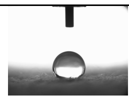
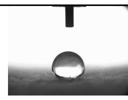
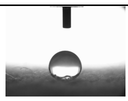
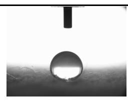
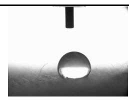
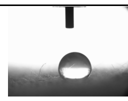
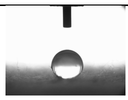
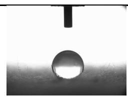
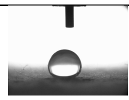
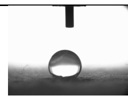
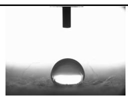
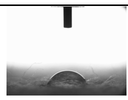
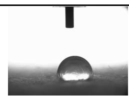
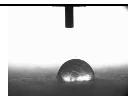
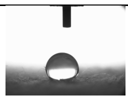
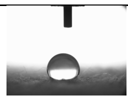
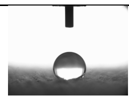
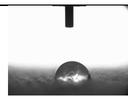
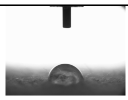
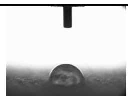
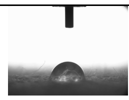
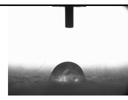
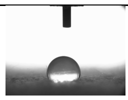
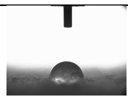
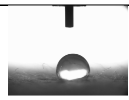
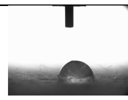


Figure 1. The contact angle values of the fabric samples

The contact angle will be less than 90° on a hydrophilic surface. If the surface is hydrophobic the contact angle will be greater than 90°. Untreated fabric had a fairly hydrophobic surface and showed a water contact of 100°.

Table 2. Images of water droplets on fabric surfaces

Fabrics				
Times (s)	0		4	
Untreated				
Process	Conventional		Microwave-assisted	
Times (s)	0	4	0	4
Method 1				
Method 2				
Method 3				
Method 4				
Method 5				
Method 6				
Method 7				
Method 8				

Microwave-assisted treatments with pectinase enzyme and its combinations were decreased contact angle values of the fabrics (<65-75°). This is an expected behavior assuming that microwave-assisted enzymatic treatments make the fabric surface more hydrophilic.

The CIE Whiteness and Yellowness Indexes of the treated fabrics were calculated in accordance with AATCC 110-2005 and ASTM D 1925-70, respectively. The CIE Whiteness Index results were found to be 6.2 for untreated and ~20 for treated fabrics. The Yellowness Index results were 30 for untreated and ~22 for treated fabrics. Microwave energy has not effective on whiteness and yellowness indexes of the fabrics.

The pilling tendency of the fabrics was assessed by Nu-Martindale (J. Heal) abrasion tester and the number of cycles for each fabric was 2000. The pilling tendency is ‘3’ for untreated fabric. The treated fabrics with conventionally and microwave-assisted processes were assessed as ‘4-5’ and ‘5’. There are no important difference between the pilling tendency results of the fabrics before and after dyeing.

The CIE Lab and colour differences values are summarized in Table 3. Generally, dyed fabrics after microwave-assisted enzymatic treatments were slightly darker.

Table 3. Colour coordinates and differences of treated fabrics

Fabrics	Process	L*	a*	b*	$\Delta E_{CMC (2:1)}$ [a]	$\Delta E_{CMC (2:1)}$ [b]
Method 1	C	37.21	-4.81	-13.79	-	1.76
	M	35.64	-4.86	-13.26	1.76	
Method 2	C	37.88	-5.01	-13.12	0.94	1.93
	M	36.09	-4.82	-12.99	1.36	
Method 3	C	37.43	-4.83	-12.92	0.70	5.41
	M	32.47	-4.52	-13.54	5.16	
Method 4	C	35.40	-4.75	-13.17	2.02	4.87
	M	39.71	-5.04	-12.53	2.91	
Method 5	C	35.08	-4.83	-13.36	2.34	4.80
	M	39.31	-4.96	-12.66	2.45	
Method 6	C	34.56	-4.91	-13.28	2.91	1.54
	M	35.90	-4.87	-12.97	1.56	
Method 7	C	36.57	-4.90	-13.07	0.90	0.57
	M	37.06	-4.81	-12.81	0.96	
Method 8	C	35.17	-4.86	-13.17	2.27	1.33
	M	36.35	-4.97	-13.20	1.06	

[a] Conventional Method 1 was taken as ‘standard’., [b] Conventional processes were taken as ‘standard’.

The washing (colour change/staining) and rubbing (dry/wet) fastness tests were carried out in accordance with the method described in ISO 105-C06 (A1S test conditions) and ISO 105-X12, respectively. The washing and rubbing fastness test results are quite good (gray scale level 4-5 +) as seen in Table 4.

Table 4. Colour fastness test results

Fabric	Process	Washing Fastness							Rubbing Fastness	
		Colour Change	Staining [c]							
			1	2	3	4	5	6	Dry	Wet
Method 1	**C**	5	5	5	5	5	5	5	5	5
	M	5	5	5	5	5	5	5	5	4-5
Method 2	**C**	5	5	5	5	5	5	5	5	4-5
	M	5	5	5	5	5	5	5	5	4-5
Method 3	**C**	4-5	5	5	5	5	5	5	5	4-5
	M	5	5	5	5	5	5	5	5	4-5
Method 4	**C**	5	5	5	5	5	5	5	5	4-5
	M	5	5	5	5	5	5	5	5	4-5
Method 5	**C**	5	5	5	5	5	5	5	5	4-5
	M	5	5	5	5	5	5	5	5	4-5
Method 6	**C**	5	5	5	5	5	5	5	5	4-5
	M	5	5	5	5	5	5	5	5	4-5
Method 7	**C**	5	5	5	5	5	5	5	5	4-5
	M	5	5	5	5	5	5	5	5	4-5
Method 8	**C**	5	5	5	5	5	5	5	5	4-5
	M	5	5	5	5	5	5	5	5	4-5

[c] 1: Secondary Cellulose Acetate, 2: Bleached Unmercerized Cotton, 3: Nylon 6.6, 4: Polyester (Terylene), 5: Acrylic (Courtelle), 6: Wool Worsted.

The microwave energy did not have an important effect on the washing and rubbing fastness properties of the dyed fabrics.

Total process times and calculated time saving are given in Table 5. It is clear that the time saving was almost 90 % after microwave-assisted treatments.

Table 5. Total process times and time saving [d]

Process	Process Times (s)		∑ Process Times (s)		Time Saving (%)
	C	M	C	M	
Method 1	3600	210 + 150	3600	360	90
Method 2	1800	100 + 90	1800	190	89
Method 3	1800	100 + 90	1800	190	89
Method 4	2400	100 + 90	2400	190	92
Method 5	1800	100 + 90	1800	190	89
Method 6	2400	100 + 90	2400	190	92
Method 7	2400	100 + 90	2400	190	92
Method 8	1800	100 + 90	1800	190	89

[d] Dyeing process time was neglected.

Generally, the results suggest that sustainable microwave-assisted pre-treatment and dyeing processes can be improved by reducing the carbon footprint in a shorter time and less energy. It is evident that in some studies, alternative-dyeing methods by optimization of microwave assisted dyeing procedures can be performed to the conventional method [8]. Even enzymatic pre-treatment and reactive dyeing process can be combined and by the way sustainable, environmentally new processes can be improved [3,9].

3. CONCLUSION

Microwave-assisted enzymatic treatments can be applied in less time. They are more effective than conventional processes. It is clear that, microwave-assisted process is an important alternative, enviromentally and sustainable method for cotton knits. Further investigations have also been carried out to optimize process time of microwave-assisted enzymatic treatments and reactive dyeing in the same bath.

4. ACKNOWLEDGMENT

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