EFFECT OF REACTIVE DYEING AND CELLULASE TREATMENT ON THE PILLING PROPERTIES OF COTTON KNITTED FABRIC

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Abstract: With the increasing popularity of cotton knitted fabric, greater demands for quality have been required as end-users have become more aware of its negative properties. Pilling has become a much more serious problem for the knitted apparel. Although cellulase treatment has been well known as a suitable method for overcoming the pilling problem so as to gain more desirable quality and appearance of dyed goods, it is important to understand how the dye affects the performance of the cellulase treatment. Therefore, this paper is aimed to study the relationship of reactive dyes and cellulase treatment on the pilling properties of cotton knitted fabric. Two commercial reactive dyes with different chemical structures were used. The dyeing and cellulase application were conducted in different sequence which were aimed to study the effect of those processes on the pilling properties of cotton knitted fabric. Experimental results revealed that the pilling results were varied depending on the different stages of applying reactive dyeing and cellulase treatment. The results were recorded and would be discussed thoroughly.

1. Introduction

With the rising popularity of cotton knitted fabric, greater demands for quality have been required as end-users have become more aware of its negative properties. Pilling has become a serious problem for the knitted apparel. Although cellulase treatment has been well known as a suitable method for overcoming the pilling problem so as to gain more desirable quality and appearance of dyed goods, it is important to understand how the dye affects the performance of the cellulase treatment [1-6]. Therefore, this paper is aimed to study the relationship of reactive dyes with different chemical structure and cellulase treatment on the pilling properties of cotton knitted fabric.

2. Experimental

2.1. Material

100% cotton knitted fabrics were used and their specifications were shown in Table 1.

Table 1: *Specifications of the scoured and bleached knitted fabrics used*

Fabric type	Yarn Count, NeC	Loop Length (per inch)	Fabric Count, Wales x Course (per inch)
Single jersey	22/1	0.802	27 x 44
Interlock	22/1	0.606	24 x 30

2.2. Enzymatic treatment

A commercially enzyme (acid cellulase with CMC activity of 2035 CMCU/g) was used. In the enzymatic treatment, fabric samples (size 150mm x 300mm) and were treated with 1% enzyme with liquor-to-goods ratio of 10:1. The pH for the enzymatic treatment was maintained at 5.0 and the treatment temperature was kept at 55ºC throughout the treatment time of 50 minutes. After enzymatic treatment, the treatment temperature was increased to 80ºC for 10 minutes for deactivating the enzyme. Afterward, the fabric samples were washed with running deionised water at room temperature for 5 minutes and then completely dried in an oven with temperature of 90ºC. After drying, the fabric samples were conditioned at 20±2ºC with relatively humidity of 65±2% for at least 24 hours prior to further treatment.

2.3. Dyeing process

Two reactive dyes were used and their characteristics were shown in Table 2 and the dyeing method was shown in Table 3. After dyeing, the fabric samples were washed with 1% non-ionic detergent to remove the unfixed and hydrolysed reactive dyes. The washed fabric samples were then completely dried in an oven with temperature of 90ºC. After drying, the fabric samples were conditioned at 20±2ºC with relatively humidity of 65±2% for at least 24 hours prior to further use.

***Table 2**: Reactive dyes used*

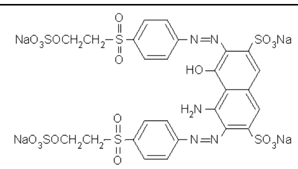
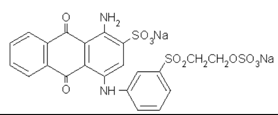
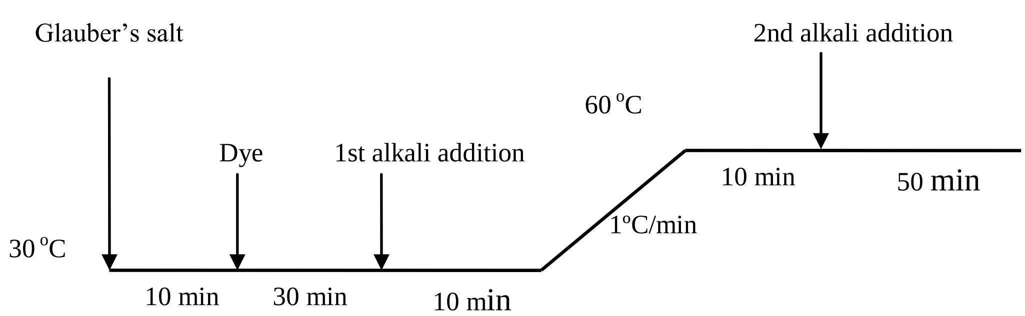
Code	Chemical Structure	Reacting system
C.I. Reactive Black 5 (RB5)		VS-VS (Bifunctional)
C.I. Reactive Blue 19 (RB19)		VS (Monofunctional)

***Table 3**: Dyeing method and parameters*

Liquor-to-goods ratio	10:1	
Dye conc. (%owf)	1.0	3.0
Glauber's salt (g/l)	40	60
Soda ash (g/l)	10	15
1st alkali addition	1/3 amount of soda ash	1/2 amount of soda ash
2nd alkali addition	2/3 amount of soda ash	1/2 amount of soda ash



2.4. Weight Change

The weight change of fabric samples were measured in accordance with ASTM D3776-96.

2.5. Pilling test and assessment

The pilling resistances of the fabric samples were evaluated by EN ISO 12945-1 (I.C.I. pilling box). Three ratings were taken and their average rate was reported.

3. Results and Discussion

3.1. Weight change of undyed fabric samples after enzymatic treatment

The percentage of weight change between undyed single and interlock fabrics after enzymatic treatment is shown in Table 4.

***Table 4**: Weight change (%) of undyed fabric samples after enzymatic treatment*

Fabric Structure	Weight Loss
Interlock	1.31%
Single jersey	1.20%

The result from the Table 4 showed that both types of knitted fabrics treated with enzyme have certain degree of weight loss. When the type of fabric was compared, the interlock knitted fabric had more weight loss than the single jersey knitted fabric with the same enzymatic treatment. This difference might be due to the fabric structure. The single jersey knitted fabric has a closer and packed fabric structure when compared with the interlock knitted fabric. The looser the fabric structure of the interlock fabric, the easier of the penetration of cellulase to access the cotton fabric resulting in more cellulose would lose due to enzymatic hydrolysis.

3.1.1. Effect of reactive dye on the weight change of dyed fabric samples

The different weight loss after enzymatic treatment with variation of dyeing treatment is shown in Figure 1. From Figure 1, it was found the single jersey and interlock fabrics continue to have weight loss while enzyme treatment was followed after dyeing. For those dyed fabric samples, different degree of weight loss was found after enzymatic treatment when compared with the undyed sample. This was not a desirable result as many studies had revealed that reactive dyes on the substrate inhibit the cellulase catalytic reaction. In Figure 2, the difference of percentage of weight loss between the dyed and undyed fabric samples was shown. This undesirable result could be explained by the removal of unfixed dye on the fabric after enzyme washing. This was possibly due to the insufficient of the removal of unfixed dye in the dyeing procedure. This experimental flaw may lead to insignificance of the data; the weight loss of the dyed fabric can no longer precisely indicate the performance of the enzyme treatment. However, the reduction in weight loss of the fabric dyed with RB5 in 3% concentration was significant as the RB19 dyed fabric which was dyed in the same concentration showed an increase in weight loss. As the two dyed fabrics undergone the same dyeing procedure with the same dye concentration, it could assume that these two dyed fabrics contain similar amount of unfixed dye with similar weight. Therefore, this variation in percentage of weight loss could indicate that RB5 has a larger inhibitory effect than RB19. The bifunctional structure of RB5 might be accountable for this effect.

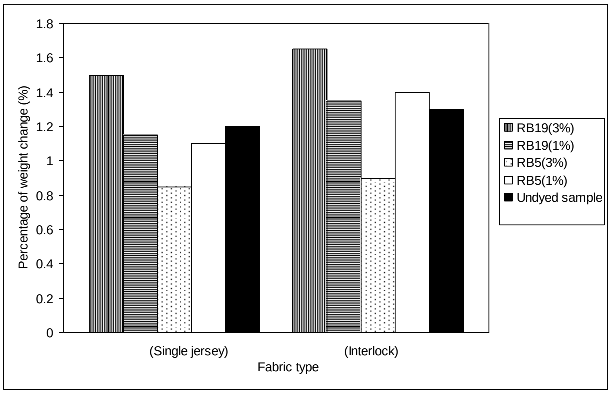


Figure 1: *Percentage weight loss of dyed fabric samples under the influence of enzymatic treatment*

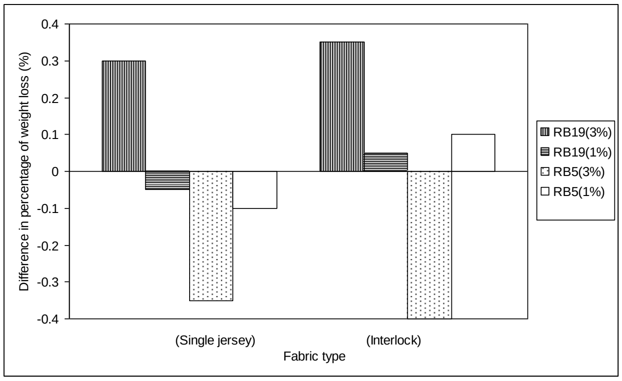


Figure 2: *Difference in percentage of weight loss between the undyed and dyed fabric samples*

3.2. Initial pilling performance

The initial pilling performance of the fabric samples was assessed right after various wet processing to determine the influence of the different combinations of dyeing and enzymatic treatments on fabric surface appearance in terms of pilling. In this initial pilling performance, the fabric samples were not subjected to pilling test. When fabrics were subjected to dyeing, abrasion and mechanical agitation can lead to the formation of pill. In order to determine the influence of dyeing process on the pilling performance, the fabric samples were assessed for the pilling performance after dyeing without any enzymatic treatment and the results are shown in Table 5.

Table 5*: Initial pilling performance of undyed and dyed fabric samples without enzyme treatment*

Fabric structure	Dye	Dye concentration (owf)	Pilling rate
Single Jersey	RB5	3%	4.28
Single Jersey	RB5	1%	4.36
Single Jersey	RB19	3%	4.31
Single Jersey	RB19	1%	4.33
Single Jersey	Undyed	--	4.38
Interlock	RB5	3%	4.11
Interlock	RB5	1%	4.16
Interlock	RB19	3%	4.25
Interlock	RB19	1%	4.13
Interlock	Undyed	--	4.42

By comparing the undyed and dyed fabric samples for the pilling performance, it was found that all fabrics after dyeing had a reduction in the pilling performance. However, the reduction was small, especially for the single jersey fabric. The interlock fabrics tend to have greater drop in the pilling rating. The looser fabric structure of the interlock fabric samples may be accountable for this result. The variation on the dye and dye concentration did not have a regular effect of the pilling performance.

3.3. Initial pilling performance after enzymatic treatment

Table 6 shows the pilling performance of enzyme treated fabric samples without dyeing. All samples reached the best rating, i.e. 5. By comparing the pilling performance of the untreated fabric samples in Table 5, the enzyme showed their capability of defuzzing.

Table 6*: Initial pilling performance of enzyme treated fabric samples without dyeing*

Fabric structure	Pilling rate
Single Jersey	5
Interlock	5

3.4. Initial pilling performance of fabric samples subjected to various combinations of dyeing and enzymatic treatment

Table 7 shows the pilling performance of the dyed fabric samples before enzyme treatment. All samples resulted better rating for RB19 dyed samples, while RB5 dyed interlock fabric samples showed a minimal drop in the pilling rate after treated with enzyme.

Table 7*: Initial pilling performance of the pre-dyed fabric after enzymatic treatment*

Fabric structure	Dye	Dye concentration (owf)	Pilling rate (dyeing before enzymatic treatment)	Pilling rate (dyeing after enzymatic treatment)
Single Jersey	RB5	3%	5	5
Single Jersey	RB5	1%	5	5
Single Jersey	RB19	3%	5	5
Single Jersey	RB19	1%	5	5
Interlock	RB5	3%	4.88	4.88
Interlock	RB5	1%	4.96	5
Interlock	RB19	3%	5	5
Interlock	RB19	1%	5	5

Table 7 shows the pilling performance of the fabric samples dyed after enzymatic treatment. Once again, the fabric samples showed an improved pilling performance. Only one interlock fabric samples have a slight drop on this performance but the rating is still better than the untreated fabric samples. By comparing the data from Tables 6 and 7, it was found that enzyme showed a similar performance for removing the initial pilling. The dyed fabric samples do not have significant effect in this aspect, although a very minimal drop in the performance was recorded for the RB5 dyed interlock samples. It was also found that both combination of wet process (dyed before enzyme treatment or dyed after enzyme treatment) showed insignificant difference on the initial pilling performance to the final products. It was suggested that the decline of the enzyme performance was caused by the inhibitory effect of the existed reactive dye and the decline of pilling performance cause by abrasion occurred during dyeing process after enzyme treatment seemed to have the equal effects on the final products [7-9].

3.5. Pilling performance after pilling test

3.5.1. Undyed fabric samples

Table 8 shows the pilling performance of the undyed fabric samples before and after enzyme treatment. The pilling rating clearly indicates that the enzyme significantly improved the pilling resistance of the treated fabric samples. The interlock fabric was found to have better pilling resistance either with or without enzyme treatment. The difference between single jersey and interlock fabric was increased when the fabric samples were enzyme treated. This result could consequently indicate that the enzyme gave better improvement of anti-pilling property on the interlock than on the single jersey.

Table 8*: Pilling performance of the undyed samples after pilling test*

Fabric	Pilling Rate	
	with enzymatic treatment	without enzymatic treatment
Single Jersey	4.33	3.50
Interlock	4.63	3.63

3.5.2. Influence of dye on the untreated fabric

The dyed fabrics without any enzymatic treatment were subjected to the pilling test. The results are shown in Table 9. By comparing the data of Table 9 with the pilling rate of the fabric samples shown in Table 8, it was found that the existence of reactive dye do not give any significant improvement to the pilling resistance property of the samples. The variation on dye and concentration has no significant influence on the result either.

3.5.3. Influence of dyeing before enzymatic treatment

In Table 9, the inhibitory effect of reactive dye on the enzyme performance was clearly indicated as all the dyed fabric samples showed a reduction on the pilling rate after enzymatic treatment while comparing the result with the pilling rate of the undyed fabric sample after enzymatic treatment as shown in Table 8. All dyed fabric samples showed a slight decline in the pilling performance after pilling test. This could prove that the reactive dye had inhibitory effect on the performance of both types of enzyme. The result could be explained by that, the reactive dyes form covalent bonds with the hydroxyl groups of cellulose, thus creating in a sense of derivatization in the amorphous regions and changing the structure of the cellulose for the enzymes to some extent. The substitution of the hydroxyl groups affects enzymatic recognition and thus impedes degradation, and finally it leads to the poorer performance of enzyme. In addition, the result showed that, samples dyed with RB5 in 3% concentration have a slightly larger drop in pilling rate when comparing with the rest of the samples which contain the same knit structure. From the data, it was found that the increase in dye concentration from 1% to 3% for the RB19 dyed fabric has no observable impact on the pilling performance which can be seen as an indicator for the performance of enzyme. However, for the RB5 dyed samples, when the concentration was increased to 3%, it showed a clear drop in the pilling performance. This result could further support the suggestion that the formation of crosslinks within the fibres were dramatically increased when the fabric were dyed with bifunctional reactive dye in higher concentration, and it consequently leaded to the increase of inhibitory effect on the enzymatic hydrolysis [7-9].

Table 9: *Pilling performance of the dyed samples without enzyme treatment after pilling test*

Fabric structure	Dye	Dye concentration (owf)	Pilling rate (dyed without enzymatic treatment)	Pilling rate (dyeing before enzymatic treatment)	Pilling rate (dyeing after enzymatic treatment)
Single Jersey	RB5	3%	3.58	3.79	4.21
Single Jersey	RB5	1%	3.63	4.02	4.37
Single Jersey	RB19	3%	3.54	3.93	4.37
Single Jersey	RB19	1%	3.63	3.93	4.28
Interlock	RB5	3%	3.58	3.83	4.58
Interlock	RB5	1%	3.54	4.15	4.67
Interlock	RB19	3%	3.58	4.08	4.67
Interlock	RB19	1%	3.54	4.08	4.54

3.5.4. Influence of dyeing after enzymatic treatment

The pilling performance of the samples, which were dyed after enzyme treatment, after pilling test was shown in Table 9. The results were closed to those recorded from the undyed samples with enzyme treatment shown in Table 8. It indicates that the afterward dyeing process with various dyestuff and concentration does not affect the pilling resistance property of fabric. Therefore, in terms of pilling performance, it was found that better result was produced when the fabric was dyed after treated with enzyme. As the afterward dyeing process did not show any significant deterioration of the initial pilling performance and better pilling resistance were observed as there was no inhibitory effect on the enzyme treatment caused by the pre-existed dyes on the fabric.

4. Conclusion

In the project, the effect of the pre-existed reactive dyes on the enzymatic reaction was further proved as the predyed samples with enzyme treatment all showed a drop on the performance of the pilling resistance property. The percentage of weight loss of the predyed samples after enzyme treatment was failed to indicate the effect of the pre-existed dyes due to the effect of undesirable removal of the unfixed dyes on the fabric after dyeing, however the distinct drop in weight loss of the RB5 dyed fabric with 3% concentration, accompanied with the similar results of the pilling test, had demonstrated the stronger inhibitory effect of the bifunctional reactive dyes than the monofunctional dye. The distinction is not observable until the concentration is increased into 3%. Since the bifunctional dyes would form crosslinks with the highly accessible regions as well as between chain molecules of differently ordered domains in the dyestuff concentration of 3%, the enzyme accessibility would be strongly reduced. On the other hand, since monofunctional dye molecules could form crosslinks in the highly order regions of the cellulose, the increase on dyestuff concentration of RB19 from 1% to 3% did not show an observable impact on the inhibitory effect to the enzyme reaction. The afterward dyeing treatment showed no observable effect on the previous enzyme treatment. Therefore, to obtain better pilling resistance for the colored fabric, the fabrics were recommended to be dyed after the enzyme treatment.

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