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Synergetic effect of DC air plasma and cellulase enzyme treatment on the hydrophilicity of cotton fabric

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ABSTRACT

The effects of DC air plasma and cellulase enzyme treatments on the hydrophilicity of cotton fabric samples were investigated. Cotton fabric samples were treated with DC air plasma (P), cellulase enzyme (E), enzyme treatment preceded by plasma (PE) and plasma treatment preceded by enzyme (EP) to improve the hydrophilicity. The physico-chemical changes that occurred during the treatments were studied using dynamic wicking test, SEM, XRD, UATR-FTIR, weight loss and air permeability measurements. The UATR-FTIR results revealed that, for EP treated fabrics, there is a decrease in the intensity of C_1–O–C_4 peak (894 cm^{-1}) indicating the glycosidic bond breakage with an increase in the intensity of C=O peak (1734 cm^{-1}) due to oxidation at C_6, C_1/C_4 carbon by enzyme and plasma treatments. SEM and XRD results reveal that, plasma and enzyme treatment leads to surface etching, removing the non-cellulosic compounds thereby increasing the hydrophilicity of the cotton fabrics. The synergetic effect of plasma and enzyme treatments on physical and chemical properties of the cotton fabric is studied and the results are discussed.

1. Introduction

Cotton fiber is composed of mainly cellulose, which is the most abundant, renewable and biocompatible polymer (Jan, Blackburn, Thomas, Jim, & Patrick, 2010). Cotton fiber contains not only cellulose but also non-cellulosic materials such as pectins, waxes, proteins, sugars etc., in the cuticle layer which are responsible for poor hydrophilic property. This leads to some problems in the quality of dyeing and finishing of cotton fabrics (Chinkap, Myunghee, & Eun kyung, 2004; Styliani, Diomi, Paul, & Dimistris, 2008). Conventionally, wet chemical processes are performed to remove these non-cellulosic materials present on the surface of the cotton fabric which is not environmentally favourable (Qiang, Xue-Rong, Zhao-Zhe, & Jian, 2007). Generally, plasma treatment and enzymatic scouring are found to be promising techniques when compared to alkaline scouring, because of its eco-friendliness and cost effectiveness (Poll, Schladitz, & Schreiter, 2001; Radetic et al., 2007).

Plasma surface treatment is an efficient surface engineering tool that alters the polymer surface, thereby inducing new functional groups onto the fabric with some morphological changes (Inbakumar et al., 2010; Pandiyaraj & Selvarajan, 2008). The surface of the fabric is activated in such a way that it improves the properties such as wettability, dyeability, hydrophobicity, adhesion and other finishing processes without affecting the bulk property of the fabric (Hartwig, 2002; Jie Rong, 1991; Pane, Tedesco, & Greger, 2001; Wang, Liu, Xu, Ren, & Qiu, 2008).

Enzyme treatment of cotton fabrics provides considerable advantages in terms of soft touch, surface luster, improved wettability and dyeability without appreciable fiber deterioration (Qiang et al., 2007). The enzymatic hydrolysis is a catalytic action, which induces a chemical change, thereby removing the non-cellulosic impurities responsible for the hydrophobicity of cotton fabrics. Currently cellulase is widely used to alter cellulose properties for potential applications in textile, pulp and paper industries (Yu & Huimin, 2002).

Many studies were done on the plasma surface modification and enzyme treatment of the cotton fabrics; however, there are only a few reports about the synergetic effect of two treatments and the study of physico-chemical properties of such modified cotton fabrics. The aim of the present study is to investigate extensively, the effects of plasma and enzyme treatment and their combinations on the chemical and physical properties of cotton—cellulose. In this paper, pre- and post-plasma treatment for enzyme treated fabrics are, hereafter, referred to as PE and EP respectively.

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2. Materials and methods

2.1. Materials

The fabric used in the present study was a finished, plain weave, 20 s count, 100% pure cotton fabric (10 cm × 10 cm) with an EPI/PPI count of 70/50. Cotton fabrics were initially immersed in boiling water at 90 °C for 15 min and dried. Commercially available acid cellulase Bio-Lish 2.0 (Avensa chemicals, India) was used for enzyme treatment.

2.2. Plasma treatment

A 12″ DC plasma chamber was used for plasma treatment. The upper electrode (cathode) diameter is 5 cm. Cotton fabric was placed on the substrate holder, acting as anode. Prior to the treatment, the chamber was evacuated to a base pressure of 10^{-3} mbar using a rotary pump. Atmospheric air, used as working gas, was admitted into the chamber by means of a needle valve. The optimized process parameters used in the study for the fabric samples to attain maximum hydrophilicity are as follows: gas pressure – 0.2 mbar, inter-electrode distance – 3 cm, exposure time – 5 min and current – 60 mA.

2.3. Cellulase enzyme treatment

The samples were subjected to cellulase enzyme treatment in an incubator shaker with a material to liquid ratio (MLR) of 1:20.The treatment was carried out for 30 min at a temperature of 55 °C with a pH of 5.5 using an enzyme dosage of 2% OWF (on weight of fiber). Subsequently, cellulase enzyme was de-activated by rinsing the fabrics thoroughly with hot and cold water (80 °C) for 10 min duration and then dried in air.

2.4. Assessment of hydrophilicity

The hydrophilicity of treated and untreated fabrics was analyzed using dynamic wicking test (BS 4554). In this test, a strip of the fabric with a dimension of 0.5 cm × 8 cm was suspended vertically with its lower edge having contact with distilled water. The rise in the height of water was measured for a period of 20, 40, 60, 120, 180 and 240 s. The wicking height measured for 240 s was considered for assessment of hydrophilicity of the treated fabric (Ferrero, 2003). The capillary rise measurements were made thrice for each sample and the average values are reported with a standard error of ±0.1 cm.

2.5. Assessment of mean pore radius

The mean pore radius on the surface of the fabric was calculated as per Lucas Washburn equation (Pandiyaraj & Selvarajan, 2008). The modified equation is stated as,

$$H^2 = (R\gamma/2\eta)t \quad (1)$$

where H – wicking height, R – pore radius, η – coefficient of viscosity of the liquid, γ – surface tension of the liquid, t – time taken for wicking.

The slope of the plot between H^2 and t gives the mean pore radius of fabrics.

2.6. Weight loss

The cotton fabrics before treatment are reported to have impurities like, pectin, wax, oil and dust particles on the fabric surface (Styliani et al., 2008). However, plasma treatment etches the surface of fabric and induces a chemical change in it by removing the surface contaminants (Pandiyaraj & Selvarajan, 2008). Enzyme treatment also aids in the removal of pectin and waxes present on the fabric surface. The removal of these impurities in the fabric samples due to plasma and enzyme treatments can be determined by percentage of weight loss. Taking this into consideration, the weight loss of the fabrics was calculated by measuring the dry weight of fabrics before and after the treatments. The percentage weight loss was calculated using the following equation:

$$\%\,\text{Weight loss} = [(W_1 - W_2)/W_1] \times 100 \quad (2)$$

where W_1 – weight of the fabric before treatment, W_2 – weight of the fabric after treatment.

2.7. UATR-FTIR analysis

The chemical changes that occurred during the DC air plasma and cellulase enzyme treatments were analyzed by UATR-FTIR (Universal Attenuated Total Internal Reflection Fourier Transformation Infrared) spectra recorded using a Perkin Elmer (Spectrum100) FTIR spectrometer in the range of 4000–650 cm^{-1} with a resolution of 4 cm^{-1}. Cotton fabrics of dimensions 10 mm × 10 mm were placed onto the Zn–Se single crystal and pressure was applied to ensure a good contact between the sample and crystal to prevent the loss of IR radiation. The spectra were obtained for each sample at a resolution of 4 cm^{-1} with 32 scans.

2.8. Structure and surface morphology studies

An X-ray diffractometer (Bruker D8 advance) was used to determine the crystalline nature of cotton fabrics, using copper Kα radiation (λ = 1.54 Å). The angles scanned were 10–30° at 0.01°/s. The surface morphology of untreated and treated cotton samples was studied using SEM (JEOL-JSAL 6360). Platinum was sputtered on to the fabric samples, as a conducting material to analyze the sample.

3. Results and discussion

3.1. Hydrophilicity of DC air plasma and cellulase enzyme treated cotton fabrics

Hydrophilicity of the untreated and treated fabric samples was assessed using dynamic wicking test and was measured in terms of wicking height. The results revealed that the samples treated with DC air plasma show a higher wicking height when compared with untreated fabrics as shown in Fig. 1. This may be due to two factors: (i) physical changes due to the etching effect of plasma species on the cotton fabrics and (ii) chemical changes due to the formation of polar groups and free radicals on the fabric surface (Inbakumar et al., 2010; Vaideki, Jayakumar, & Rajendran, 2009; Ward, Jung, Hinojosa, & Benerito, 1979).

The PE treated fabrics show an increase in wicking height of 4.2 cm for a wicking time of 4 min, when compared with control (2.9 cm), plasma (3.4 cm) and enzyme (3.7 cm) treated fabrics. The increase in wicking height is due to the etching effect of plasma pre-treatment on the enzymatic hydrolysis of cotton fabrics. The reactivity of the enzyme is enhanced by plasma pre-treatment, thus removing the non-cellulosic impurities, making the fabric more hydrophilic as reported by many authors (Radetic et al., 2007; Wong, Tao, Yuen, & Yeung,, 2000; Nam Sik, Yong Jin, Mitsuru, & Toru, 1996).

EP treated fabric show a remarkable increase in wicking height of 5 cm for a wicking time of 4 min, when compared with other fabrics as shown in Fig. 1. It is found that cellulase enzymatic pretreatment enhances both the removal and the degradation of seed

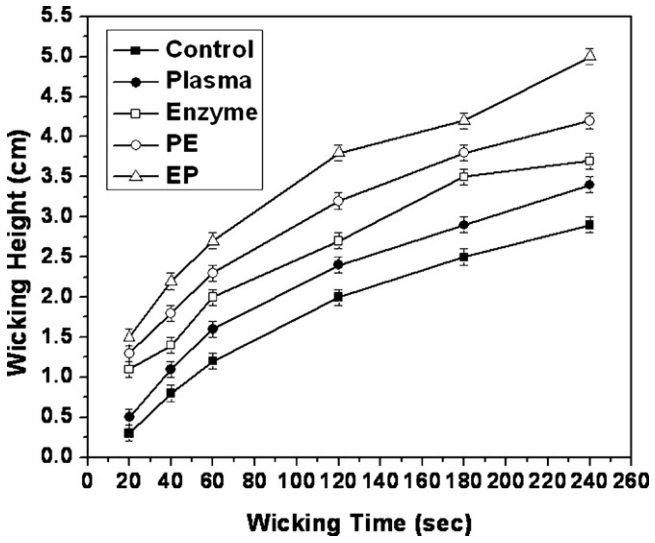

Fig. 1. Wicking height of untreated (control), DC air plasma (P), enzyme (E), PE, EP treated cotton fabrics.

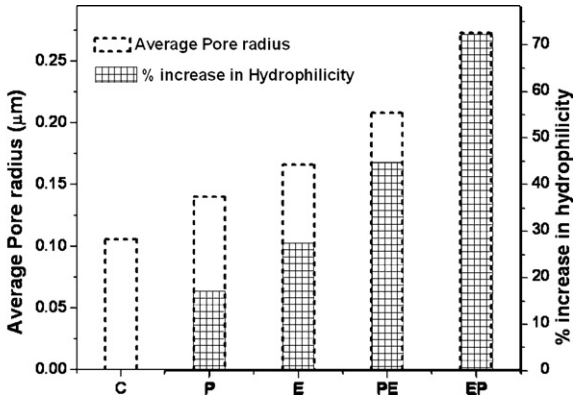

Fig. 2. Bar diagram for percentage increase in hydrophilicity and mean pore radius of untreated and treated fabrics.

coat fragment impurities present on the cotton fabric surface. These results indicate that there is a removal of small free end surface fibrils and other impurities, increasing the accessible surface area on the fabric by enzyme hydrolysis (Csiszar, Urbanszki, & Szakacs, 2001). Later, when the fabrics are treated with plasma, it further modifies the surface chemistry and morphology inducing the formation of polar groups and free radicals on the surface of the fabric making it more hydrophilic. Thus, enzyme pre-treatment has facilitated the plasma species to react with the fabric surface by removing non-cellulosic components. The percentage increase in hydrophilicity and mean pore radius of all samples were also calculated and represented in Fig. 2. It is evident from the figure that, in EP treated fabrics, there is 72% increase in hydrophilicity when compared with the untreated one.

The mean pore radius calculated using Lucas Washburn equation, was found to increase in the following order: untreated (C) < plasma (P) < enzyme (E) < PE < EP treated cotton fabrics as

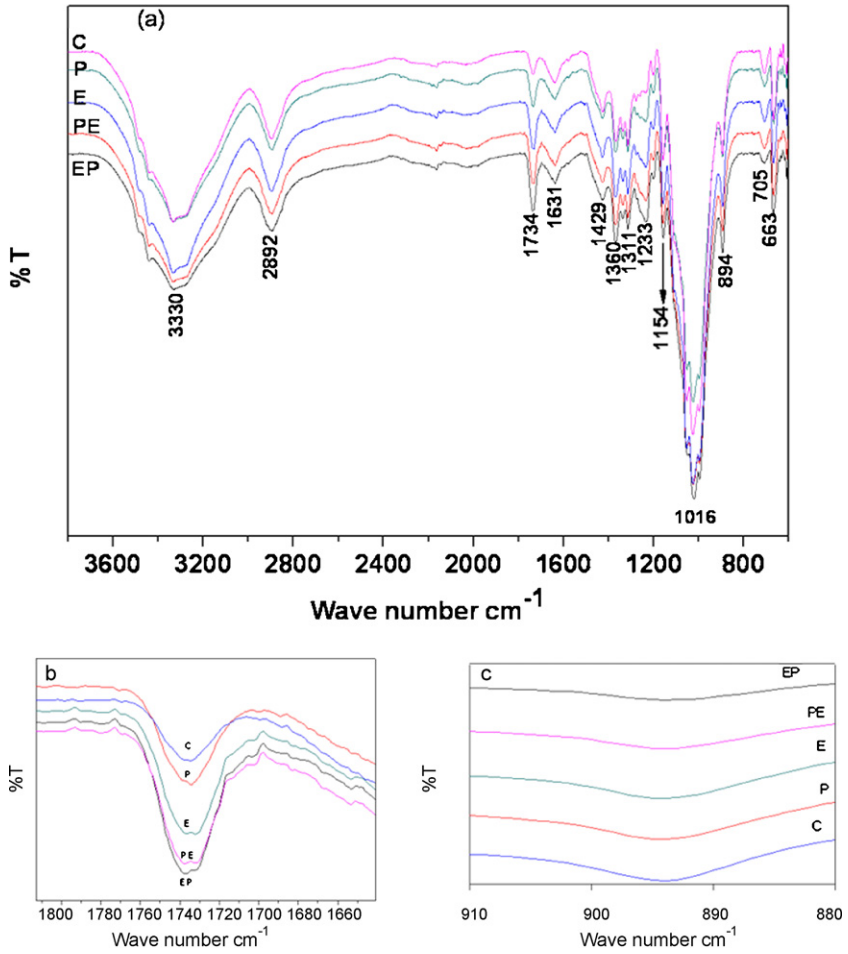

Fig. 3. UATR-FTIR spectra of control (C), DC air plasma (P), cellulase enzyme (E), PE, EP treated cotton fabrics.

Table 1
Infrared absorption frequencies of untreated and treated cotton fabrics (cellulose).

Literature (cm^{-1})	Experimental peaks obtained (cm^{-1})		Peak characteristics
	Untreated cotton fabric	Treated cotton fabrics	
3570–3200	3330	3330	H– bonded OH stretch
3000–2800	2892	2892	C–H stretching
1728	1734	1734	C=O stretch in –COOH
1650–1633	1631	1631	Adsorbed H_2O
1430	1429	1429	CH wagging (in-plane bending)
1372	1360	1360	CH bending (deformation stretch)
1336	1331	1331	O–H in-plane bending
1320	1311	1311	CH wagging
1236	1233	1233	OH in plane bending
1130	1154	1154	Asym. Bridge C_5–O–C_1
1042	1016	1016	Asym. In-plane ring stretch
898	894	894	Asym. out-of-phase ring stretch: C_1–O–C_4; β glucosidic bond

shown in Fig. 2. EP treated cotton fabrics show the highest value of mean pore radius (0.273 μm); this confirms the removal of impurities on the surface by enzyme and thereby exposing the clean fabric surface to plasma. This implies that enzyme pre-treatment modifies cellulose in such a way so as to make it more susceptible to plasma, increasing the pore radius. The increase in hydrophilicity is accompanied by the increase in pore radius because the pores provide paths for water to diffuse into fabric surface.

3.2. UATR-FTIR analysis

Fig. 3a shows the UATR-FTIR spectra of the DC air plasma (P), cellulase enzyme (E), PE and EP treated cotton fabrics. The characteristic cellulose peaks in the spectra is compared with the literature (Chinkap et al., 2004; Jan et al., 2010) and reported in Table 1. The spectra reveal the presence of all the peaks corresponding to various functional groups both in the untreated (control) and the treated fabrics. According to Beer's law (Brian, 1999), the concentration of the functional groups is proportional to the intensity of the corresponding absorption peaks.

Primarily, the emphasis is about the changes in the peak 1734 cm^{-1} corresponding to C=O stretching in COOH group, after plasma and enzyme treatments (enlarged Fig. 3b). It is evident from Fig. 3b that, the intensity of 1734 cm^{-1} peak is increasing for the samples in the order: Control < Plasma < Enzyme < PE < EP. This indicates that the concentration of C=O group in COOH has increased for the samples treated with plasma, enzyme and their combinations of treatment. This increase in the carbonyl group on the fabric surface has enhanced the hydrophilic property of the fabric in addition to the physical changes. On the other hand, absorption around 894 cm^{-1} confirms the presence of C_1–O–C_4 glycosidic bond in all the treated fabrics, but the intensity of the peak is found to be reducing for plasma and enzyme treated fabrics when compared to untreated fabric (Fig. 3c).

In plasma treated cotton fabric, the increase in concentration of carbonyl group (1734 cm^{-1}) may be attributed to the dehydrogenation at C_6 carbon and oxidation of primary alcohol leading to the formation of –COOH on the fabric surface (Vaideki et al., 2009). The slight decrease in the concentration of C–O–C group is due to the cleavage at ether linkage followed by oxidation which would have contributed to the increase in carbonyl group.

In enzyme treated fabrics, there is a decrease in the intensity of C_1–O–C_4 group, due to cellulase enzyme hydrolysis of β (1–4) glucosidic linkages in cellulose. The increase in intensity of C=O group (1734 cm^{-1}) in enzyme treated fabric may be attributed to the oxidation of C_1/C_4 carbon on the fabric surface after enzyme hydrolysis. This may be attributed to the acidic pH (5.5) condition prevailing during the enzyme treatment which would have facilitated a basic reaction by subsequent oxidation of OH groups at C_1 carbon in cellulose forming –COOH group. The presence of 894 cm^{-1} peak in enzyme treated fabric indicates that the enzyme has not hydrolyzed the cotton fabric (cellulose) completely to glucose. The increase in carbonyl group content with decrease in glycosidic linkage confirms the cleavage of C_1–O–C_4 bond by cellulase, followed by oxidation at C_1/C_4 carbon due to acidic pH condition resulting in –COOH group formation. Thus enzyme and plasma treatments increase the polar groups on the fabric surface thereby increasing the hydrophilicity of the fabric. In PE and EP treated fabrics, the synergetic effect of

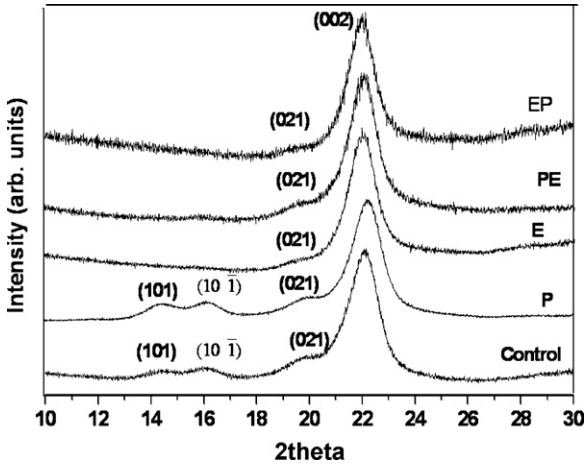

Fig. 4. XRD spectra of the untreated (control), DC air plasma (P), enzyme (E), PE, EP treated cotton fabrics.

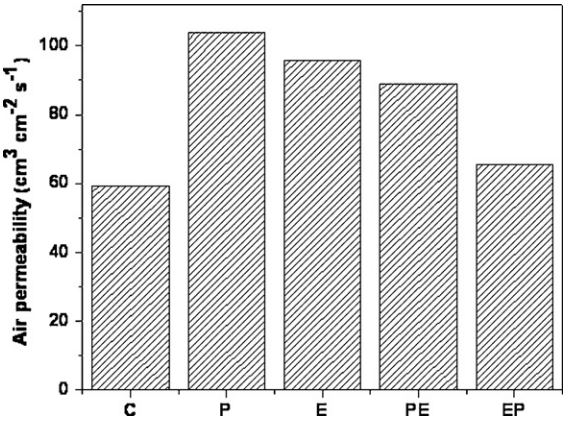

Fig. 5. Effect of plasma (P), enzyme (E), PE, EP treatment on the air permeability of the cotton fabric.

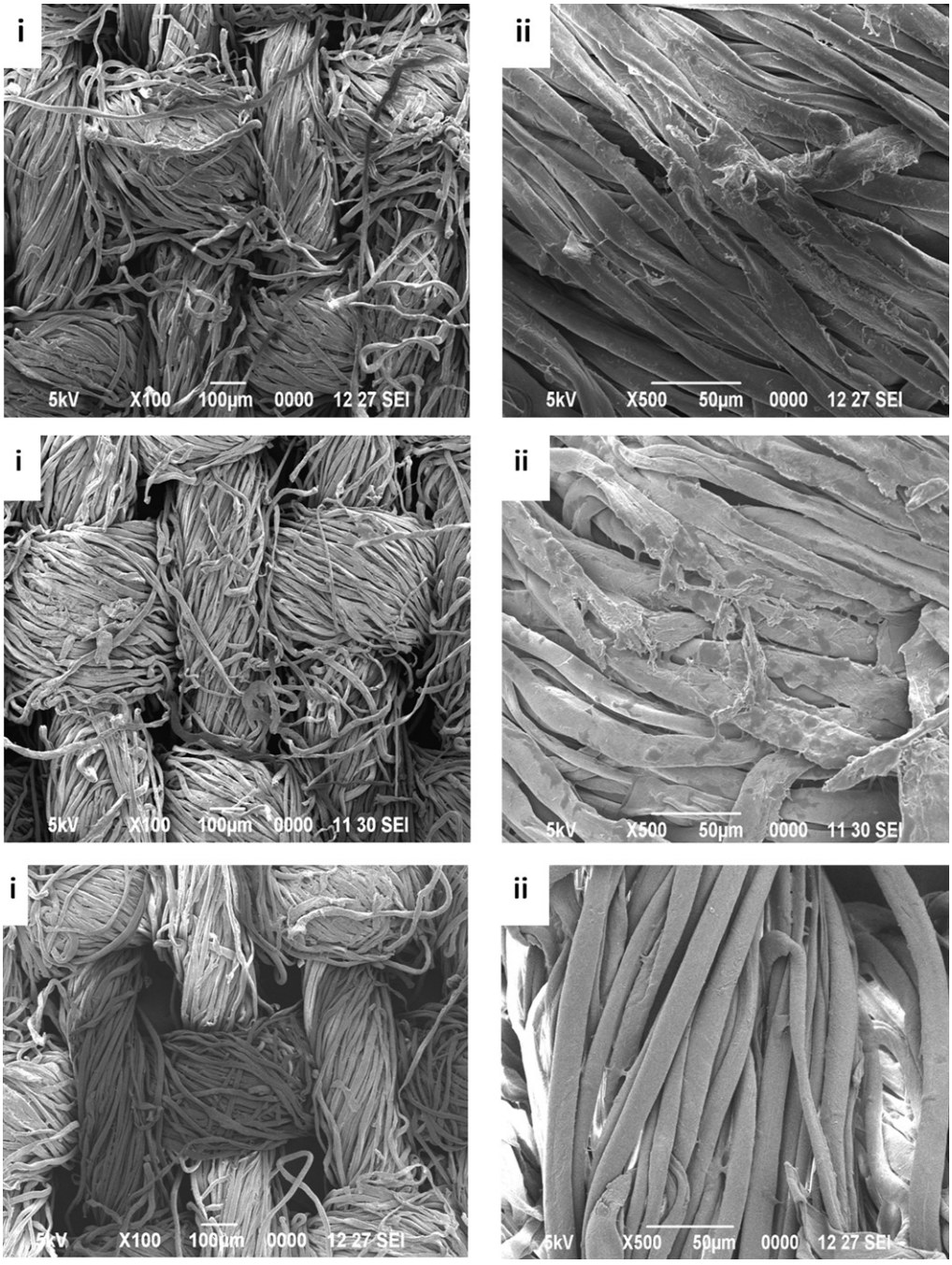

Fig. 6. (a) SEM images of untreated cotton fabric (i) warp and weft (ii) fibers. (b) SEM images of DC air plasma treated cotton fabric (i) warp and weft (ii) fibers. (c) SEM images of enzyme treated cotton fabric (i) warp and weft (ii) fibers. (d) SEM images of PE treated cotton fabric (i) warp and weft (ii) fibers. (e) SEM images of EP treated cotton fabric (i) warp and weft (ii) fibers.

plasma and enzyme treatments has increased the concentration of the carbonyl group further which is evident from the spectra. It is clear from the FTIR and dynamic wicking test results that, increase in hydrophilicity may be attributed to the chemical modification of the fabric surface by enzyme and plasma treatments.

3.3. Crystalline nature of cotton fabrics—XRD analysis

XRD spectra of the treated and untreated cotton fabrics are shown in Fig. 4.The peaks are indexed with standard JCPDS data. The presence of most prominent peaks along (0 0 2), (0 2 1), (1 0 1) and (1 0 $\bar{1}$) direction in the spectra is in accordance with the standard results reported for the native cellulose (Segal, Creely, Martin, & Conrad, 1959; Yu & Huimin, 2002). The control and plasma treated cotton fabrics reveal the amorphous regions along (1 0 1) and (1 0 $\bar{1}$) directions (Segal et al., 1959; Yu & Huimin, 2002). XRD shows that there is no change in the crystallinity for both control and plasma treated cotton fabrics. However, the absence of broad peaks along (1 0 1) and (1 0 $\bar{1}$) directions reveals the removal of amorphous contents such as pectins, waxes and non-cellulosic compounds from the surface of fabric. It is evident that when the fabrics are treated with enzyme there is an increase in the crystallinity of the fabric at the expense of the impurities. Also, from the spectra it is clear that plasma treatment has no influence on the crystalline nature of the cotton fabrics.

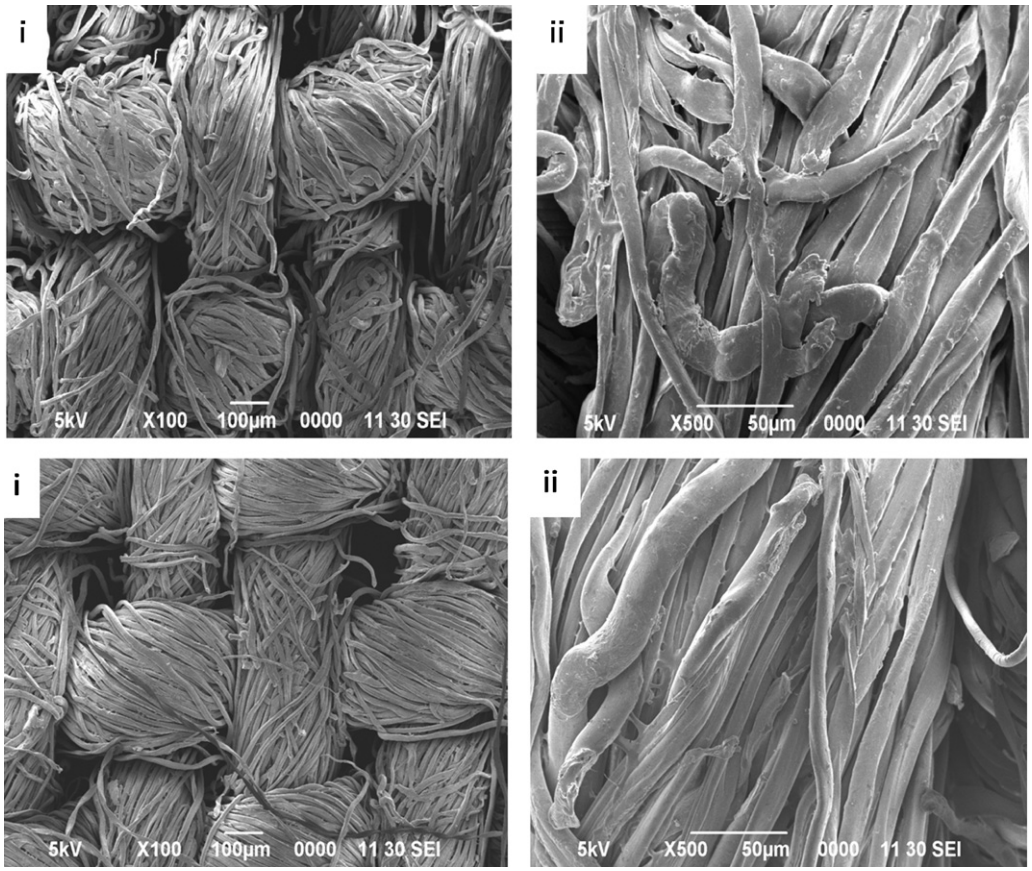

Fig. 6. (*Continued*).

3.4. Air permeability and weight loss of cotton fabrics—SEM analysis

The air permeability of the treated fabrics was ascertained by Kawabata evaluation system (KES) and is reported in Fig. 5. It is evident from Fig. 5, that the air permeability increases for the treated samples when compared with the control fabric. Fig. 6a shows the SEM micrograph of untreated (control) fabric. The SEM micrographs of DC air plasma, enzyme, PE and EP treated cotton fabrics are shown in Fig. 6b–e respectively. SEM micrographs also reveal that the increase in interstitial pore size has enhanced the air permeability. SEM micrographs (Fig. 6b) confirm that the DC air plasma treatment has etched the hair like protrudants present on the surface of the fabric which resulted in the increase of interstitial pore size, thus increasing the air permeability of the fabric as 103.79 $cm^3\ cm^{-2}\ s^{-1}$. In the case of the enzyme treated fabrics (Fig. 6c), it was found to be smoother, which revealed that the surface fibrils and other impurities had been removed, thereby increasing the air permeability to a value of 95.8 $cm^3\ cm^{-2}\ s^{-1}$,when compared with untreated fabric (59.3 $cm^3\ cm^{-2}\ s^{-1}$). The PE treated samples (Fig. 6d) show smoother surface with an increase in the interstitial pore size, when compared with the untreated cotton fabric. The air permeability of EP treated samples was found to be 65.55 $cm^3\ cm^{-2}\ s^{-1}$, which is slightly higher than the control fabric (59.3 $cm^3 cm^{-2}\ s^{-1}$). This may be due to the smoothening and etching on the surface due to the effect of enzyme and plasma (Fig. 6e) respectively. The percentage weight loss of the treated fabrics was calculated and the results are shown in Fig. 7. The etching effect of plasma and the removal of surface fibrils by enzyme treatment resulted in a weight loss of 1% for enzyme, 0.4% for plasma and 1.5% for EP treated fabrics. In the case of PE treated samples, micro pores and cracks introduced by plasma pretreatment enhanced the penetration and reaction of enzymes with fiber resulting in higher weight loss of 2.5% when compared with other treatments. The similar trend of weight loss was observed by Nam Sik et al. (1996) for plasma pre-treated fabrics with subsequent enzyme treatment.

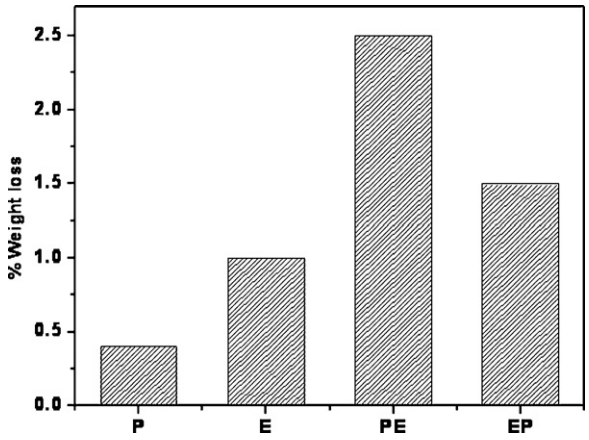

Fig. 7. Percentage weight loss in the cotton fabric due to plasma (P), enzyme (E), PE, EP treatment.

4. Conclusions

DC air plasma and cellulase enzyme treatments were found to be effective in improving the hydrophilicity of cotton fabrics. The combination of treatments has resulted in enhanced hydrophilicity when compared to plasma (P) and enzyme (E) treated fabrics. In particular, EP treated fabrics shows a drastic increase in

hydrophilicity of nearly 70%. This increase in hydrophilicity is attributed to the physico- chemical changes due to plasma and enzyme.

UATR-FTIR results shows the presence of polar groups C=O, OH, COOH on the fabric surface and the concentration of C=O group was found to be higher for those fabrics treated with EP. The formation of polar groups on the fabric surface is one of the important factors responsible for the increase in hydrophilicity.

The physical etching of the fabric surface is the other factor, leading to an increase in mean pore radius, enhancing the water retention property of cotton fabric. The EP treated cotton fabric samples exhibit effective surface modification and highest value of mean pore radius due to enzyme followed by plasma treatment. SEM micrographs reveal etching of the fabric due to plasma treatment and smoothening due to enzyme treatment which can be attributed to the surface modification in the treated fabric. The interstitial pore size of the fabric matrix was found to increase with plasma and enzyme treatment, resulting in higher air permeability when compared to the untreated fabric.

Further, XRD results substantiate the etching effect in cotton fabrics by the removal of amorphous regions due to enzyme treatment. The presence of amorphous regions in the plasma (P) treated fabrics along (1 0 1) and ($1\ \ 0\ \ \bar{1}$) directions reveal that there is no change in crystallinity by plasma treatment, and it is only due to the enzyme hydrolysis. It is concluded that EP treatment is the most promising treatment for the improvement of hydrophilicity and it can be exploited for further finishing processes.

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