The durable hydrophobic and antibacterial polyester textile coating with $ZnO/Zn(OH)_2$/starch/stearic acid composite

DOI: 10.35530/IT.075.06.2023129

PHIPHOP NARAKAEW
SIWAT THUNGPRASERT
SAMROENG NARAKAEW
WIPANOOT BAISON
THEERAPORN PROMANAN
PAKORN SANTAKIJ
SUKEE SUKDEE
CHAINET CHANOGKUN
KANJANA RUTTANATEERAWICHIEN
APHIRUK CHAISENA
PIYAPORN KRACHODNOK

ABSTRACT – REZUMAT

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Herein, we prepare the antibacterial and hydrophobic polyester weaved textile using ammonium hydroxide (NH_4OH) hydrolysis and dip-coating method which can be fabricated zinc oxide (ZnO)/zinc hydroxide $Zn(OH)_2$)/starch/stearic acid (STA) composite on the textile surface. Phase identification, surface morphology, and chemistry of the composite were revealed by Raman spectroscopy and SEM/EDX techniques. The durable hydrophobic and antibacterial properties were investigated by water contact angle measuring, and against gram-negative Escherichia coli and gram-positive Staphylococcus aureus bacterium as per the colony count method according to the AATCC 100 antibacterial test method. Raman spectrum result shows the modified polyester textile sample surface, not only ZnO crystallized in the hexagonal wurtzite structure but also $Zn(OH)_2$, starch, and STA composite is included. SEM images confirmed a smooth surface of the treated fibre before washing due to using STA as a second coating agent. Despite the composite being peeled out from the surface fibre after 5 washing cycles, the finished polyester woven textile is good durable water-repellent. Moreover, good durable antibacterial activity against Staphylococcus aureus strains after 20 washing cycles. The colour differences and the whiteness index of the treated polyester textile after 20 repeated washing were not significantly changed.

Keywords: *$ZnO/Zn(OH)_2$/starch/STA composite, antibacterial activity, hydrophobic effects, coating, polyester textile*

Învelișul textil durabil hidrofob și antibacterian din poliester cu compozit $ZnO/Zn(OH)_2$/amidon/acid stearic

În acest studiu, a fost realizată o țesătură antibacteriană și hidrofobă din poliester folosind hidroliză și metoda de acoperire prin imersie cu hidroxid de amoniu (NH_4OH), care poate fi fabricat cu compozit oxid de zinc (ZnO)/hidroxid de zinc $Zn(OH)_2$)/amidon/acid stearic (STA) pe suprafața textilă. Identificarea fazelor, morfologia suprafeței și chimia compozitului au fost identificate prin spectroscopie Raman și tehnici SEM/EDX. Proprietățile hidrofobe și antibacteriene durabile au fost investigate prin măsurarea unghiului de contact cu apa și împotriva bacteriei Escherichia coli gram-negative și Staphylococcus aureus gram-pozitive, conform metodei de numărare a coloniilor în conformitate cu metoda de testare antibacteriană AATCC 100. Rezultatul spectrului Raman arată suprafața eșantionului textil din poliester modificat, nu numai ZnO cristalizat în structura hexagonală de wurtzit, ci și $Zn(OH)_2$, amidonul și compozitul STA. Imaginile SEM au confirmat o suprafață netedă a fibrei tratate înainte de spălare datorită utilizării STA ca al doilea agent de acoperire. În ciuda faptului că acest compozit a fost desprins de pe suprafața fibrei după 5 cicluri de spălare, s-a observat că țesătura din poliester finisată are o rezistență bună la apă. În plus, s-a observat și o activitate antibacteriană bună și durabilă împotriva tulpinilor de Staphylococcus aureus după 20 de cicluri de spălare. Diferențele de culoare și indicele de alb al țesăturii din poliester tratată după 20 de cicluri de spălare repetată nu au fost modificate semnificativ.

Cuvinte-cheie*: compozit $ZnO/Zn(OH)_2$/amidon/STA, activitate antibacteriană, efecte hidrofobe, acoperire, țesătură din poliester*

INTRODUCTION

Smart textiles are materials for our daily life and healthcare with more proper self-cleaning, hydrophobic, antibacterial, UV protection, and other properties that are essential requirements [1]. Currently, synthetic polyethylene terephthalate (PET) or polyester fibres have been the most popular textiles owing to their superior performances. There is known to have water-repellent properties, however, textiles manufactured thereof have a complex capillary-porous structure and do not possess waterproof properties. Therefore, it should be subjected to alteration waterproof treatment [2], included it is an unfavourable microorganism [3] for smart textiles. The smart textile modified with ZnO nanomaterial has been focused,

for example, the achieving of fabricated UV protection and self-cleaning properties on polyester textiles, wherewith excellent antimicrobial agents of ZnO nanoparticles, promoted reactive oxygen species (ROS) on the surface of these oxides as reported in previous paper [4].

The hexagonal wurtzite phase of ZnO, a general theoretical photocatalyst active under UV light with a maximum wavelength at 376 nm corresponding to band gap energy (E_g) at around 3.37 eV, even in the dark which can generate ROS, i.e., especially H_2O_2 and $^{\cdot}OH$, to destroy *Escherichia coli* or *Staphylococcus aureus* strains as found in previously reports [5–6]. Other advantages are low-cost, nontoxic, and chemical uniform. The process for ZnO preparation is normally initiated by the obtained $Zn(OH)_2$, zinc ion in an alkaline aqueous solution or utilizing the hydroxide ion (OH^-) source, followed by their transformation into ZnO with air annealing [2, 7–10].

The modified textile process can be done by using colloidal nanoparticles of ZnO or solution of zinc ion batch, deposited electrochemistry, dip-pad, and dip-coating, and in situ on textile with precipitation, hydro/solvothermal, ultrasonic, mechanical-chemistry, microwave, and sol-gel techniques. We had prepared successfully the coated $Zn(OH)_2$/ZnO polyester woven textile with pre-alkaline-treatment of hot ammonium hydroxide (NH_4OH) act as scourer and ester hydrolysed agents, hydroxide ion ($OH^–$) source for promoting ZnO deposited surface, controlled ZnO morphology, and environmental friendly [10] than compare to commonly used highly alkaline sodium hydroxide (NaOH) as reported in [11]. In addition, the coated polyester woven textile showed a hydrophobic water contact angle of 138º and 136º before and after 5 washing cycles [10]. Moreover, Ma and coworkers (2016) [12] reported there are a lot of hydroxyl functional groups on the biopolymer structure of starch that can be enhanced and absorbed in a large quantity and also reduce agglomeration of ZnO nanoparticles on textile fibered surfaces, and researchers used STA agent resulting of waterproof character on the textile fibre due to it can reduce the surface energy [13–14].

In this work, the functionalized antibacterial and hydrophobic characteristics of polyester woven textile by using ZnO/$Zn(OH)_2$/starch/STA composite were prepared and compared with the pristine textile. The coated textile was identified phase, surface morphology, and surface chemistry by using Raman spectroscopy and SEM-EDX techniques.

The durable waterproof textile was investigated by water contact angle measuring. The air permeability according to the ASTM D 737:2004. In the study of antibacterial activity, gram-negative *Escherichia coli* and gram-positive *Staphylococcus aureus* bacteria were evaluated using the colony count method (AATCC Test Method 100:2012 standard). To evaluate the appearance and surface colour of the treated polyester textile was tested before and after treatments including 5-10-20 repeated washing according to the ISO 105-J01:1989 standard, and whiteness index (WI) according to the ASTM E313: 2020 standard.

MATERIALS AND METHOD

The 17 warps and 17 wefts in centimetre white plain weave polyester textile was purchased from Sonibrazar shop, Lampang, Thailand. Ammonium hydroxide (NH_4OH) was ordered from Qrec, New Zealand. STA (*n*-octadecanoic acid) and 95%ethanol (95%EtOH) were purchased from World Chemical Group Co., Ltd., and PTT Home-Proservice Ltd., respectively. Starch (glutinous rice) was derived from a Thai commercial shop. Zinc chloride ($ZnCl_2$) was ordered from J.T. Baker. Non-phosphate standard reference detergent (SDCE ECE type A) nonionic soap was ordered from Union TSL Ltd, and distilled water was used.

The fabrication of ZnO/$Zn(OH)_2$/starch/STA composite on polyester textile

The fabricated composite on the polyester textile sample was a modified method from our previous work [10]. Firstly, the white plain weave polyester textiles size of 10×10 cm were washed and cleaned with detergent and distilled water, and continually immersed in 20 gl^{-1} NH_4OH aqueous solution at 90°C for 30 min. Subsequently, dipped for 1 min in the mixed aqueous solution of 6 gl^{-1} $ZnCl_2$, 2 gl^{-1} starch, and 0.05 M NH_4OH, followed by immersing in 50 gl^{-1} STA EtOH solution for 24 hours, and last, dried at 100°C for 1 hour (sample code: PZOS (figure 1, *b*). The controlled textile (sample code: P0 (figure 1, *a*), does not have any chemical solution added, and those processes use a liquor ratio used in textile of 50:1.

The durability of ZnO/$Zn(OH)_2$/Starch/STA composite on polyester textile

The washing test method (ISO 105 C01:2006) was performed to evaluate the washing with soap effect only on the ZnO/$Zn(OH)_2$/starch/STA composite coating fastness of the textile. The pristine and treated polyester textile samples were washed with a liquor ratio used in textile of 50:1 at 40±2°C for 30 min, using 5 gl^{-1} nonionic soap washing liquor. Then, removed the textile sample from the washed cylinder, taken subsequently to half the amount of distilled water, at ambient temperature, of 4 l container, followed softly stirring for 1 min, then rinsed with distilled water for 1 min, squeezed out the excess water by hand after, and continuously pressed the sample onto filter paper, and air dried at ambient temperature. The evaluation of durable composite on the sample fibre surface was done in 5, 10, and 20 washing cycles (Sample codes: PZOS-5WC, PZOS-10WC, and PZOS-20WC (figure 1, *c, d, e*).

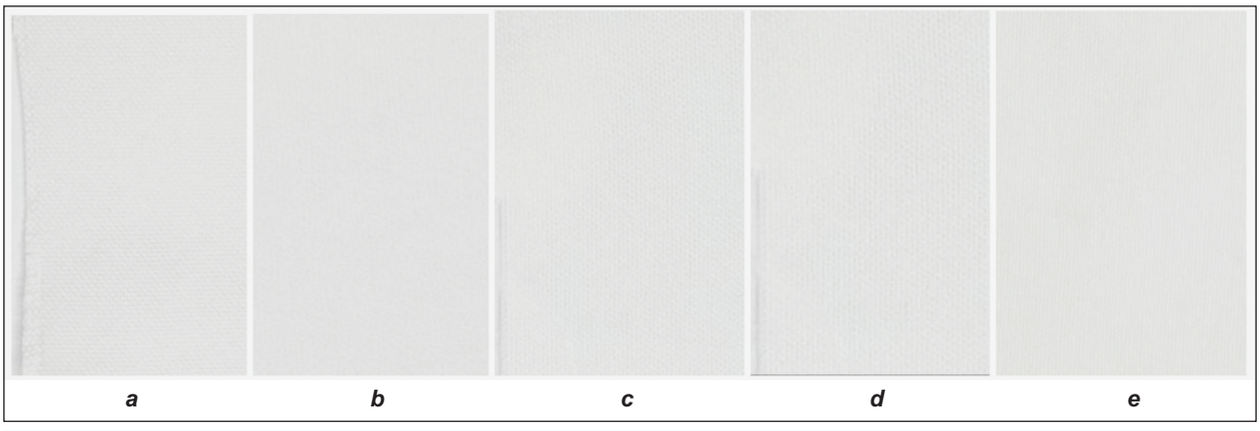

Fig. 1. Textile samples of: *a* – P0; *b* – PZOS; *c* – PZOS-5WC; *d* – PZOS-10WC; *e* – PZOS-20WC

Characterization methods

Raman spectroscopy

The phase identification of untreated (P0) and the treated polyester textile (PZOS) were investigated by a confocal Raman spectrometer (Jobin Yvon horiba LabRAM HR) using 532 nm excitation.

Scanning electron microscopy (SEM)

The surface morphology of the untreated (P0) and the treated polyester textile (PZOS) including 5-10-20 repeated washing were performed on a field emission scanning electron microscope (FE–SEM, JSM 6335 F) integrated with the incorporation of an energy-dispersive X-ray spectrometer (EDX) (Oxford Instruments). The electron microscope operating at 15.0 kV as accelerating voltage, was used to scan the surface morphology and potential elemental analysis on the fibered surface.

The hydrophobic property measurement

The hydrophobic surface of the pristine (P0), the treated polyester textile (PZOS), and 5–20 repeated washing were performed by optical contact angle measuring of water and contour analysis (Dataphysics, OCA40), operating the volume of distilled water dropping of 10 µl for 3 seconds at the humidity of 63% and temperature of 23°C.

The air permeability and antibacterial activity tests

The PZOS and 20-repeated washing were tested in the air permeability according to the ASTM D 737: 2004, using an M021A air permeability tester under the pressure difference between the textile surface of 125 Pa. For the antibacterial activity, the test organisms used a gram-positive organism, *Staphylococcus aureus* (ATCC 6538), and a gram-negative organism, *Escherichia coli* DMST 4212 (ATCC 25922), as per AATCC 100 antibacterial test standard [15]. A colony count method using AATCC Test Method 100:2012 standard (AATCC 100, 2012) evaluated quantitatively antibacterial activity of the textiles coated with ZnO/$Zn(OH)_2$/starch/STA composite (PZOS) before and after 20 washing cycles. The test specimens were prepared by the client and sterilized before testing by using the autoclave at 121°C and 15 psi for 15 min. 4.8±0.1 cm treated sterilized and control samples were inoculated with a suspension of microorganisms. After inoculation, a 100 mL volume of neutralizing solution was added to the bottles containing both the test and control swatches. The bottles were shaken forcefully for 1 min and serial dilutions were made with water subsequent by the standard plate count method in repetitive using nutrient agar. The plates were continually incubated for 24 hours at 37°C and the bacterial colonies were counted using a colony plate counting method. The assessment of antimicrobial activity was evaluated the comparing the percent reduction of bacteria between control and treated samples Percent reduction of bacteria (R) by specimen treatments was calculated using the following equation 1 formula:

$$\%\text{Reduction (R)} = 100(C - A)/C \quad (1)$$

where A and C are the number of bacteria (CFU/sample) recovered from inoculated treated test specimen swatches in the jar after 24 hours and 0 hour contact time, respectively.

Colour fastness and whiteness measurements

To evaluate the appearance and surface colour of the treated polyester textile were tested before and after treatments including 5-10-20 repeated washing, in terms of colourimetric values (CIE Lab) and colour differences (ΔE) according to the ISO 105-J01:1989 standard, and whiteness index (WI) according to the ASTM E313:2020 standard. Measurement conditions were performed for a field of view 10° standard observer under a D65 standard light source for conducted to quantify the ability of the human eye to perceive colours. Before the measurement, the samples were put on the white standard plate ($L^* = 93.38$, $a^* = 2.70$, and $b^* = -4.00$), and the degree of lightness (L^*), redness (a^*) or greenness (a^*), and yellowness (b^*) or blueness (b^*) of the treated polyester textile were measured. The following equations (2) and (3) were used to calculate the overall colour difference (ΔE) and WI.

$$\Delta E = \sqrt{(L^*_{sample} - L^*_{standard})^2 + (a^*_{sample} - a^*_{standard})^2 + (b^*_{sample} - b^*_{standard})^2} \quad (2)$$

$$WI = 100 - \sqrt{(100 - L^*)^2 + (a^*)^2 + (b^*)^2} \quad (3)$$

RESULT AND DISCUSSION

Raman spectroscopic results

In figure 2, *a* shows the characteristic bands of the pristine textile (P0) that were observed at 275 cm^{-1} corresponded to deformation of the C–C skeleton, at 658 and 699 cm^{-1} related to stretching of C–C ring, at 791 and 856 cm^{-1} matched to bending of C–C and C–O–C functional groups, at 994, 1093 and 1113 cm^{-1} assigned to stretch of C–O and C–C, at 1178 cm^{-1} matched to stretch of C–C ring, at 1289 cm^{-1} corresponded of COO stretching, and at 1371, 1412, and 1460 cm^{-1} related to bending of CH_2 functional groups [10, 15]. Figure 2, *b* reveals Raman scattering of vibration modes mentioned to $Zn(OH)_2$, ZnO, starch, STA, and polyester fibres in the treated PZOS. The observed bands at 228 cm^{-1} are also ascribed to the symmetric stretching Zn–O vibrational mode in $Zn(OH)_2$ [10, 16–17], whereas a strong band of 750 cm^{-1} is defined to the translational modes and the OH librations of $Zn(OH)_2$ and STA, respectively, as found in previous papers [18–19]. The hexagonal wurtzite structure of ZnO has the C^4_{6v} point group symmetry with each Zn metal ion surrounded by tetrahedral coordinated oxygen atoms and vice versa. This arrangement gives rise to polar symmetry along the hexagonal vertical axis (*c* axis). The optical phonons at the Γ point of the Brillouin zone correspond to the irreducible representation: Γ opt = A1 + + E1 + 2E2 + 2B1 [20]. The nine optical phonons are divided into one polar A1 mode and one doubly degenerate polar E1 mode which are split out into transverse (TO) and longitudinal optical (LO) phonons (Raman and IR active) while two doubly degenerate E2 modes (Raman active only), and two inactive B1 modes. Peaks appeared at 347, 396, 432, and 509 cm^{-1} assigned to $E2^{(high)}$-$E2^{(low)}$, $E2^{(high)}$, and B1, respectively. The others at 548, 551, and 579 cm^{-1} corresponded to A1(LO). Last, at 672, and 685 cm^{-1} matched with $B1^{(high)}$ +TA and $E2^{(high)}$ [7, 10, 16, 18–19]. In addition to the disappeared peaks at 275 and 856 cm^{-1} due to the hot NH_4OH possibly hydrolysed surface of polyester textile (P0) at ester-bonded carbonyl carbon and formed of ammonium terephthalate salt. Moreover, its carboxylate and hydroxyl groups of terephthalate and ethylene glycol were bonded with Zn^{2+} ion after being treated with the mixed solutions of Zn and starch, following coated with the alcoholic STA, and dried at 100°C for 1 h for dehydration formed PZOS as reported in our previous paper [10], others at 624 and 793 cm^{-1} corresponded δ(COC) and ring deformation of polyester as reported from [19]. Raman peak at 457 cm^{-1} is also assigned to δ(CCO), δ(CCC), ring deformation, and skeletal bending of glucose units in starch that is observed in [21]. Additionally, the experimental modes observed at about 339, 369, and 402 cm^{-1} were assigned as chain deformations, δ(CCC), of STA, constituting the most important vibrational modes in this spectral region as the previous report [22].

Morphological, elemental, and hydrophobic property studies

Figures 3, *a* and *b* report SEM images of PZOS revealing quite smooth fibered surface ZnO/$Zn(OH)_2$/Starch/STA composite as similar to the P0 due to using immersing time of about 24 hr for STA as final coated agent. In the case of before washing, the EDX spectrum shows the amount of Zn element about 6.38 %w/w as shown in figure 4, *a* and table 1. Whereas the composites were peeled off from the fibered surface after 5 and 20 washing cycles as observed from SEM images in figures 3, *c* and *d*, the EDX spectra show the amount of Zn element decreased about 1.39 and 0.87 %w after 5 and 20 washing cycles, respectively, as shown in figures 4, *b* and *d* and table 1, reported % weight element in only 20 washing cycles. The other peaks corresponded to the metal supporter and material that was conducted for sample preparation coating of SEM/EDX operation. In addition, synthetic polyester woven textile has hydrophilic behaviour with a water contact angle of

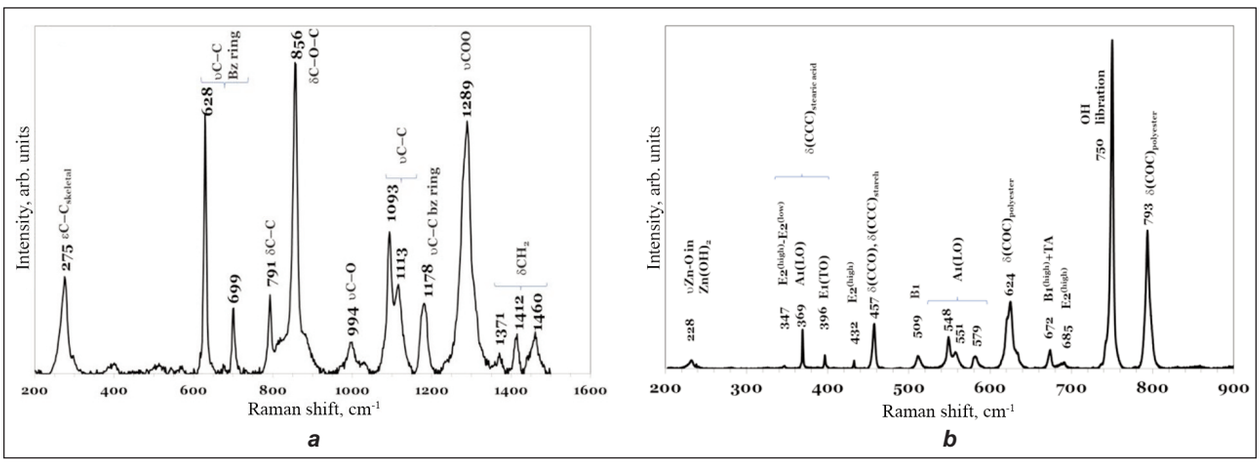

Fig. 2. Raman spectra of: *a* – P0; *b* – PZOS

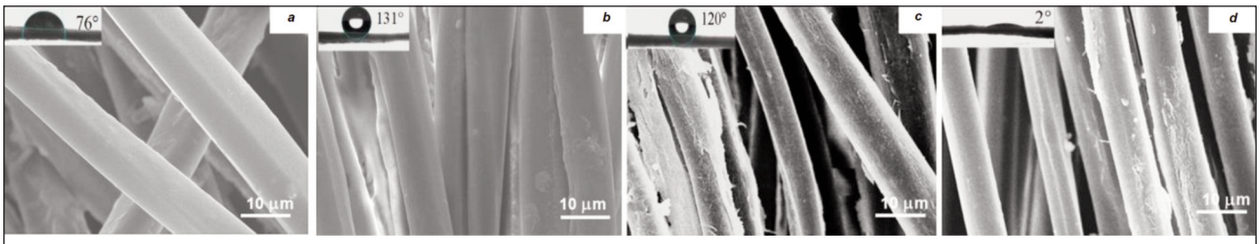

Fig. 3. SEM images of: *a* – pristine (P0); *b* – PZOS; *c* – PZOS-5WC; *d* – PZOS-20WC included water contact angles of 76°, 131°, 120° and 2°, respectively

76º, figure 3, *a*, due to the textile manufactured thereof having an intricate capillary-porous structure and not possessing hydrophobic properties [2], while PZOS consisting of ZnO/$Zn(OH)_2$/starch/STA composite enhanced to reduce the surface energy on polyester woven textile that can be enhanced the hydrophobic property with a water contact angle of 131º as shown in figure 2, *b*. Figures 3, *c* and *d* show the durability of ZnO/$Zn(OH)_2$/ starch/STA composite decreased with increasing the washing cycles, therefore, the water contact angle decreased to 120º and 2º of 5 and 20 washing cycles.

The air permeability and antibacterial activity studies

Table 1 shows the air permeability value of the PZOS-treated textile is high when compared with the 20-washing cycles due to the PZOS-treated textile containing a high amount of ZnO/$Zn(OH)_2$/starch/STA composite coated on the fibered surface. Moreover, good durable antibacterial activity against *Staphylococcus aureus* strains after 20 washing cycles was observed. As suggested the antimicrobial activity of functionalized PZOS-coated textile materials against gram-positive and gram-negative bacteria

Table 1

%WEIGTH ELEMENTS, THE AIR PERMEABILITY AND ANTIBACTERIAL ACTIVITY

Samples	%Weight elements			The air permeability (mm/s)	%Reduction (AATCC TM 100: 2012)	
	C	O	Zn		*Staphylococcus aureus*	*Escherichia coli*
PZOS	63.97	29.65	6.38	219.00	>99.92	>99.95
PZOS-20WC	60.49	38.64	0.87	87.96	>99.92	0

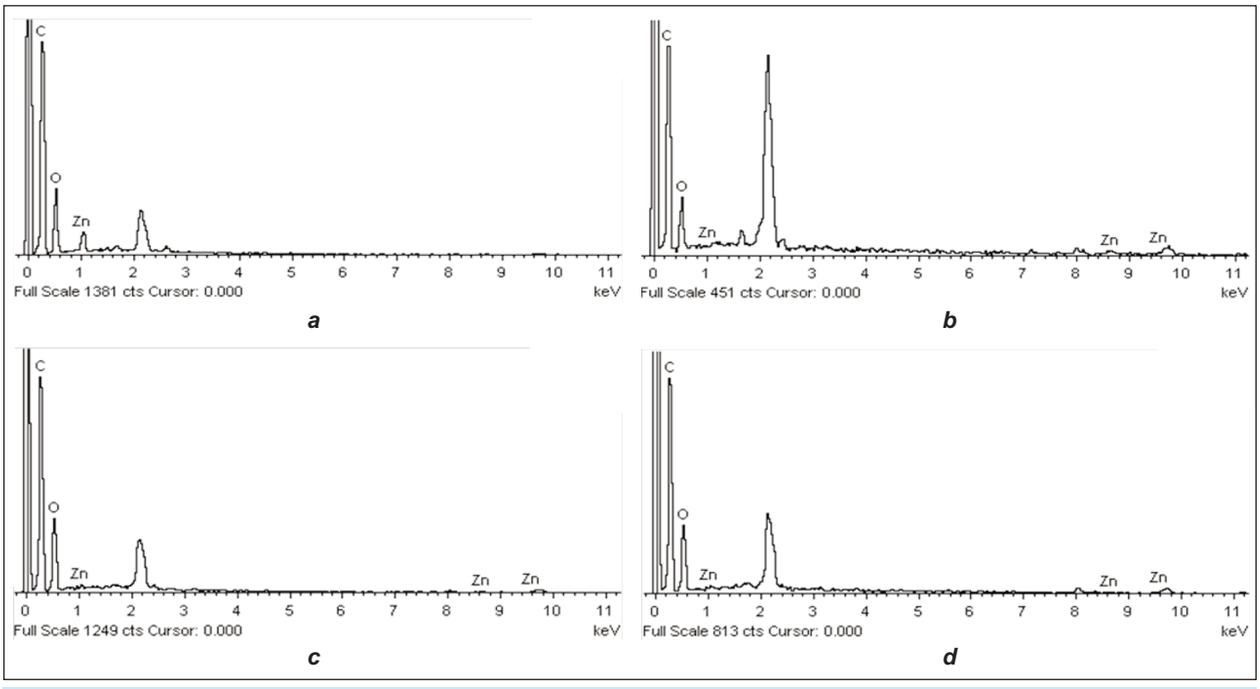

Fig. 4. EDX analyses of: *a* – PZOS; *b* – PZOS-5WC; *c* – PZOS-10WC; *d* – PZOS-20WC

Table 2

COLOUR FASTNESS AND WHITENESS INDEX					
Samples	**Textile colour**			**Colour differences**	**Whiteness index**
	L*	***a****	***b****	**Δ*E***	
P0	93.38	2.70	–4.00	0	91.81
PZOS	91.78	2.25	–3.59	0.94	90.75
PZOS-5WC	93.39	2.47	–3.35	0.57	92.19
PZOS-10WC	93.07	2.62	–3.28	0.63	91.90
PZOS-20WC	93.06	2.75	–3.95	0.16	91.55

because the surface of polyester textile was deposited with approximately in the range of 0.87–6.38 %w of ZnO-based composite that is more the crystal form which was confirmed by Raman spectroscopic result. Therefore, ZnO-based composite even in the dark can generate ROS, i.e., especially H_2O_2 and $^{\cdot}OH$, to destroy *Escherichia coli* or *Staphylococcus aureus* strains as found in previous reports [5, 6].

Colour fastness and whiteness index studies

The colour differences and the whiteness index of the treated polyester textile after repeated washing did not significantly change as reported in table 2.

CONCLUSION

Raman spectroscopy and SEM-EDX techniques revealed clear identifications of phase, surface morphology, and surface chemistry of the finished polyester woven textile with ZnO/$Zn(OH)_2$/starch/STA composite through NH_4OH hydrolysis and dip-coating method. The hydrophobic finished polyester woven textile with a water contact angle of 131°, the optimum durable hydrophobicity of 5 washing cycles. Notably, the water contact angle and the air permeability values decreased with increasing repeated washings. The highly durable antibacterial finished polyester woven textile with >99.92% reduction of a gram-positive organism, *Staphylococcus aureus*, while >99.95% reduction of a gram-negative organism, *Escherichia coli* was observed only on the finished textile before washing. Nevertheless, the colour differences and the whiteness index of the pristine, treated, and 20 repeated washing polyester textiles were not markedly changed.

ACKNOWLEDGEMENT

Financial assistance from LPRU/TRIS:002/2020 and Lampang Rajabhat University is gratefully acknowledged.

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Authors:

PHIPHOP NARAKAEW[1], SIWAT THUNGPRASERT[2], SAMROENG NARAKAEW[2], WIPANOOT BAISON[2], THEERAPORN PROMANAN[3], PAKORN SANTAKIJ[4], SUKEE SUKDEE[3], CHAINET CHANOGKUN[5], KANJANA RUTTANATEERAWICHIEN[6], APHIRUK CHAISENA[2], PIYAPORN KRACHODNOK[7]

[1]Department of Physics, Lampang Rajabhat University, 52100, Lampang, Thailand

[2]Department of Applied Chemistry and Center for Innovation in Chemistry, Lampang Rajabhat University, 52100, Lampang, Thailand

[3]Department of Chemistry, Lampang Rajabhat University, 52100, Lampang, Thailand

[4]Department of Information Technology, Lampang Rajabhat University, 52100, Lampang, Thailand

[5]Department of Communicative Thai for Foreigners, Lampang Rajabhat University, 52100, Lampang, Thailand

[6]Department of Digital Business Management, Lampang Rajabhat University, 52100, Lampang, 52100, Thailand

[7]School of Telecommunication Engineering, Institute of Engineering, Suranaree University of Technology, 30000, Nakhon Ratchasima, Thailand

Corresponding author:

SAMROENG NARAKAEW
e-mail: krachodnok@lpru.ac.th