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Ink jet printing of bio-treated linen, polyester fabrics and their blend

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ABSTRACT

Cellulosic fabrics were surface modified using Brewer's yeast filtrate and cellulase enzymes (Valumax A828, Valumax A356) to enhance its affinity to ink jet printing. The effect of enzymes on the surface structure and morphology of the cellulosic fabrics used has been illustrated using scanning electron microscope. Related test as tensile strength have been measured. The bio-treated cellulosic fabrics were digitally printed and the colour strength (K/S) and % increase in K/S were measured. Effect of different conditions (enzyme concentration, temperature and time) was investigated to obtain the optimum condition of each enzyme for each fabric that or which was indicated by higher colour strength. Results show a noticeable increase in the K/S especially for cellulosic linen and its blend compared to the standard samples. The optimum conditions to obtain the higher K/S by using Brewer's yeast filtrate and the other two cellulase enzymes in the pre-treatment of ink jet samples were obtained.

1. Introduction

Recently, there has been an increasing interest in the application of ink-jet printing for textile applications. The technique of ink-jet printing offers benefits such as speed, flexibility, creativity, cleanliness, competitiveness and eco-friendliness (Gupta, 2001; Kan, Yuen, Ku, & Choi, 2005; van Parys, 2002). In conventional textile printing, dyes are applied along with the printing chemicals in the form of a printing paste. However, due to the requirements of ink purity and specific conductivity for the digital ink-jet printing (Aston, Provost, & Masselink, 1993; Schulz, 2002; Siemensmeyer, Siegel, Ervine, & Bullock, 1999), none of the conventional printing chemicals such as alkali, urea and sodium alginate can be directly incorporated into the ink formulation and therefore a printing medium should be prepared for textile digital ink-jet printing (Aston et al., 1993; Choi et al., 2005; Kan, Yuen, Tsoi, & Chan, 2011). Inks for fabric printing are usually classified into two categories of dye-based and pigment-based inks. Pigment-based ink printing shows better water fastness and light fastness of the fabric than dye-based ink printing. It saves water and energy, and the most important thing is that it is environmentally friendly.

Polyester is one of the most important textile materials in ink jet printing since it has a lot of special characteristics, such as superior strength and resilience. But the molecular part of poly(ethylene terephthalate) (PET) lacks polar groups, which causes it to have low surface-energy and poor wettability (Yuen, Ku, Kan, & Choi, 2007). When polyester fabric is printed with pigment-based inks, the printed patterns will have poor colour yield and may bleed easily, especially for thin fabric. Thus, the pre-treatment process is necessary for polyester fabric ink jet printing. On the other hand, linen, which is a natural bast fibre, has unparalleled characteristics such as a feel of freshness and a magnificent brilliance. It is very hygienic and it imparts an air of satisfaction and style to the wearer (Behera, 2007). With the trend of fashion towards natural, comfortable yet elegant fabrics, linen and linen-blended fabrics have gained prestige and increased reputation. The objective of our work is to improve the colour parameter of ink jet printing polyester and linen fabrics and their blend using eco-friendly method by applying different enzymes.

2. Experimental

2.1. Materials and reagents

2.1.1. Fabrics

- Semi finished cellulosic linen fabrics supplied by Textile Industries Egyptian Co. Ointex, Egypt.
- Polyester (PE) knitted fabric of 150 g/m^2 supplied by a private sector company.
- Linen/polyester blend 50/50 supplied by Textile Industries Egyptian Co. Ointex, Egypt.

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2.1.2. Enzymes and yeast

The following two different commercial enzymes and Brewer yeast were used:

(a) Valumax® A828 from *Aspergillus oryzae* was supplied by Novozymes, Denmark, of declared activity 2000 ECU/g; Cellulase Unit per gram.
(b) Valumax® A356 was purchased from Novozymes, Denmark: its declared activity 120 CVU/g; Combi Cellulase Units per gram. It is produced by submerged fermentation of genetically modified microorganisms.
(c) Locally available Brewer's yeast was also used.

2.2. Methods

2.2.1. Scouring

Before using, the fabrics were washed with a solution containing 1 g/l non-ionic detergent at 70 °C for 1/2 h. Then they were washed, and air dried at room temperature.

2.2.2. Preparation of Brewer's yeast filtrate

The suspension of active yeast was prepared according to a reported method (Marie, El-Hamid, El-Khatib, & El-Gamal, 2004). The procedure was adopted as follows.

450 g dry weight of Brewer's yeast was pasted with 150 g sugar, and then 1 L of warm water (40 °C) was added to the paste of yeast while stirring for a period of time until the yeast was brewed. Finally the solution was filtered and frozen.

2.2.3. Bioprinting of the applied fabrics (linen, polyester and their blend)

The fabric samples were first printed with a printing paste containing only the thickening agent and the enzyme (i.e. uncoloured paste). The recipe of the uncoloured printing paste was as follows:

Enzyme	*X* g
Sodium alginate	30 g
Water	*Y* g
Total	1000 g

Sodium alginate "thickening agent" was soaked in small amount of water overnight at room temperature before preparing the uncoloured printing paste. The enzymes used (50, 100, 200, 220, 240 and 260 g/kg) or Brewer's yeast filtrate (50, 100, 300, 600 and 900 ml/kg) was then added on the thickener suspension and the whole paste was adjusted to 1 kg with the addition of the necessary amount of water. The pH of the paste was adjusted according to the enzyme used and then the samples were printed and left in the oven for different intervals of time (60, 120, 180 and 240 min) and also at different degrees of temperatures (30, 40, 50, 60, 70 and 80 °C).

2.2.4. Ink jet printing of bio-printed fabric

The bio-printed linen, polyester and its blend samples were digitally printed with yellow pigment based ink by ink jet machine (inkjet plotter printer – mimaki) followed by air drying.

2.2.5. Fixation

The resist printed fabrics were fixed via thermo fixation at 150 °C for 5 min.

2.3. Analysis and measurements

2.3.1. Colour measurements

The colour strength (K/S) was measured by reflection spectroscopy with a Hunter Lab UltraScan PRO spectrophotometer according to a standard method (Judd & Wyszecki, 1975).

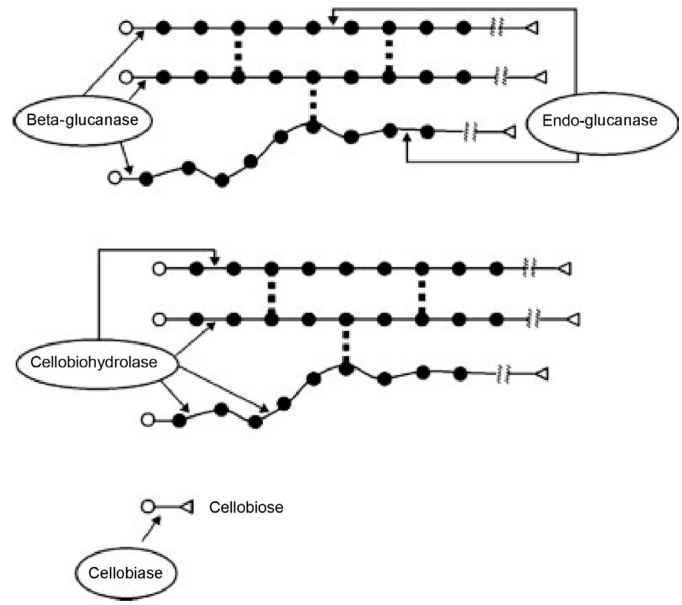

Fig. 1. Enzymatic reaction of cellulose by cellulase.

2.3.2. Tensile strength of fabric

The test was carried out according to the ASTM Standard Test method D 682 1924 on a tensile strength apparatus type FMCW 500 (Veb Thuringer Industries Work Rauenstein 11/2612 German) at 25 ± 2 °C and 60 ± 2% relative humidity (ASTM Standard C33 (ASTM D1682-64 e1), 1975).

2.3.3. Morphology study of the fabrics

The untreated and treated fabrics were analysed by scanning electron microscopy (SEM), Topcon-Microscope (ATB-55) to investigate morphological changes of the surface structure.

2.3.4. Determination of fastness properties

The treated samples were washed as per the conditions specified in the test AATCC test method (AATCC Test Method (36-1972), 68, 1993). The colour fastness to rubbing, perspiration and light were determined according to the AATCC test methods (AATCC Test Method (8-1989), 68, 1993; AATCC Test Method (15-1989), 68, 1993; AATCC Test Method (16A-1989), 68, 1993).

3. Results and discussion

Commercial enzyme treatments have usually target for cotton, however, there were limited reports on other cellulose fibres (Buschle-Diller, Zeronian, Pan, & Yoon, 1994; Pedersen, Screws, & Cedron, 1992). Thus one of the objectives of our work is to determine whether the previous findings for cotton pre-treatment with cellulase were valid for other cellulosic fabrics such as semi finished linen, linen/polyester blend and for non-cellulosic fibres such as polyester to be applied in inkjet printing. Cellulases are secreted by various fungi and bacteria as complex mixtures of three major kinds namely endoglucanase (EGs), exocellobiohydrolase (CBHs), and beta glucosidases. The proposed mechanism of cellulase action onto cellulose is illustrated in Fig. 1. Endoglucanase EGs hydrolyze cellulase that are/because they are randomly long the chains, preferentially the amorphous region. CBHs attack the chain ends and produce primarily cellobiose coupled with the binding domains associated with the enzyme. The cellobiose and any small chain oligomers produced by CBHs are then hydrolyzed by the third enzyme beta glucosidase into glucose (Kan, Yuen, Jiang, Tung, & Cheng, 2007). The biological analysis of Brewer's yeast (*Saccharomyces cerevisiae*)

Table 1
Effect of enzyme concentration on K/S and %K/S of inkjet printed samples.

Enzyme used	Concentration	Fabric					
		Linen/polyester		Linen		Polyester	
		K/S	%K/S	K/S	%K/S	K/S	%K/S
Untreated	–	0.76	100	1.76	100	12.02	100
Brewer's yeast	50 ml/kg	0.88	115.79	0.55	31.25	0.28	2.33
	100 ml/kg	0.93	122.37	0.73	41.48	0.49	4.08
	300 ml/kg	1.01	132.90	0.92	52.28	0.72	5.99
	600 ml/kg	2.40	315.79	2.17	123.30	3.62	30.11
	900 ml/kg	4.01	527.63	3.11	176.71	7.60	63.22
Valumax A356	50 g/kg	1.56	205.26	3.20	181.82	9.51	79.12
	100 g/kg	1.70	223.68	3.25	184.66	9.72	80.86
	200 g/kg	2.78	356.79	4.10	232.95	11.23	93.43
	220 g/kg	2.60	342.11	3.48	197.73	10.75	89.43
	240 g/kg	2.53	332.89	2.84	161.36	10.47	87.10
	260 g/kg	2.21	290.79	2.78	157.95	10.28	85.52
Valumax A828	50 g/kg	1.19	156.58	3.05	173.30	7.11	59.15
	100 g/kg	1.45	190.79	3.16	179.55	8.28	68.88
	200 g/kg	3	394.74	4.16	236.36	12.69	105.57
	220 g/kg	2.62	344.74	3.62	205.68	10.75	89.43
	240 g/kg	2.23	293.42	2.83	160.80	10.55	87.77
	260 g/kg	1.69	222.37	2.10	119.31	9.93	82.61

filtrate states the presence of different types of enzymes viz. protease, lipase and amylase, beside several proteolytic enzymes in the suspension (Reed & Nagodawithana, 1991). In previous work (Salem, 2004), it was used to improve the physical properties and printability of polyester fabric yielding an increase in the wettability and colour strength (K/S) of the treated polyester samples attributed to the hydrolytic effect of yeast on ester linkages of polyester polymer chain. After explaining the reaction mechanism of each enzyme the paper will study the optimum condition of each and its effect on the colour strength (K/S) of printed ink jet fabrics.

3.1. Effect of enzyme concentration

The effect of enzyme concentration on the K/S of the printed fabrics was investigated as follows: a series of white printing pastes containing thickening agent only or using different concentrations of the enzyme or Brewer's yeast filtrate were prepared with required conditions of each one (pH, treatment time and temperature). Finally the temperature was raised to kill the enzyme before printing. The digitally printed fabrics were fixed as mentioned in Section 2. Brewer's yeast filtrate concentrations (0, 50,100, 300, 600, 900 ml/kg of the white printing paste) were prepared and applied to the three fabrics samples using flat screen printing technique. The samples were placed in the oven for 2 h and half at 30 °C. While for the two cellulase enzymes the prepared printing paste was adjusted at pH = 10 and then the samples were printed and put in the oven at 50 °C for 1 h. Finally all the printed samples were digitally printed and fixed as mentioned in Section 2. The K/S and %K/S are illustrated in Table 1.

Table 1 shows that by increasing the concentration of Brewer's yeast filtrate from 50, 100, 300 until it reaches 900 ml/kg paste the %K/S increases from 115.79, 122.37, 132.90 to 527.63 for linen while for polyester/linen blend and polyester is (31.25, 41.48, 52.28 to 176.71 and 2.33, 4.08, 5.99 to 63.22) respectively. It is clear that linen printed samples give the maximum increase in %K/S but for polyester it has not reached the standard. This could be attributed to the construction of the fabric structure and/or those temperature and treatment times used are not the optimum for this fabric. In another hand if we compare these results with the two cellulase enzymes, we notice that for all the fabric samples used %K/S increases by increasing the concentration until it reaches the maximum at 200 g/kg paste given viz. for linen samples 356.79, 394.74 for Valumax A356 and Valumax A 828 relatively then it starts to decrease. The polyester samples treated by Valumax A828 give slight increase in the K/S than the standard (12.69) could do. The difference in the activity of both two cellulase enzymes plays a role in the increase in the values of K/S and %K/S of the printed samples, such that Valumax A 828 gives the higher values than Valumax A356.

3.2. Effect of treatment temperature

To investigate the effect of treatment temperature on the K/S and %K/S of the treated fabric, three printing paste containing 900 ml/kg of Brewer's yeast filtrate and 200 g/kg paste for Valumax A356 and Valumax A 828 were prepared. The pH of the printing pastes was adjusted at 4 and 10 for Brewer's yeast filtrate and the two Valumax enzymes relatively. Then all the used fabrics were printed and kept in oven at different temperatures (30, 40, 50, 60, 70 and 80 °C) for 2 and a half an hour for Brewer's yeast filtrate samples and one hour for the other two cellulase enzyme samples. Finally the samples were digitally printed and fixed.

From Table 2 we conclude that the optimum temperature for using Brewer's yeast filtrate was at 70 °C, while for Valumax A356 and A828 it was at 50 °C regardless of the kind of fabric used.

3.3. Effect of treatment time

Time of pre-treatment has a remarkable effect on enzymatic reaction, in determining the optimum time required to obtain the higher K/S for all the fabrics using the three printing pastes; each contains the optimum concentration of enzyme and adjusted at the required pH (4 and 10 for Brewer's yeast filtrate and the two Valumax enzymes relatively) during preparation. The fabrics were printed and subjected to enzymatic treatment for different durations viz., 60, 120, 180 and 240 min at an optimum temperature for each enzyme (the optimum temperature for using Brewer's yeast filtrate was at 70 °C while for Valumax A356 and A828 was at 50 °C).

Table 3 illustrates the results obtained for all samples.

Table 3 shows bio-treatment of cellulosic linen fabric with Brewer's yeast filtrate before inkjet printing resulting in an increase in the %K/S by 1184.21 after treatment for 60 min at 70 °C in the oven. In the second place linen/polyester blend has given an increase in %K/S by 771.59 and finally for polyester fabric printed

Table 2
Effect of treatment temperature on the K/S and %K/S of the inkjet printed fabric.

Temperature (°C)	Enzyme used	Fabric					
		Polyester		Linen/polyester blend/polyester		Linen	
		K/S	%K/S	K/S	%K/S	K/S	%K/S
Untreated	–	0.76	100	1.76	100	12.02	100
30	Brewer's yeast	4.01	527.63	3.10	176.13	7.60	63.23
	Valumax 356	2.33	306.58	3.54	201.14	8.81	73.29
	Valumax A828	2.46	323.68	3.73	211.93	11.53	95.92
40	Brewer's yeast	4.61	606.58	3.66	207.95	9.29	77.29
	Valumax A356	2.20	289.47	3.73	211.93	8.76	72.88
	Valumax A828	2.29	301.32	3.95	224.43	9.78	81.37
50	Brewer's yeast	5.46	718.42	4.69	266.48	10.75	89.43
	Valumax A356	2.78	365.79	4.10	232.96	11.23	93.43
	Valumax A828	3	394.74	4.16	236.36	12.69	105.57
60	Brewer's yeast	6.40	842.11	5.20	295.45	10.75	89.43
	Valumax A356	2.13	280.26	3.95	224.43	9.78	81.36
	Valumax A828	2.35	309.21	4.07	231.25	8.66	72.05
70	Brewer's yeast	5.27	693.42	5.19	294.89	13.19	109.73
	Valumax A356	2.32	305.26	3.71	210.79	9.73	80.95
	Valumax A828	2.32	305.26	3.66	207.95	8.27	68.80
80	Brewer's yeast	4.16	547.37	4.69	266.48	8.92	74.21
	Valumax A356	2.31	303.95	3.57	202.84	9.47	78.79
	Valumax A828	2.13	280.26	3.45	196.02	8.33	69.03

samples, the increase is by 113.39 but after treatment for 120 min at the same temperature. Otherwise Valumax A828 increases the %K/S by 394.74, 236.37 and 105.57 for linen, linen/polyester blend and polyester relatively at 50 °C for 60 min. Valumax A356 (the lower activity cellulase enzyme) has no effect on the polyester on K/S but for linen and its blend the %K/S increases by 365.79 and 232.95.

3.4. SEM study of the fabrics

We interpret the effects of the enzyme treatments by comparing the micrographs of linen, linen/polyester blend and polyester from different treatment and the micrographs of reference specimens (control samples). Multi-cellular flax fibres are connected to each other by pectin and the outer surface of fibres consists of pectin, lignin, and waxy materials. As seen in the SEM image of untreated fibres (Fig. 2A) the outer surface of non-cellulosic materials around the fibre bundles forms a smooth surface. While Fig. 2B–D shows removing of the outer surface with different degree depending on the enzyme used beside the formation of cracks in the direction of the fibre axis. The breaking down of the linen fibres at the nodes can be understood when we remember that absorbance of water occurs in these regions and that dyes and finishing agents often accumulate there (Batra, 1985). We suggest that the enzymes react rapidly at nodes and degradation is concentrated at these areas.

Fig. 3A presents the SEM pictures of linen/polyester blend while Fig. 3B–D shows the morphology change after the three different treatments using Brewer's yeast, Valumax A828 and Valumax A356. The figures show surface areas pore width, and pore volume of the bio-treated fabric became a little larger than those (untreated) linen/polyester blend leading to better wettability than those of untreated membranes. Brewer's yeast filtrate treatment gives higher number of pores with larger size than the others two cellulase enzymes this could be attributed to its low viscosity.

Fig. 4A shows a typical SEM micrograph of control polyester fibres to have a relatively smooth microscopic with characteristic discrete prominence and less roughness. Fig. 4B–D shows the presence of hemispherical holes and cracks in the surface of polyester which increases the surface area for absorption of water molecules

Table 3
Effect of treatment time on the K/S and %K/S of printed fabrics.

Time (min)	Enzyme used	Fabric					
		Polyester		Linen/polyester		Linen	
		K/S	%K/S	K/S	%K/S	K/S	%K/S
Untreated	–	0.76	100	1.76	100	12.02	100
60	Brewer's yeast	9	1184.21	13.58	771.59	13.43	111.73
	Valumax A356	2.78	365.79	4.10	232.95	11.23	93.42
	Valumax A828	3	394.74	4.16	236.37	12.69	105.57
120	Brewer's yeast	8.52	1121.10	12	681.82	13.63	113.39
	Valumax A356	2.25	296.05	2.63	149.43	10.37	86.27
	Valumax A828	2.28	300	3.66	207.95	11.07	92.10
[5pt] 180	Brewer's yeast	6.24	821.05	4.87	276.7	12.50	103.99
	Valumax A356	2.26	297.37	3.12	177.27	10.94	91.01
	Valumax A828	2.45	186.2	3.21	182.39	8.25	68.64
240	Brewer's yeast	5.69	748.68	4	227.27	10.69	88.93
	Valumax A356	2.51	330.26	2.64	150	9.55	79.45
	Valumax A828	2.55	335.53	3.17	180.11	8.53	70.97

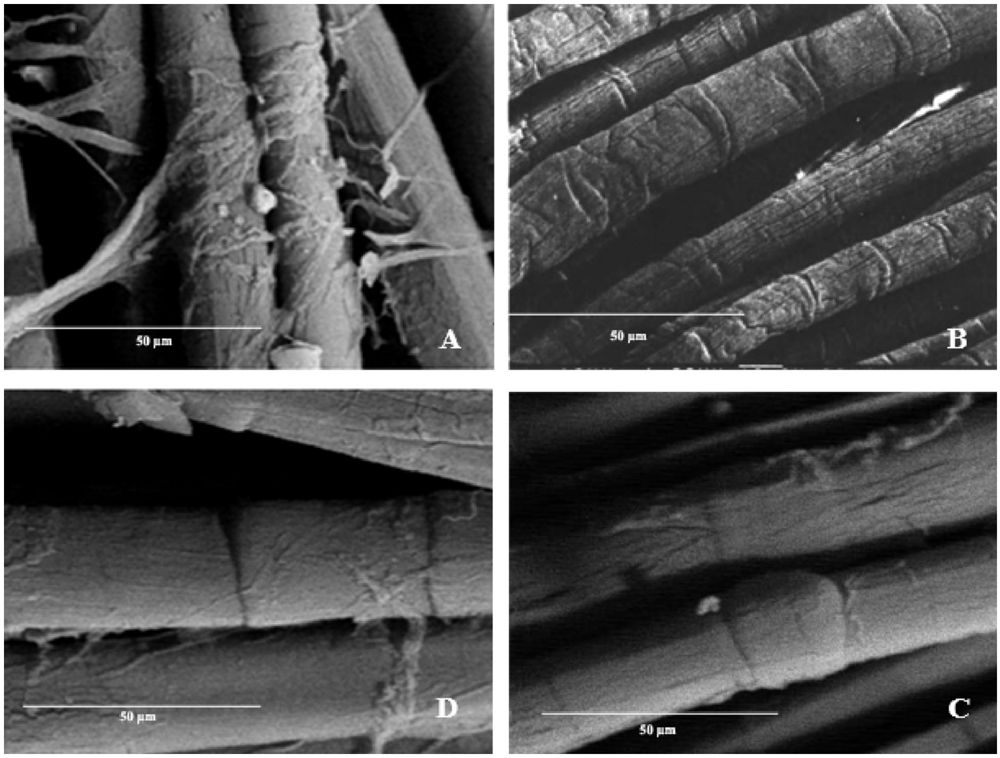

Fig. 2. SEM micrograph of untreated and treated linen fibre.

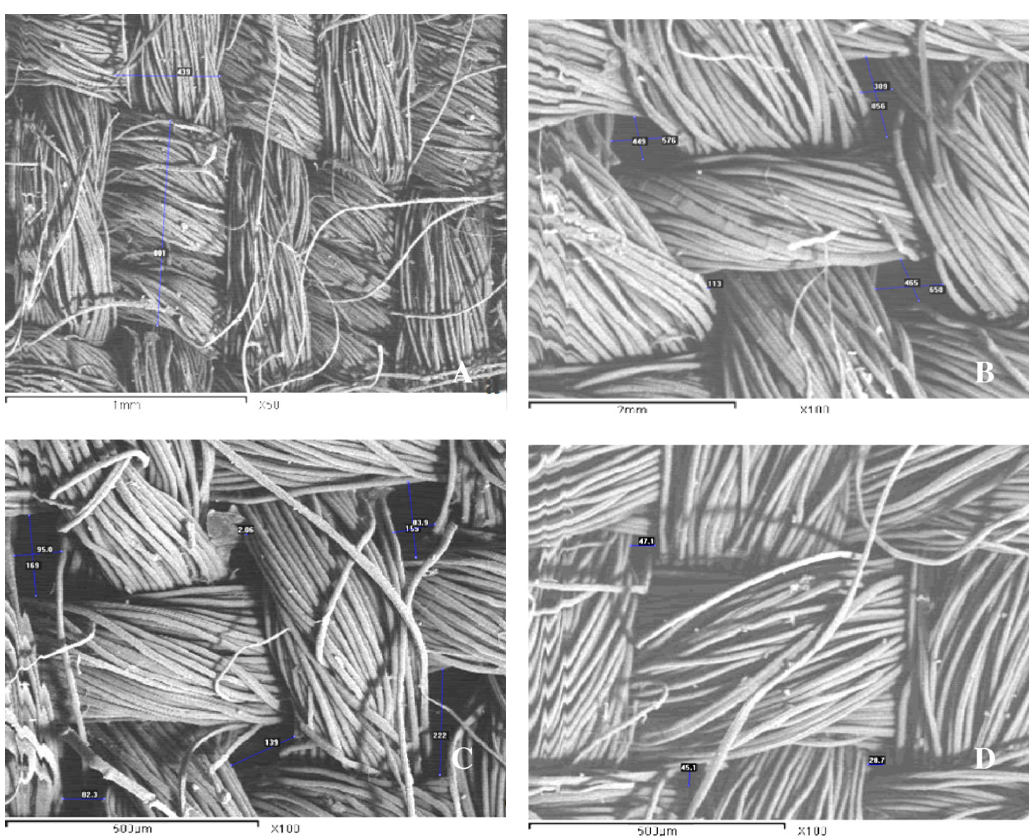

Fig. 3. SEM micrograph of untreated and treated linen/polyester blend fibre.

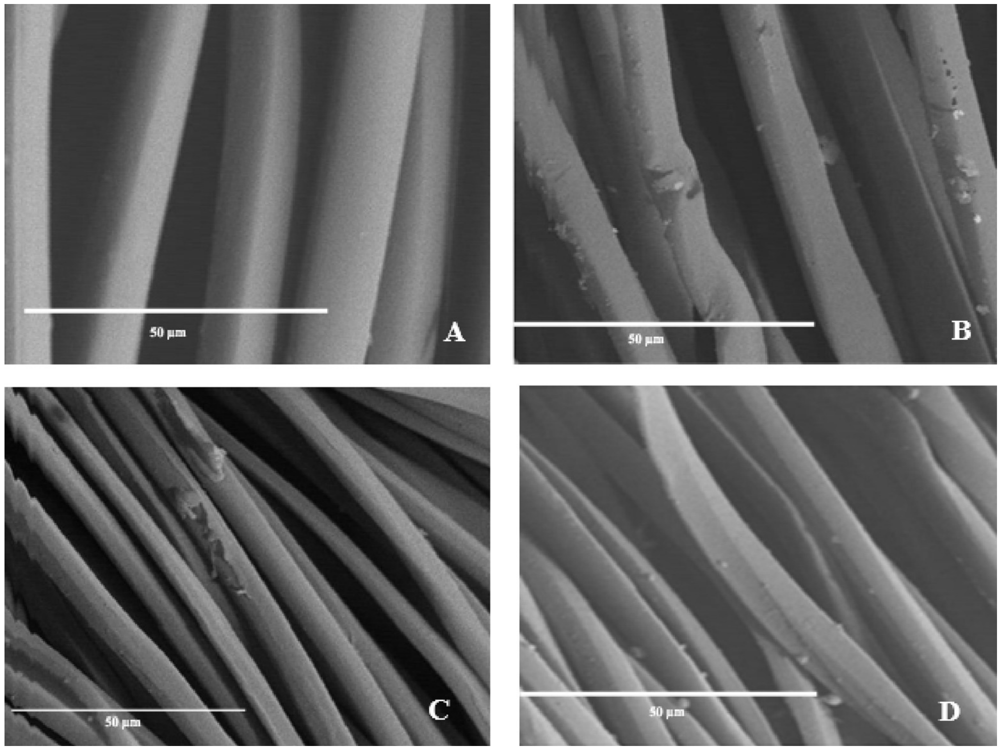

Fig. 4. SEM micrograph of untreated and treated polyester fibre.

and ink trapped (Wavhal & Balasubramanya, 2011). It was shown that the damage after biodegradation by enzyme or microorganisms was apparent as cracks and holes in the polyester fibres improve water penetration and the absorbent properties of regular polyester fabric. These confirmed by previous SEM investigations (Barratt, Ennos, Greenhalgh, Robson, & Handley, 2003; Djordjevic, Petronijevic, & Cvetkovic, 2005; Tokiwa & Calabia, 2004) showed that the damage after biodegradation by enzyme or microorganisms was apparent as cracks and holes in the polyester fibres and that lipases significantly improves water penetration and the

Table 4
Effect of enzyme pre-treatment on the tensile strength of the printed cotton samples.

Enzyme used	Fabric					
	Linen		Linen/polyester		Polyester	
	T.S. (kg/5 cm)	% decrease in T.S.	T.S. (kg/5 cm)	% decrease in T.S.	T.S. (kg/5 cm)	% decrease in T.S.
Untreated	90	–	52	–	130	–
Brewer's yeast	89	1.12	50	3.84	80	38.46
Valumax A356	88	2.22	48	7.69	130	–
Valumax A828	87	3.33	42	19.23	100	23.07

Table 5
Fastness properties of untreated and treated printed fabrics.

Enzyme used	Fabric	Colour fastness							
		Rubbing		Washing		Perspiration			
		Dry	Wet	St.	Alt.	Alkali		Acidic	
						St.	Alt.	St.	Alt.
Untreated	Linen	3	2-3	2-3	2-3	2-3	2-3	2-3	2-3
	Linen/polyester blend	4	4	4	4	4	4	4	4
	Polyester	4	4	4-5	4-5	4-5	4-5	4-5	4-5
Brewer's yeast filtrate	Linen	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Linen/polyester blend	4	3	4	4	4	4	4	4
	Polyester	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Valumax A356	Linen	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	Linen/polyester blend	4	3	4	4	4	4	4	4
	Polyester	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Valumax A828	Linen	4-5	4	4	4	4	4	4	4
	Linen/polyester blend	4-5	4-5	4	4	4-5	4	4	4
	Polyester	3	3	3-4	3-4	3	3	4	4

absorbent properties of regular polyester fabric. Otherwise cellulase enzyme Valumax A828 (2000 ECU/g; Cellulase Unit per gram) cause slight initial degradation appears as cracks cause increase in the %K/S by 105.57 while Valumax A365 (120 CVU/g) has shown almost no effect on the polyester morphology as it deposit on the fibre surface as shown in Fig. 4D (Li, Frey, & Browning, 2010).

3.5. Effect of enzymatic printing on tensile strength of cotton fabric

It is of great interest to investigate the influence of the enzymes on the tensile strength of the used fabrics. To achieve this, white printing paste containing the optimum concentration of the enzymes and adjusted at the optimum pH (The pH of the paste was adjusted at 4 and 10 for Brewer's yeast filtrate and the two Valumax enzymes relatively) of each were prepared and used in printing the samples followed by drying at optimum temperature (the optimum temperature for using Brewer's yeast filtrate was at 70 °C while for Valumax A356 and A828 was at 50 °C) and time. Another fabric samples were printed with white printing paste in the absence of the enzymes were put to undergo the same conditions as control samples. Table 4 represents the tensile strength of the printed samples as well as the original sample.

The results in Table 4 show that all % decrease in T.S. for all bio-treated fabrics are in the acceptable range and do not affect the fabric retention. The % decrease in T.S. depends on the hydrolytic effect of each enzyme and the fabric used.

3.6. Fastness properties

The results of fastness properties of the printed fabrics are listed in Table 5. The results show very good fastness to rubbing, from very good to excellent in case of perspiration and washing fastness properties; this indicates that the prints have some improvement after enzymatic treatment.

4. Conclusion

The changes in the surface structure and morphology of the cellulosic fabrics depend mainly on kind and activity of enzymes used. The optimum condition of ink jet printing of linen, linen/polyester blend and polyester fabrics was improved using bio-treatment process before ink jet pre-treatment. This treatment was considered as a new technique while saving time and energy via, improving digital printing of all cellulosic fabrics with oil base inks. Also, colour fastness of bio-treated cellulosic printed samples was improved and tensile strength was not affected to a great extent. Bio-treatment of cellulosic linen fabric and its blend with Brewer's yeast filtrate increases the surface area for absorption of water molecules and ink trapped giving an increase in the colour strength and overall fastness properties.

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