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Hydrophilic modification of polyester fabric by applying nanocrystalline cellulose containing surface finish

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ABSTRACT

In this study, polyethylene terephthalate (PET) fabric was modified by applying a hydrophilic surface finishing agent that contains nanocrystalline cellulose (NCC). To impart superior hydrophilicity, NCC was further cationically modified through quaternization by grafting glycidyl tri-methyl ammonium chloride (GTMAC). A textile binder, PrintRite595®, was added to the finishing system. The surface finish was applied on the fabric using a rolling–drying–curing process. The modified fabric was characterized in terms of coating durability, moisture regain, and wettability. The durability of the surface finish was tested by six repeated washing steps. The surface properties of the fabric changed from hydrophobic to hydrophilic after heat treatment with the NCC-containing surface finishing agent. The results from the washing fastness, SEM, FTIR, and EDX analyses confirmed that the cationic NCC-containing textile surface finish showed superior adhesion onto the cationic dyeable (anionic) PET surface over the unmodified NCC. Furthermore, the cationic textile surface finish was capable of withstanding multiple washing cycles.

1. Introduction

Polyester is one of the fastest growing synthetic fibers in the textile industry due to its excellent properties, such as excellent wash and wear, good dimensional stability, anti-wrinkle, and quick drying properties. However, 100% polyester has an extremely poor moisture management (transport of moisture vapor and liquid away from the body) property, as compared to natural fibers, such as cotton. Polyester has a moisture regain of only 0.42% (measured at a relative humidity of 65% and 20 °C for 24 h) (Alakara, Karakisla, & Sacak, 2008), while cotton has a moisture regain of about 8.5% (Su, Fang, Chen, & Wu, 2007). The poor water and moisture absorption of PET make it less desirable material in many textile applications, such as sportswear, under garment, furniture, and bedding.

The origin of hydrophobicity of polyester is the lack of polar groups (—OH, COOH, —NH_2, etc.) on its polymer backbone. When one perspires, the polyester tends to keep the perspiration trapped against the body. Due to the hydrophobicity, polyester is also more electrostatic compared to the natural fibers. Electrostatic nature of the polyester is highly undesirable as it causes clothes to cling to each other.

Surface modifications using various methods have been performed to improve the water and moisture absorption properties of polyester fabric (Alakara et al., 2008; He, Gu, & Tian, 2003; Kim & Song, 2006, 2008; Lee et al., 2007; Lee & Song, 2010; Parvinzadeh & Ebrahimi, 2011; Ploymalee, Charuchinda, & Srikulkit, 2010; Siriviriyanun, O'Rear, & Yanumet, 2007). The Denier reduction by aminolysis or hydrolysis is a widely used technique for improving the hydrophilicity of polyester (Bech, Meylheuc, Lepoittevin, & Roger, 2007; Bide et al., 2003; Croll, O'Connor, Stevens, & Cooper-White, 2004; Elsaeed & Farag, 2009; Hall & Whinfield, 1952; Latta & Pensa, 1976; Mangovska, BogoevaGaceva, & Pohlers, 1996). This method involves polymer chain scission at the ester linkage and produces water compatible functional groups, such as —COOH, —OH, —NH_2 on the polyester fiber surface. However, the denier reduction suffers from the loss of mechanical strength of the fabric and also produces waste in the effluent, therefore increasing the cost for textile effluent treatment. Multi-component and composite fibers also attracted significant attention to improve the hydrophilicity of the polyester (Backwell, 1997; Bolon & Boldebuck, 1983; Mathes, Lange, & Gerlach, 1983; Su et al., 2007). However, the production of this kind of fibers needs a sophisticated technology

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(Ploymalee et al., 2010). Several studies have also been reported in the literature on the hydrophilic improvement of polyester through co-polymerization and graft co-polymerization (Alakara et al., 2008; Maity, Kartha, & Srivastava, 1984) of hydrophilic monomers in the presence of an initiator. However, considering the high cost and lengthy processing time, such treatments are not commercially viable for applying to polyester fabrics in the textile industry. In recent years, application of plasma process for the modification of textile fabrics has received attention (Morent et al., 2008), which has the advantage of being an environmental friendly technique. However, this is a sophisticated technology and selection of the plasma source and treatment conditions are crucial for the non-destructive treatment of the fabric.

The application of surface finish is a commercially viable alternative to improve the hydrophilic properties of polyester fabric. Significant amount of research has been conducted on the development of smart textiles by applying surface finish, such as (1) antimicrobial fabric (El-Rafie, Mohamed, Shaheen, & Hebeish, 2010; Kim, Choi, & Yoon, 1998), (2) ammonia decomposing fabric (Dong, Bai, Liu, & Zhu, 2007), fragrance-containing textile (Sohn et al., 2007; Voncina, Vivod, & Chen, 2009), UV-protective fabric (Zohdy, El Hossamy, El-Naggar, Fathalla, & Ali, 2009), etc. Several studies have been reported in the literature on the application of surface finish to improve the hydrophilicity of polyester fabrics (Adler & Walsh, 1984; Eom, 2001; Ferguson, 1982; Ploymalee et al., 2010; Xiao, Chen, Wei, & Wu, 2009). The surface finishing agent usually contains hydrophilic polymer or fluorinated compounds. The benefit of this technology is that it can be easily adapted to any existing textile facility at a relatively low cost.

Nanocrystalline cellulose (NCC) has received much attention recently (Araki, Wada, Kuga, & Okano, 1998; Beck-Candanedo, Roman, & Gray, 2005; Cranston & Gray, 2006; Jahan, Saeed, He, & Ni, 2011), due to many unique characteristics, including: (1) high strength, Young's modulus and tensile strength of NCC can be up to 145 GPa and 7.5 GPa, respectively (Lahiji, Reifenberger, Raman, Rudie, & Moon, 2008); (2) high aspect ratio (up to 100) (Cranston & Gray, 2006); (3) high specific surface area (up to several hundred m^2/g) (Chazeau, Cavaille, Canova, Dendievel, & Boutherin, 1999); (4) polar groups (—OH) enriched surface, which provide excellent means of moisture adsorption and surface reactivity. The above unique characteristics give them improved material properties, therefore, a large variety of applications in various industries, such as pulp and paper, textile, biomedical, personal care products, plastics, electronics.

In this study, the authors used cationically modified nanocrystalline cellulose (NCC), which was prepared based on an early procedure (Zaman, Xiao, Chibante, & Ni, 2012), as part of the surface treatment agents, and applied to the plain weaved polyester fabric based on a rolling–drying–curing process. The hypothesis is that the surface finishing system consists of cationic NCC would render superior hydrophilicity and affinity toward the cationic dyeable polyester fabric (anionic PET). The goal of this study was to develop a durable hydrophilic surface finishing system to improve the hydrophilic properties of polyester.

2. Experimental

2.1. Materials

100% (cationic dyeable) PET fabric (style#763), which was plain weaved, with the fabric weight of 163 g/m^2, was provided by Testfabrics, Inc., USA. A commercial textile binder, PrintRite595®, was obtained from Lubrizol, Inc., USA. A textile thicker (SC6477) was provided by StanChem, Inc., USA. NCC sample, prepared from the sulfuric acid hydrolysis process, was obtained from FPInnovations, Canada. GTMAC was used as a cationization agent and obtained from Sigma–Aldrich, Canada. Potassium poly vinyl sulfate (PVSK) with a MW of 100,000–200,000, 97.7% esterified, was provided by Wako Pure Chem. Ltd. Japan. Cellulose dialysis membrane (MW cut-off 12,400) was obtained from Sigma–Aldrich. Ethanol, sodium hydroxide were all analytical grade chemicals from Fisher Scientific Co., Canada.

2.2. Preparation of cationic NCC

A water based semi-dry process was used for the cationic modification of NCC (Fatehi, Ates, Ward, Ni, & Xiao, 2009; Liu, Ni, Fatehi, & Saeed, 2011; Zaman et al., 2012), which is given as follows: 1 g of water dispersible freeze dried NCC and 50 mg of powdered sodium hydroxide were mixed thoroughly using a mortar and pestle for 5 min at room temperature and left to cool to room temperature. The solid mixture was then transferred to a polyethylene bag followed by addition of the water. The cationization agent (GTMAC) was added dropwise to the previous mixture and mixed thoroughly by hand kneading. Subsequently, the reaction mixture was kept in a thermostated ultrasonic water bath at 65 °C. The reaction conditions were as follows: water content of the reaction system of 36 wt%, molar ratio of GTMAC to anhydroglucose units in NCC of 3:1, catalyst (NaOH) dosage of 5% (by weight of NCC), Temperature of 65 °C, reaction time of 4 h. The reaction mixture was hand kneaded every 15 min during the reaction. After 4 h, the reaction was stopped by precipitating the reaction mixture in 95% ethanol. The unreacted reagents and by-products were removed by centrifugation. After centrifugation, the sediment was kept and the supernatant was removed and replaced by fresh 95% ethanol and recentrifuged. After that, the product was redispersed in deionized and distilled water and diluted 4 times. The suspension was then dialyzed by using cellulose dialysis membrane against deionized and distilled water for 3 days to remove the last residue of any unreacted reagents and by-products. The distilled and deionized water was replaced every 2 h during the first day and twice a day in the following 2 days. The concentration of the product was then increased by evaporation in a rotary evaporator.

2.3. Application of NCC coating on polyester fabric

Two commercial textile additives, a self-crosslinking acrylic binder, PrintRite595®, and a SC6477 thickener, were employed to form the coating formulation. SC6477 is an alkali swellable acrylic copolymer emulsion which is widely used for thickening coating formulations. Aqueous suspension of NCC, the binder (at various binder to NCC mass ratio), the thickener (binder to thickener mass ratio is equal to 3), were mixed by an electromagnetic stirrer until the coating paste attained sufficient viscosity to be applied. A small amount of alkali was also added to start the thickening mechanism. Before applying the surface finish, a 3 in. × 3 in. fabric piece was etched with alkali, washed with distilled and deionized water, and dried in an oven. The coating paste was applied onto the fabric piece with a K303 Multicoater from RK Print Coat Instruments. The speed of the coater was adjusted to 2 m/min. Coating paste was applied to both surfaces of the fabric. The net weight addition due to coating was 30% by weight of dried fabric. After applying the coating, the fabric was dried at 80 °C for 20 min and cured at 160 °C for 15 min.

2.4. Characterization of NCC-coated polyester fabric

2.4.1. Evaluation of moisture regain properties

The moisture regain is the percent weight of moisture gain compared to the initial dry fabric weight. To determine the moisture regain properties, treated fabrics were kept in a conditioned room at a relative humidity of 55 ± 5% and temperature of 23 ± 3 °C for 24 h. The moisture regain value was calculated from the difference in weight of the initial dried fabric and conditioned fabrics. Three repeated measurements were performed for each sample.

2.4.2. *Evaluation of wettability*

The wettability of the treated and untreated fabrics was evaluated by two different tests: (1) wicking ability and (2) contact angle analysis. In order to measure the wicking distance, the fabric sample was cut into 3 in. × 1 in. samples and hold at the top by a plastic tape. The bottom edge of the fabric was immersed by 2 mm in distilled water for 5 min. The distance the water had moved up was then measured. Three repeated tests were performed for each sample.

Wettability of the treated fabrics was also evaluated by measuring the water contact angle. The water contact of the untreated and treated fabric was measured using a DataPhysics PSL 250 Contact angle analyzer from Future Digital Scientific Corp., GmbH, Germany. A drop of distilled water (4 μl) was placed on the fabric surface at a rate of 0.5 μl/s using a microsyringe at room temperature. Images of the drop were recorded up to 10 s after the drop was set on the fabric surface initially. The analysis was performed on five different spots of the sample. The contact angle and percentages of water absorption were calculated by analyzing the shape of the water droplet.

2.4.3. *Evaluation of coating durability of treated polyester fabrics*

The evaluation of washing fastness of the treated fabric was performed according to the methods described in the literature (Dong et al., 2007; Kim et al., 1998; Uchida, Uyama, & Ikada, 1991). Each laundering cycle consists of one washing and two rinsing steps. The washing experiments with a commercial detergent were performed as follows. First, the treated fabrics were washed with a 0.25 wt% soap aqueous solution using a liquor ratio of 70:1 at room temperature for 5 min. Subsequently, two identical rinsing steps were performed at room temperature for 2 min. During washing and rinsing, mechanical agitation was created using Burrell Wrist Action Shaker Model 75 (Burrell Corporation, USA). Finally, the washed fabrics were dried to constant weight in an oven at a temperature of 100 °C.

2.4.4. *Surface characterization of treated polyester fabric*

The surface of the treated fabrics was characterized using scanning electron microscopy (SEM), Fourier transformed infrared spectroscopy (FTIR), and energy dispersive X-ray spectroscopy (EDX). In order to confirm the presence of cationic NCC after multiple washing, the fabric sample after washing for 3 times was used for analysis. The surface morphology of the untreated and treated fabrics was characterized using a JEOL JSM-6400 scanning electron microscope. Samples were coated with gold using an Edwards S150A sputter coater, and the SEM images were taken at an accelerating voltage of 10 kV. Samples were carbon coated using an Edwards E306A evaporative carbon coater and analyzed with an EDAX Genesis4000 EDS system under the following conditions: a beam current of 1.5 nA and an accelerating voltage of 15 kV. FTIR spectra of samples prepared as KBr pellets were obtained by using a Thermo Scientific Nicolet 6700 spectrometer in transmission mode.

3. Results and discussion

3.1. *Moisture regain properties and coating durability of treated polyester fabric*

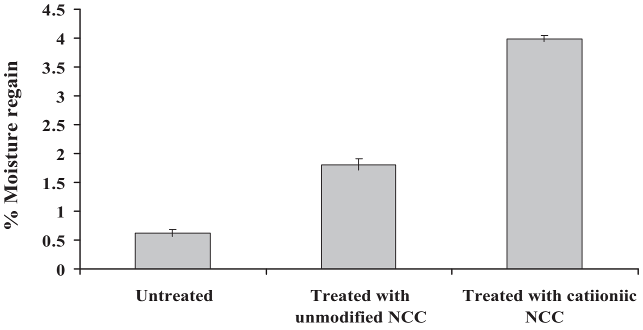

Fig. 1. Moisture regain properties of untreated and treated polyester fabrics.

Fig. 1 shows the standard moisture regain evaluation results for the untreated and treated fabrics. Untreated polyester fabric showed a moisture regain of 0.62% at 55% relative humidity and 23 °C after 24 h conditioning, which is in good agreement with the hydrophobic nature of polyester fiber. From Fig. 1, it can be seen that both original and cationic NCC treated polyester fabrics shows significant increase in moisture regain properties. The original NCC treated polyester shows a moisture regain of 1.81%, while the cationic NCC treated fabric shows a moisture regain of 3.99%. This clearly indicated that the surface of the polyester becomes hydrophilic after coating with NCC. The higher hydrophilicity of the cationic NCC treated fabric can be attributed to the presence of quaternary ammonium groups, which is well known for enhancing water uptake of polymers and resins (Cao et al., 2010; Qirong, 1992; Sosnik & Sefton, 2005). Table 1 shows the hydrophilicity improvement of polyester fabrics treated with various methods. By comparing the percentage of moisture regain of polyester fabrics treated with different methods, it can be observed that cationic NCC coating is one of the most effective surface treatment methods for improving the hydrophilicity of polyester fabric.

Fig. 2a shows the washing durability of the coating as a function of binder to NCC mass ratio in the coating formulation. As shown in Fig. 2a, when the binder to NCC mass ratio is below 0.7, there is a significant loss of the coating. Therefore, a NCC to binder ratio within the range of 0.7–1 would be more acceptable for applying this formulation on the fabric surface.

From the commercialization point of view, high washing fastness is one of the most important pre-requisites for any surface finishing agent on textile fabric. In this study, the washing fastness of NCC treated fabric for up to six consecutive laundering cycles was evaluated. Fig. 2b shows the percentages holding of coatings for the original and cationic NCC coated fabrics. It can be seen that the majority of the loss of coating occurred in the first three washing steps. This is due to the fact that unbound or loosely fixed NCC were easily removed in the first few washing steps, leaving the strongly attached NCC that were hard to remove in the following washing steps. It can be also noticed that for the original NCC containing

Table 1
Moisture regain property of polyester fabric treated with various methods.

Treatment method	Moisture regain (%)	Sources
Atmospheric air plasma treatment	6.20	Parvinzadeh and Ebrahimi (2011)
Cationic NCC coating	3.99	This study
Methacrylamide grafting	3.01	Alakara et al. (2008)
Enzymatic (lipase) hydrolysis	1–2.2	Kim and Song (2006), Kim and Song (2008) and Lee and Song (2010)
Acrylic acid grafting	2.1	He et al. (2003)
Ozone-gas treatment	0.94	Lee et al. (2007)
Poly(methyl methaacrylate) coating	0.87	Siriviriyanun et al. (2007)
PEG bisphenol A coating	0.6	Ploymalee et al. (2010)
Alkaline treatment	0.568	Kim and Song (2006)

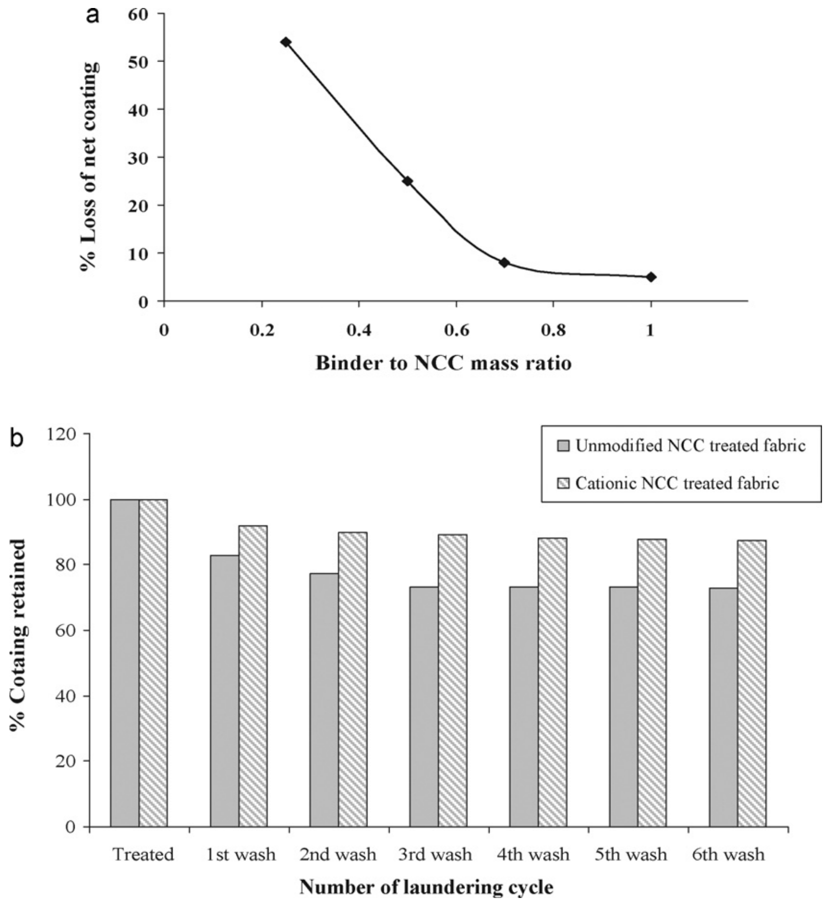

Fig. 2. (a) Percentage loss of coating after the first washing cycle as a function of the binder amount. (b) Coating durability of original and cationic NCC treated fabric after multiple washing steps.

coating after six washing cycle, only 73% of the coating remained on the fabric surface, while nearly 88% coatings remained on the fabric surface when cationic NCC was used in the coating formulation. This can be due to the fact that at high curing temperature, the cationic NCC coating formulation is more effectively penetrated into the anionic PET substrate compared to the original NCC coating formulation.

Fig. 3 shows the percentages of moisture regain of the treated fabrics as a function of the number of launderings. These results are in agreement with those from the coating durability experiments. The percentages of moisture regain decreased gradually with each washing cycle. The percentages of moisture regain of the treated fabric after six consecutive laundering cycles remained as high as 3%, which is significantly higher compared to the unmodified PET fabric.

3.2. Wetting behavior of the untreated and treated fabrics

The results from wicking test of unmodified and NCC-treated fabrics are in agreement with the percent moisture regain experiments. Fig. 4 shows the wicking distance of untreated and NCC-treated fabric before and after multiple washing. It can be

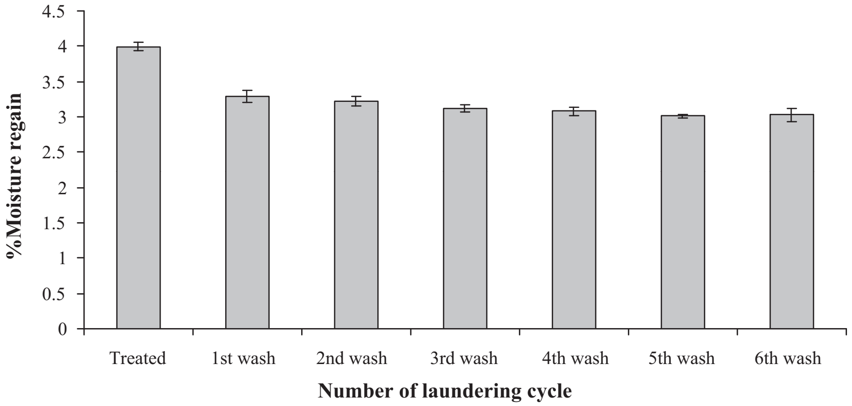

Fig. 3. Percentages of moisture regain of treated fabric as a function number of laundering cycle.

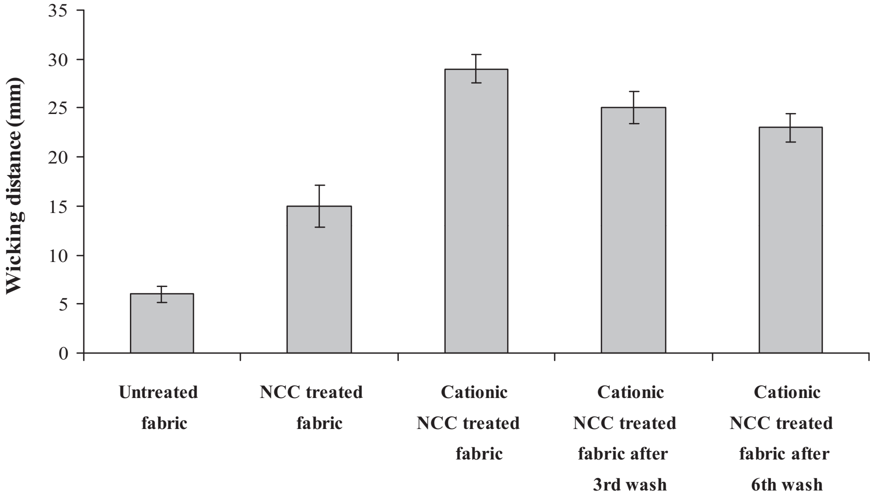

Fig. 4. Wicking distance of both untreated and NCC-treated fabric after multiple washing.

seen that the untreated fabric shows a wicking distance of 6 mm while the original and cationic NCC treated fabric showed a much higher wicking distance of 15 and 29 mm, respectively. Despite the decrease in wicking distance after multiple washing, the wicking distance of 23 mm after six washing cycles for cationic NCC treated fabric was still remarkably higher than the untreated fabric. The wicking test clearly indicated the increase in hydrophilicity of both NCC treated fabrics.

The water contact angle analysis was also performed to investigate the wetting behavior of treated fabrics. The lower the

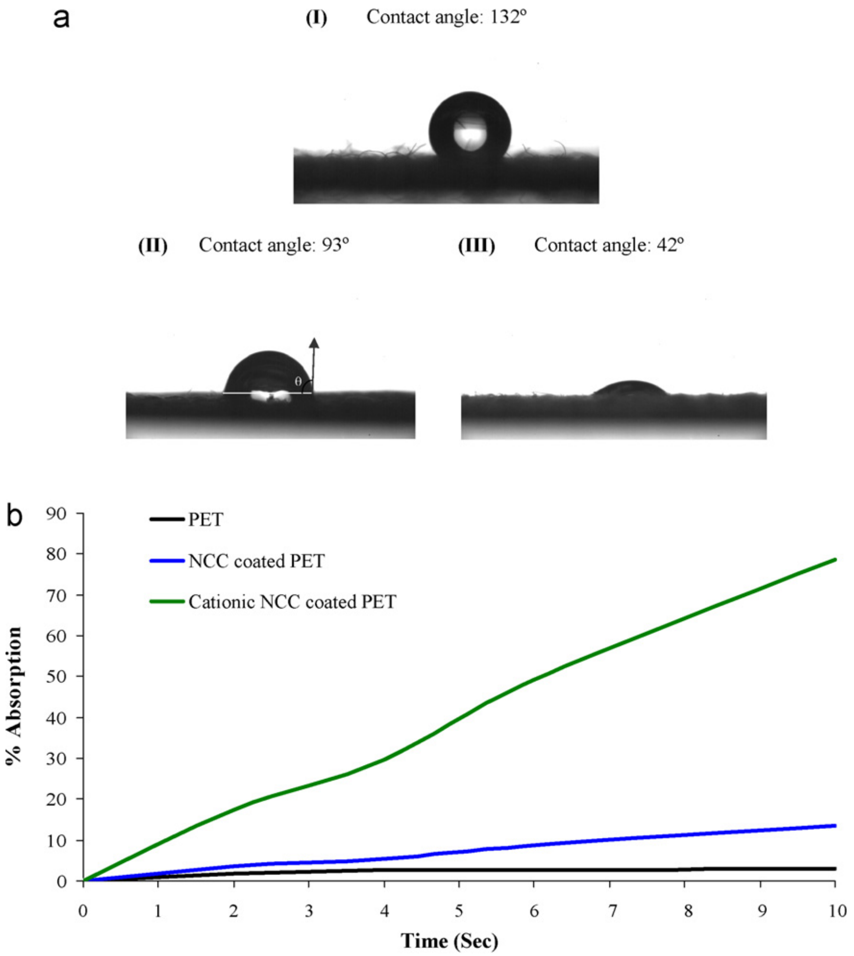

Fig. 5. (a) Water contact angle and wetting behavior: (I) untreated PET; (II) original, NCC treated PET; (III) cationic NCC treated PET. (b) Water absorption kinetics of untreated and NCC-treated PET fabrics.

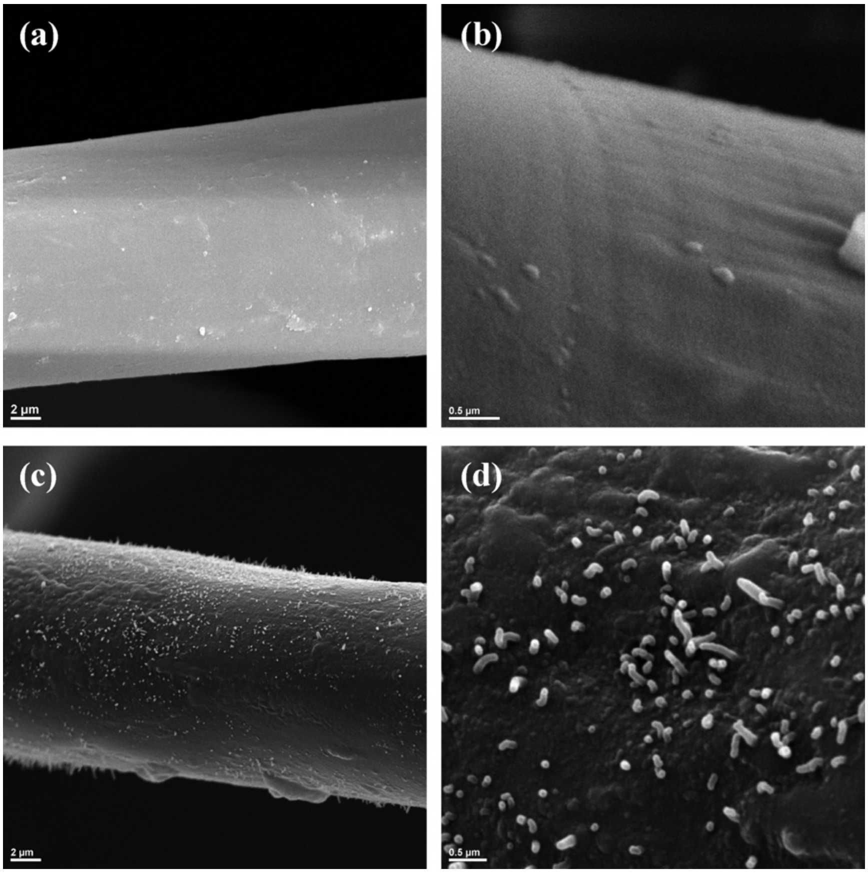

Fig. 6. SEM micrograph of untreated (panels a and b) fabric and NCC treated (panels c and d). PET fabric after three washing cycle.

water contact angle, the higher the hydrophilicity of the PET fabric. Fig. 5a shows results of the water contact angle on both untreated and NCC-treated fabric after 10 s from the initial contact.

From Fig. 5a(I), it can be seen that the water droplet on the surface of untreated PET fabric formed a contact angle of about 132°. The high value of the contact angle of the untreated PET is due to its highly hydrophobic nature. However, the NCC treated fabric showed a significant reduction in water contact angle, as shown in Fig. 5a. The increase in hydrophilicity of treated PET fabric can be attributed to the presence of hydrophilic NCC on the fabric surface. The hydroxyl group on the NCC surface favored formation of hydrogen bonds upon contact with water, which increased the hydrophilicity of the fabric. Moreover, it can be also observed from Fig. 5a(III) that the cationic NCC led to a further increase in hydrophilicity of polyester fabric by giving a contact angle of only 42°, which is significantly lower compared to the untreated and original NCC treated fabric. Fig. 5b shows the percentage absorption of water by the fabrics as a function of time. It can be seen that the cationic NCC treated fabric absorbed water more rapidly compared to the untreated and original NCC treated fabric. The presence of quaternary ammonium chloride groups in the matrix can induce an osmotic effect that led to an enhanced water absorption (Sosnik & Sefton, 2005).

3.3. Surface characterization of NCC treated PET fabric

Fig. 6 illustrates the SEM micrographs of untreated (Fig. 6a and b) and NCC treated (Fig. 6c and d) fabrics. The SEM images in Fig. 6a and b demonstrate the smooth surface structure of the PET fabric before applying the NCC coating. Fig. 6c and d depict the morphological change in appearance of PET after coating with NCC: the presence of NCC on the surface is rather evident. Compared to the individual NCC in aqueous suspension, the dimension of the NCC involved in the coating was increased. This can be due to the fact that NCC particles aggregated during the thermal curing process (Dong et al., 2007; Li & Ding, 2007).

The energy dispersive X-ray (EDX) measurements were carried out to determine the surface chemical composition of the untreated and NCC-treated fabrics. Fig. 7 shows the EDX spectra of the untreated, unmodified NCC treated and cationic NCC treated fabric after multiple washing. As shown in Fig. 7b and c, the small sulfur pick in both original NCC and cationic NCC treated fabric is due to the presence of sulfate ester group on the NCC surface. Compared with the EDX spectrum of the untreated fabric (Fig. 7a) and original NCC treated fabric (Fig. 7b), the EDX spectrum of cationic NCC treated fabric (Fig. 7c) showed an additional peak for chlorine atom, which signifies the presence of quaternized NCC on fabric surface after multiple washing steps. The above characteristics also proved the successful immobilization of cationic NCC on the fabric surface.

The surface chemistry of original and NCC-treated fabrics was also analyzed by FTIR. Fig. 8a and b shows the FTIR spectra of cationic NCC, untreated fabric, and cationic NCC treated fabric, respectively. In Fig. 8a, a strong band for hydrogen bonded hydroxyls (O—H) stretching appeared at 3420 cm^{-1} (Peng et al., 2009), which was due to absorbed moisture by cationic NCC. In Fig. 8b, a peak at 1720 cm^{-1} was observed for both untreated and NCC-treated fabric. This can be attributed to the characteristic stretching vibration band of ester carbonyl (C=O) group, which confirmed

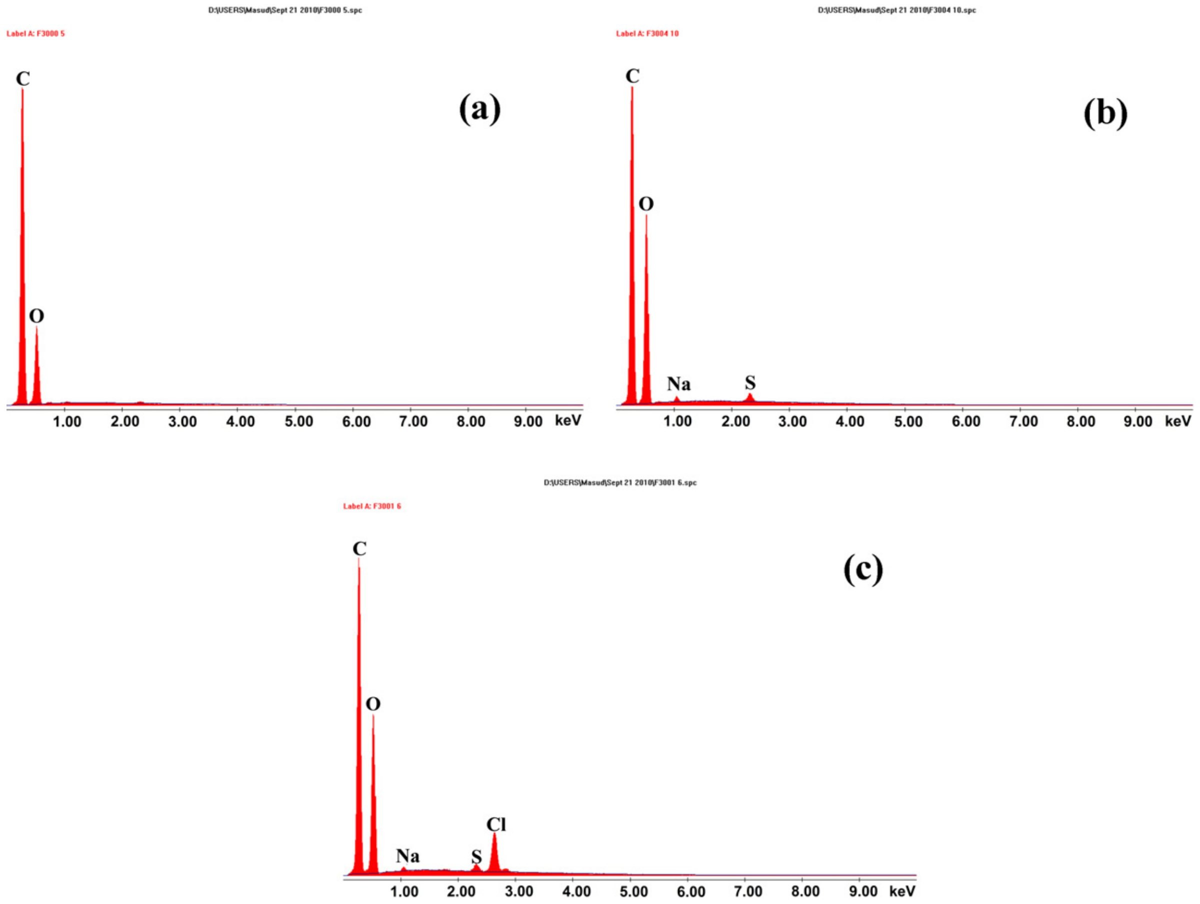

Fig. 7. EDX spectrum of untreated (a), original NCC treated (b), and cationic NCC treated PET (c) after 3 washing cycle.

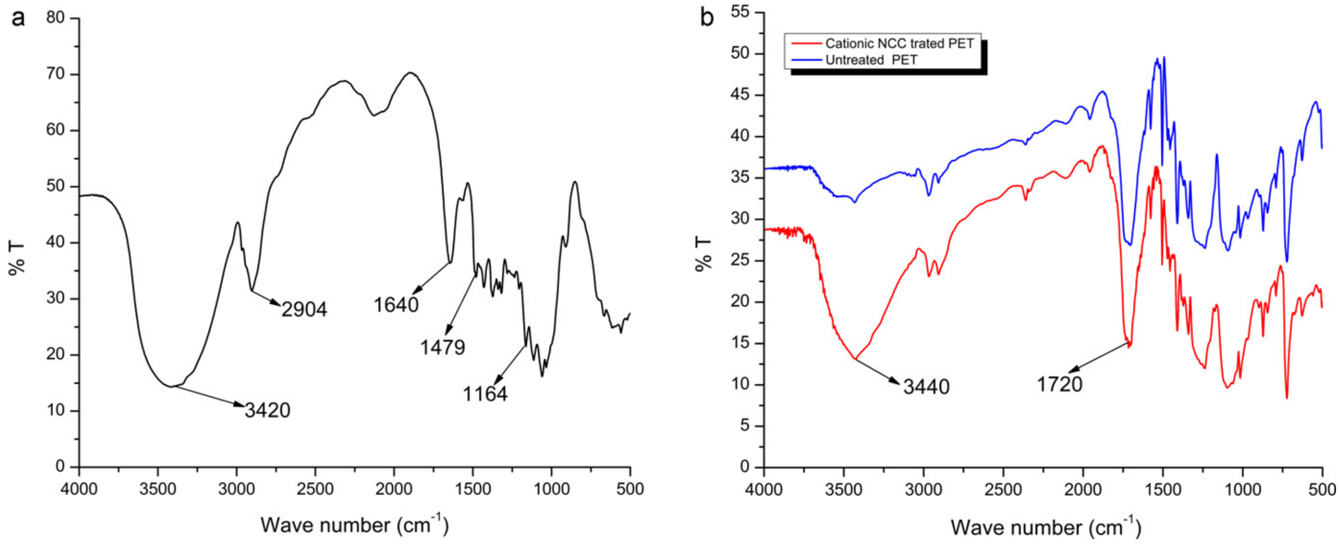

Fig. 8. FT-IR spectrum of cationic NCC (a), untreated PET (b), and cationic NCC treated PET fabric after three washing cycle.

the existence of esters (Alakara et al., 2008). In Fig. 8b, comparing the spectra of untreated and cationic NCC treated fabrics, the wide hydroxyl band found in the region of 3300–3500 cm^{-1} indicated the presence of cationic NCC on the fabric surface (Ploymalee et al., 2010). The presence of the hydroxyl band in case of NCC-treated fabric after multiple washing steps again confirmed the durability of the coating.

4. Conclusions

A durable and hydrophilic surface finishing system has been developed for polyester fabric by applying NCC-containing surface finishing system using a rolling–drying–curing process. The improvement in hydrophilicity was determined in terms of moisture regain, and wettability measurements. The moisture regain and wetting properties of the NCC-treated fabric improved significantly over the untreated PET fabric. The increase in the moisture regain and hydrophilicity indicated the improvement in moisture transport mechanism of the treated fabric. The results from the washing cycle and durability study demonstrated that the cationic modification of NCC not only improved the hydrophilicity but also increased the durability of cationic NCC-treated PET. SEM, EDX, and FT-IR studies confirmed the presence of cationic NCC on the fabric surface after multiple washing steps.

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