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Temperature and moisture responsive nanocomposite treated polyester fabric for smart bagging recovery

Nazife Korkmaz Memiş[a] & Sibel Kaplan

Textile Engineering Department, Faculty of Engineering, Süleyman Demirel University, Isparta 32200, Turkey

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In this study, polyester fabrics having temperature and water/moisture responsive dynamic recovery function have been prepared with a nanocomposite finishing treatment composed of shape memory polyurethane (SMPU) and cellulose nanowhiskers (CNWs). The effect of CNW concentration (5-20 wt%) within SMPU-CNW nanocomposite on bagging recovery has been investigated by a test procedure, including relative humidity (RH), air, and water media at different temperatures simulating laundry and drying processes. Characterization of fabrics is conducted by FTIR, SEM and DMA studies. FTIR and SEM analyses results confirm the existence of SMPU-CNW nanocomposite on fabric. SMPU-CNW treatment significantly increases the weight and bursting strength of the fabrics, as expected. Most notably, SMPU-CNW nanocomposites enable a dynamic bagging recovery of up to 100% above T_a of SMPU in water. Results give implications of an active polyester fabric production possibility having dual responsiveness to both body physiological and environmental changes besides acceptable hand and strength.

Keywords: Bagging recovery, Cellulose nanowhisker, Polyester, Shape memory polyurethane, Temperature responsive, Moisture responsive

1 Introduction

Fabric bagging is a partial or total permanent three-dimensional deformation, which happens during repetitive or prolonged multidirectional tension or shear of fabric during wear[1]. As it is considered as an aesthetically undesirable deformation[2], the bagging problem has been widely studied theoretically and experimentally[1, 3, 4]. An ideal garment should provide the required bagging recovery performance and smooth appearance[5]. Generally, the features of freedom in movement and bagging recovery are enabled in fabrics with elastane in different forms. But preceding study results revealed that thermo physiological comfort performance could be better with lower elastane content and stretch. On the other hand, lower elastane in polyester fabric was inadequate for recovery characteristics[6, 7]. Smart polyester fabrics having the mentioned functions can be directly produced by SMPU finishing application besides its usage in filament form[7, 8] with the easy application procedure and lower cost. Nevertheless, sufficient shape memory effect obtained with a high concentration of SMPU leads to poor fabric hand[9].

Shape memory polymers (SMPs) may be applied on fabric to obtain a novel shape memory effect creating bagging recovery and dimensional stability with external heat stimuli, such as domestic laundry, steaming and hot wind tumble drying process[4, 6]. Through various thermo-responsive SMPs, SMPU is the most suitable material for the textile industry and can be used in various forms, such as film for smart breathability[10-12], fibre to produce fabrics for bagging recovery, smart breathability[6, 8-18], and coating/finishing applications for smart breathability, wrinkle recovery, crease retention and anti-felting[19-30]. Among the mentioned properties, bagging recovery of SMPU filament for a knitted fabric at different blend ratios (100%, 50% and 16%) with cotton were investigated, as compared to elastane[25, 28]. They found out that the bagging recovery performance of SMPU knitted fabrics is changed according to temperature (better performance at >60°C) and shape-memory fibre content.

However, besides temperature, moisture/water is also important for clothing comfort and end use processes. Nevertheless, SMPU has limitations in commercial applications, such as higher activation temperature, response time and concentration required for sufficient shape memory effect, which leads poor fabric handle[30]. Therefore, developing materials with

[a]Corresponding author.
Email: nazifekorkmaz@sdu.edu.tr

water responsiveness as well as temperature is within strong industrial demands.

Taking into account these limitations, nanocomposite technology has been used for obtaining water/moisture responsiveness by modifying thermally responsive SMPs with hydrophilic nanoparticles[31]. Among these hydrophilic nanoparticles, CNWs have been used recently as a filler for water/moisture responsiveness with their large surface area presenting a high concentration of (-OH) groups[32], hence hydrophilicity. Modification of SMPU matrix by CNWs provides nanocomposites with water responsive shape memory effect depending on the plasticisation and percolation network formed by CNWs whose hydrogen bonds can be reversibly regulated by water molecules[33-36]. Moreover, nanocomposite materials formed by introducing CNWs into SMPU matrix could improve fabric hand[34, 36].

In this study, a novel nanocomposite finishing treatment has been applied on polyester knitted fabric for shape memory performance in case of dimensional or fabric geometrical change under different media, simulating different laundry and drying processes or body wearing conditions. Besides dynamic dimensional changes in terms of bagging recovery, physical and mechanical properties (weight, thickness, bending rigidity, and bursting strength) are also studied. The morphology, chemical compositions and thermomechanical characterization of the treated fabrics are investigated by SEM, FTIR and DMA respectively.

2 Materials and Methods

2.1 Materials

100% staple polyester single jersey fabric having weight of 139 g/m^2, density of 26 courses/cm and 20 wales/cm and yarn count of 46 tex was used. Before finishing treatment, fabric samples were washed 5 times in a washing machine (Wascator FOM71 CLS, Electrolux) by using ECE standard detergent according to TS EN ISO 6330: 2012.

Temperature and water/moisture responsive nanocomposite suspensions were prepared with pellet-type MM-3520 SMPU as matrix and CNWs as nanofiller. The pellet-type MM-3520 SMPU (SMP Technologies Inc., Japan) has temperature responsive shape memory effect and also T_g of 32.12°C (T_{trans}=T_g) according to DSC result[37], which is suitable for body applications. CNWs, prepared from the sulphuric acid hydrolysis process, were supplied directly from Grafen Co in Turkey, having a length of 150-200 nm and width of 20 nm (by TEM) and crystallinity of 98.98% (by XRD)[34]. The non-ionic surfactant polyoxyethylenesorbitan monooleate (Tween80, Sigma Aldrich, USA) with high HBL around 15 was used to enhance the dispersion of CNWs in SMPU matrix. N, N-dimethylformamide (DMF) (Sigma Aldrich, USA) was used as solvent. All chemicals (analytical grade) were used as received without further purification.

2.2 Application of SMPU-CNW Nanocomposite Treatments on Polyester Fabric

SMPU-CNW nanocomposite suspensions were produced by introducing CNWs into SMPU matrix. According to preliminary experiments, first SMPU pellets were dissolved in DMF at a concentration of 10 wt% at 60°C for 6 h. This concentration was determined according to optimum shape memory and comfort properties of 5, 10, 15, and 20 wt% SMPU treated fabrics[37]. In the second step, homogeneous and stable 0.5 wt% dispersion of CNWs in DMF was prepared by adding Tween80 at 1:2 w/w of CNW: non-ionic surfactant by using ultrasonic stirring until dispersion became visually homogenous. The CNW dispersions were then drop-wise transferred into the SMPU solution under ultrasonic stirring for another 1 h. The final solutions consisted of 10 wt% SMPU and CNW concentrations ranging from 0 wt% to 20 wt% relative to the polymer weight[34].

The polyester fabrics were treated with nanocomposite suspensions using pad-dry-cure technique and passed through squeeze rollers at 3 bar pressure and 2 m/min speed to reach an average wet pick up of 90%. After applying nanocomposite suspensions, fabrics were dried at 85°C for 3 min and cured at 120°C for 2 min. The samples were abbreviated in Table 1. Also, the samples were pre-conditioned according to standard ASTM D1776-08e1 (2009) at 20 ± 1°C and 65 ± 2% RH for 24 h prior to testing and analyses.

Table 1 — Samples codes of treated polyester fabrics

Sample	Concentration of finishing agent, wt%	
	SMPU	Nanofiller
Reference polyester fabric (PES)	-	-
PES-SMPU	10	-
PES-SMPU-CNW5	10	5
PES-SMPU-CNW10	10	10
PES-SMPU- CNW20	10	20

2.3 Characterization of SMPU-CNW Nanocomposite Treated Polyester Fabrics

2.3.1 Morphological, Chemical and Thermomechanical Analyses

Surface morphologies of the samples were characterized by SEM analysis using a Fei Quanta FEG 250 SEM (Thermo Scientific, USA). The SEM images were taken at an accelerating voltage of 20 kV and a current of 10 µA at a high magnification power up to 2500. Elemental analysis and mapping by Energy-Dispersive X-ray spectroscopy (EDX) were performed to obtain a qualitative analysis of elemental contents.

A series of FTIR spectra were recorded by using a Perkin Elmer Spectrum BX FTIR spectrometer to evaluate the presence of polymer/nanocomposite finishing treatment in fabric structure and interactions between fabric and treatment solutions. FTIR spectral measurements were performed on a KBr disk and each specimen was scanned in the 400-4000 cm^{-1} range with a resolution of 4 cm^{-1}, 2 cm^{-1} interval and 16 scans.

The thermodynamic properties of fabric samples were determined by dynamic mechanical analysis (DMA). The measurements were made at a frequency of 1 Hz and in the temperature range from -50°C to 180°C at a heating rate of 5°C/min with liquid nitrogen as a coolant gas.

2.3.2 Physical and Mechanical Tests

Weight, thickness and bending rigidity tests of the samples were carried out according to standards TS EN 12127, ASTM D 1777-96 by James Heal R&B Cloth Thickness Tester and ASTM D 1388-92:2002 by WIRA Cantilever Bending Rigidity Test Apparatus, respectively. Bursting strength of the samples was tested by using the Lloyd LR5K+ Tensile Tester with a ball-burst attachment according to the standard ASTM D6797-15 (ball burst test).

2.3.3 Bagging Fixity and Recovery Based Shape Memory Performance Tests

Bagging recovery performances of the samples (9 cm × 9 cm) in different environments (air, water, and RH) were tested to clarify dynamic dimensional change based on shape memory effect with temperature and water/moisture stimuli. The comprehensive test procedure is consist of 4 stages as listed below and shown schematically in Fig. 1.

(i) Evaluation of Original Shape — Fastening samples on the top of circular bottom plate (diameter; 5 cm) on the reverse side up and measuring initial lengths in the course and wale directions.

(ii) Shape Deformation (Making Bagging) — Exerting a force to the sample using the steel ball at a speed of 20 mm/min, holding at 20 mm (maximum extension) displacement for 5 min. In this study, maximum bagging extension was determined according to bursting strength and extension results. Relaxing the sample by rising the steel ball to the starting point and measuring the deformed lengths in the course and wale directions.

(iii) Recovery Process — Pinning the samples into rubber tape and keeping within a temperature-controlled chamber of 20°C, 40°C, and 65°C at 50% RH and also 20%, 40%, and 65% RH for the constant temperature at 20°C for 30 min to simulate drying conditions for shape recovery process in air media. For shape memory in water media, immersing the samples in water environments of 20°C, 40°C, and 65°C for 5 min to simulate different washing temperatures. Then, drying the samples on hangers at 20°C, 65% RH for 24 h.

(iv) Evaluation of Shape Recovery — Measuring lengths of the samples in course and wale directions and then calculating bagging fixity (R_f) and recovery (R_r) ratios for both directions according to folloowing equations:

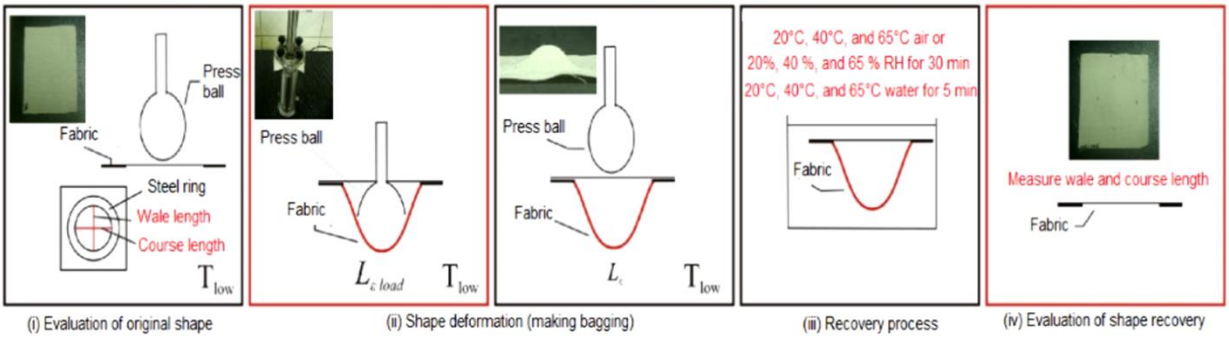

Fig. 1 — Bagging recovery process depending on shape memory effect[36]

$$R_f = \frac{L_\varepsilon - L_o}{L_{\varepsilon,load} - L_o} \quad ...(1)$$

$$R_r = \frac{L_{\varepsilon,load} - L_f}{L_{\varepsilon,load} - L_o} \quad ...(2)$$

In the formulas, L_o, $L_{\varepsilon,load}$, L_ε and L_f represent the original length, the extended length under stress, the fixed-length after stress removal, and the final length of dried samples (Fig. 1). Average values were calculated by using results of course and wale directions.

2.4 Statistical Analysis

SPSS Statistics 22.0 for Windows (IBM, Armonk, USA) were used for statistical analysis of the test results. The effects of finishing agent type/concentration, media, RH, and temperature factors on bagging recovery and fixity performances were tested by MANOVA (multivariate analysis of variance). Student-Newman-Keuls (SNK) tests were used to examine the differences among measured parameters of the samples.

3 Results and Discussion

3.1 Surface Morphology, Chemical and Thermomechanical Characterization of Treated Polyester Fabrics

The surface morphologies of the fabrics have been determined by using SEM at different magnifications and the obtained results are displayed in Fig. 2. For the reference sample, the SEM images [Figs 2 (a) and (b)] clearly show that grooves and fibres are

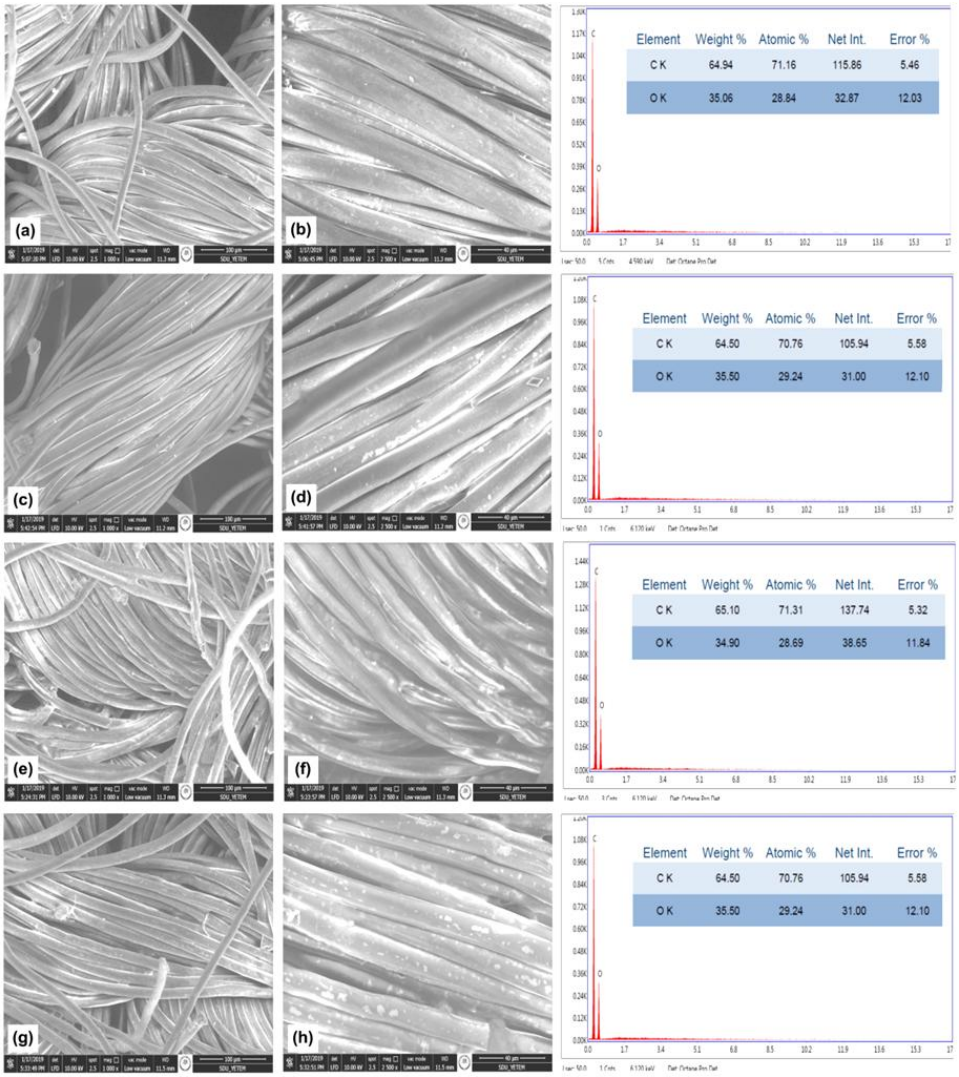

Fig. 2 — SEM micrographs at ×1000 and ×2500 and EDX spectrum of the reference fabric (a, b), PES-SMPU (c, d), PES-SMPU-CNW5 (e, f), and PES-SMPU-CNW20 (g, h)

prominent. Also, the fibres have a smooth surface with some small spots which might be oligomers formed during the melt spinning process[38]. After treating with SMPU, a thin layer is observed not only on the fibre surface but also between the two neighbouring fibres [Figs 2 (c) and (d)]. It presents the successful immobilization of SMPU polymer onto polyester fabric. As compared to treatment with SMPU, SMPU-CNW nanocomposite treatment with 5 wt% [Figs 2 (e) and (f)] and 20 wt% CNW create a relatively uniform, and higher filling among fibres. Also, higher CNW concentration (20 wt%) results in thicker nanocomposite film and a more apparent distribution of nanoparticles on the fabric surface.

The EDX measurements have been carried out to determine the surface chemical composition of the fabric samples and results are reported in Fig. 2. As shown in Fig. 2 (a), the reference sample has a relative elemental composition of 71.16% carbon and 28.84% oxygen atoms. After treating with SMPU-CNW nanocomposite suspensions having 5 wt% and 20 wt% nanoparticles, the oxygen/carbon ratio of the samples increased (0.41) compared to SMPU treated ones (0.40). This increase in oxygen/carbon ratio confirms the SMPU-CNW nanocomposite presence on the fabric surface.

FTIR has been used to analyse fabric surface components before and after functional finishing treatment to ensure the presence of SMPU and SMPU-CNW nanocomposite in the fabric structure. FTIR spectrum of SMPU polymer, reference and treated polyester fabrics are given in Fig. 3. As shown in Fig. 3 (a), the characteristic peaks of polyester at 3434 cm^{-1} (C=O, carbonyl overtone), 2965 cm^{-1} (glycol C-H stretching), 1577 cm^{-1} (C-C ring stretching), 1506 cm^{-1} (CH ring in-plane bending and C-C ring stretching), 1469 cm^{-1} (CH_2 bending, O-C-H bending), 1411 cm^{-1} (C-H ring in-plane bending and C-C ring stretching), 1345 cm^{-1} (C-H vibration, O-C-H bending), 1239 cm^{-1} (C(=O)-O stretching, C-C ester ring stretching, C=O in-plane bending), 1094 cm^{-1} (glycol C-O stretching), 1009 cm^{-1} (C-C-C ring bending, C-C ring stretching, CH ring in-plane bending), 964 cm^{-1} (O-CH_2 stretching, C(=O)-O stretching, chain folding), 872 cm^{-1} (C-H ring out-of-plane bending, C-C ester ring out-of-plane bending, C=O out-of-plane bending, ring bending), 844 cm^{-1} (C-C ring stretching, C=O in-plane bending, C-H_2 vibration), 790 cm^{-1} (C-H ring out-of-plane bending, C=O vibration and CCO bending), 727 cm^{-1} wavelengths (C=O out-of-plane bending vibrations) were determined, respectively. In Fig. 3 (c), comparing the reference fabric, the peaks at 1714 cm^{-1} belong to the (C=O) groups of urethane and 1220-1362 cm^{-1} assigned to (C-O) stretching vibration of SMPU indicates the presence of SMPU on the fabric surface. In the FTIR spectrum of treated samples, a free urethane C=O group at 1714 cm^{-1} is observed in the SMPU treated ones [Fig. 3 (c)], while this peak is not observed in the SMPU-CNW treated ones [Figs 3 (d) and (e)]. Moreover, the peak broadening and intensities increase of hydrogen-bonding C=O group[39] at 1703 cm^{-1} with CNW concentration prove the presence of SMPU-CNW nanocomposite on the fabric surface. Also, improved strength of the alkane (C-H) asymmetric stretching vibration peak at 2906 cm^{-1} and 2964 cm^{-1} indicates more (-CH_2-) in SMPU, which belongs to CNWs and is another proof of SMPU-CNW nanocomposite presence on the fabric surface.

DMA has been performed to determine dynamic mechanical properties of the fabrics including storage modulus (E'), loss modulus (E'') and phase angle (tan δ). E' is a measure of deformation energy, stored in the material and also an indicator of elastic character. According to Fig. 4, treated fabric with SMPU-CNW nanocomposite has the highest storage modulus value. PES-SMPU and PES-SMPU-CNW20 samples exhibit three regions such as a glassy region, transition region and rubbery plateau. Such a distinction is not observed in the reference samples. E' of the treated polyester fabrics at both glassy region and the rubbery plateau is higher than that of the reference ones. In the presence of SMPU and SMPU-CNW20 treatments, the increase of E' at both glassy region and rubbery plateau might be ascribed to polymer chain coating on the fabric surface and formation of percolation cellulose network via strong hydrogen bonding of CNW particles. E' of these samples decreases significantly with temperature increase due to the relaxation of SMPU matrix[40] on fabric surface which is not the case for reference one. When PES-SMPU and PES-SMPU-CNW20 are compared, the E' of PES-SMPU-CNW20 was lower than PES-SMPU in the rubbery plateau, indicating higher elasticity of the material. This result can be attributed to the more elastic and thicker polymer layer formation on polyester fibres with the plasticizing effect of CNW[41] (Fig. 2).

E'' is the component of dynamic modulus which measures hysteresial energy dissipation and polymer chain mobility. Wider E'' curve indicates better mechanical properties[42]. As shown in Fig. 5, E'' value

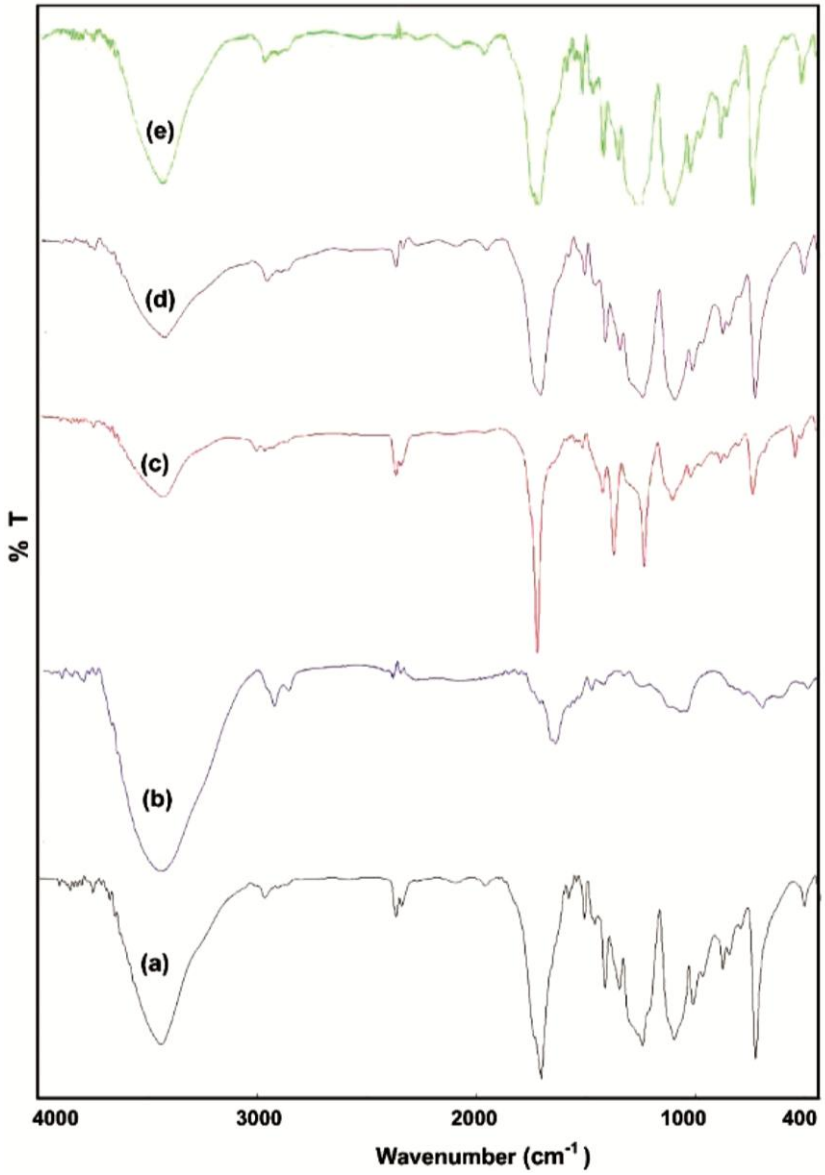

Fig. 3 — FTIR spectra of reference fabric (a), SMPU polymer (b), PES-SMPU (c), PES-SMPU-CNW5 (d), and PES-SMPU-CNW20 (e)

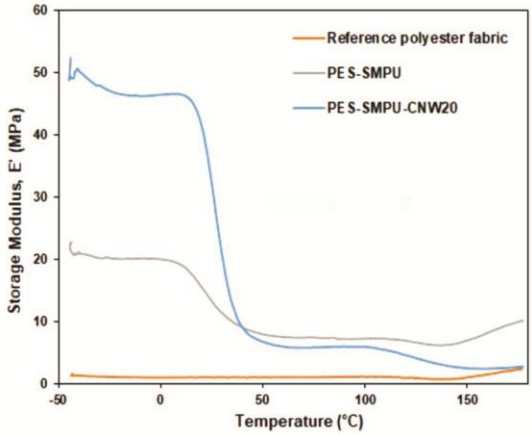

Fig. 4 — Storage modulus of reference and selected treated polyester fabrics

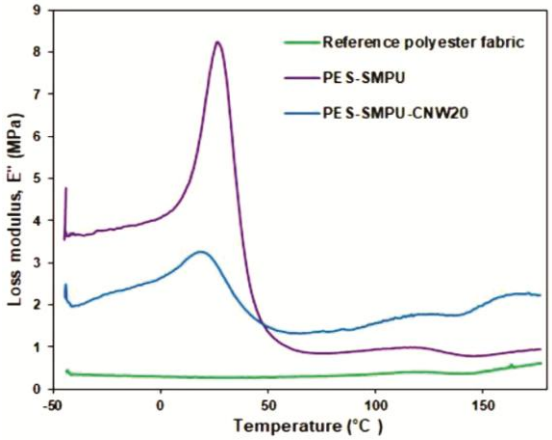

Fig. 5 — Loss modulus of reference and selected treated polyester fabrics

of samples increases with both SMPU and SMPU-CNW20 nanocomposite treatment. Also, *E″* of the treated samples at both glassy region and rubbery plateau is higher than that of the reference ones. There is a broadening of *E″* curve with an increase in transition range on PES-SMPU-CNW20. Besides this, the *E″* of PES-SMPU-CNW20 is higher than the reference at the rubbery plateau. These results indicate that the mechanical properties of polyester fabric are increased with SMPU and especially with SMPU-CNW20 nanocomposite treatment.

Phase angle (tan δ), the ratio of *E″* to *E′*, is known as the damping and measure of energy dissipation. T_g values of the fabrics are determined by maximum values of tan (δ) peaks shown in Fig. 6. T_g of the reference sample is 37.18°C, whereas the maximum values of tan (δ) peaks of polyester fabrics treated with SMPU and SMPU-CNW20 nanocomposite are seen at 34.83°C and 29.50°C respectively. The decrease in T_g temperature of SMPU-CNW20 nanocomposite may be attributed to the plasticizing effect of CNWs. Peak height increase of tan (δ) indicates the molecular mobility of materials, while the broadening of tan (δ) represents homogeneity of material crosslinking[42]. The peak height of PES-SMPU-CNW20 is decreased as compared to the reference and PES-SMPU ones. This indicates that the molecular chain mobility of SMPU matrix and the fabrics is reduced by CNW particles within SMPU matrix. Furthermore, the reduction of peak height in nanocomposite treated samples also shows the interaction between the SMPU matrix, -OH groups on the surface of CNW particles, and the fabric samples (detected in FTIR analysis) and the restriction of segmental movement in polymer chains due to this interaction.

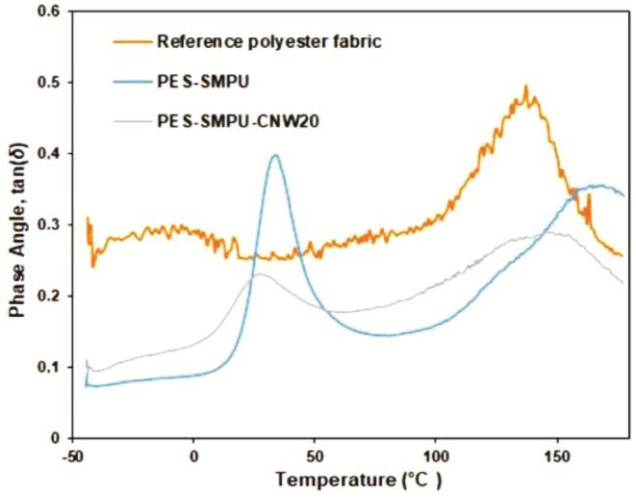

Fig. 6 — Phase angle tan (δ) curves of reference and selected treated polyester fabrics

3.2 Physical and Mechanical Test Results of Treated Polyester Fabrics

The physical and mechanical properties of the fabrics and their statistical analysis results are compiled with different superscript letters in Table 2 and given in Fig. 7. Results reveal that weight values of the treated fabrics increase significantly (p<0.05), as expected. While all SMPU-CNW nanocomposite treated fabrics have statistically identical weight values except for PES-SMPU-CNW20, having the highest value. Thickness of the fabric is increased significantly with SMPU and SMPU-CNW nanocomposite treatment at around 27% and 13%, respectively (Fig. 7). The thicker coating at higher CNW concentration (20 wt%) is also seen in SEM images (Fig. 2), promoting the mentioned results.

Table 2 — Physical and mechanical properties of polyester fabrics

Sample	Weight g/m^2 [SD]	Bending rigidity mg.cm [SD]	Bursting strength N [SD]
Reference polyester fabric	139^{a} [7.852]	55.543^{a} [2.352]	518^{a} [43.524]
PES-SMPU	150^{b} [1.386]	287.888^{e} [6.815]	583^{b} [24.572]
PES-SMPU-CNW5	151^{bc} [3.274]	159.635^{d} [12.035]	630^{c} [23.067]
PES-SMPU-CNW10	157^{bc} [4.406]	144.747^{c} [8.342]	637^{c} [3.373]
PES-SMPU-CNW20	159^{c} [0.462]	93.707^{b} [6.161]	641^{c} [24.351]

SD — Standard deviation.

Superscripts a, b, c, d and e show statistical differences among the results (p<0.05).

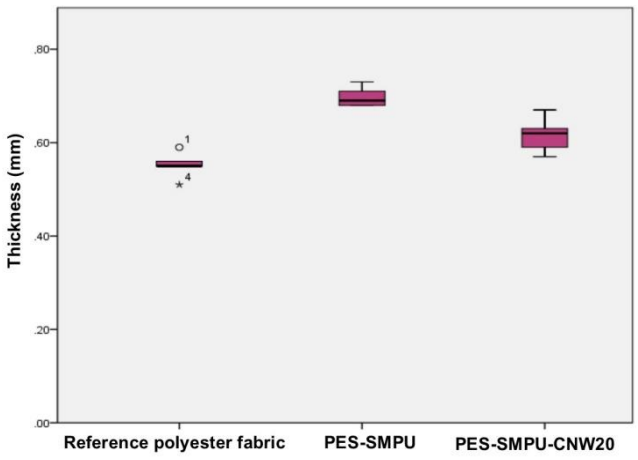

Fig. 7 — Boxplot diagram of thickness values of reference and treated polyester fabrics

SMPU and SMPU-CNW nanocomposite treatment leads to a significant increase in bending rigidity as expected. The bending rigidity of the treated fabrics is reached 287.888 mg.cm for PES-SMPU samples, 5 times higher than reference ones. This increase is thought to be originated from a reduction in the relative motion of fibres/yarns within the fabric during bending. Nevertheless, the bending rigidity of the other treated fabrics with SMPU-CNW nanocomposites is reduced proportionally with CNW concentration. As a bending rigidity increase is expected from all kinds of finishing treatments, a minimum increase for PES-SMPU-CNW20 (93.707 mg.cm) is found acceptable, which is confirmed also by subjective evaluation. Reduction in bending rigidity of polyester fabric with CNW can be attributed to enhancement of elastic property of the yarn due to the elastic polymer layer formation on fibre surface[41] with the plasticizing effect of CNW. It is also thought that CNW particles cause a reduction in inter yarn friction, hence increases the mobility of the yarn due to the softener effect originated from the plasticization of CNWs. The mechanical properties results show that SMPU and SMPU-CNW nanocomposite significantly increase bursting strength of polyester fabric (p<0.05), but CNW concentration does not lead to a significant difference; a result consistent with DMA results. The slight increase of bursting strength with SMPU treatment could be the result of contradictory effects of polymer limiting yarn movement (decreasing effect) and supporting fibres/yarns (increasing flexural stiffness and stress modulus of fabric[43]).

3.3 Shape Memory Performances of Treated Polyester Fabrics

Effect of CNW concentration within SMPU matrix on dynamic shape recovery performance in the case of fabric geometry or dimensions has been investigated through bagging recovery of the fabrics under different temperatures and media. To evaluate the temperature and water/moisture sensitive shape recovery performance, bagging fixity (R_f) and recovery (R_r) values are calculated and effects of finishing treatment type/concentration, temperature, environment (water /air), RH, and their interactions on the mentioned parameters are determined by MANOVA. According to MANOVA result, finishing treatment type, temperature, environment (water/air), RH, and all their interactions have significant effects on bagging recovery and fixity performances of the fabrics (p=0.00<0.05), meaning a smart shape memory function.

R_r and R_f ratios of the reference and treated samples according to different water and air temperatures are given in Table 3. As results are analysed according to finishing treatment type, the reference samples have the lowest R_r and R_f ratios, as expected. Also, bagging recovery performance is significantly increased with SMPU and SMPU-CNW treatment, especially directly proportional to CNW concentration and the significantly highest R_r and R_f ratios belong to PES-SMPU-CNW20 among all samples. This performance about dynamic bagging recovery may be attributed to the fact that shape memory characteristic is imparted to polyester fabric probably through grafting SMPU matrix and CNW particles onto the fibre surface to form inter-fibre and inter-yarn bonds as also seen in FTIR results. Increase in fabric elasticity and thicker polymer film formation on polyester fibres (Fig. 2) with SMPU-CNW nanocomposite treatments may be the other reasons for this function[44]. Furthermore, the higher E' of PES-SMPU-CNW20 in comparison with reference fabric at rubbery plateau might led to higher dynamic bagging recovery. The increase of E' indicates that the elastic energy storage capacity of the material is diminished with CNW within SMPU matrix. Therefore, PES-SMPU-CNW20 has tendency to store lower degrees of entropic energy and release this energy above $T_{trans.}$ (T_g in this study) in stress-free mode, hence higher R_r ratios[45].

As R_r and R_f ratios are evaluated according to temperature, statistical analyses show that R_r of all samples increases with temperature, especially abrupt increase at 65°C. At this temperature, the highest bagging recovery performance (55% in dry air and 100% in water) belongs to PES-SMPU-CNW20 samples. Also, R_f results, meaning the fixing capacity of the fabric structure after deformation, show a similar trend that PES-SMPU-CNW20 samples have

Table 3 — Bagging recovery (R_r) and fixity (R_f) performances of polyester fabrics according to air and water temperature

Sample	20°C		40°C		65°C	
	R_r	R_f	R_r	R_f	R_r	R_f
Reference polyester fabric	0.00/ 0.00	0.00/ 0.00	0.00/ 0.00	0.00/ 0.00	0.00/ 8.33	0.00/ 0.00
PES-SMPU	0.00/ 12.50	0.00/ 12.50	0.00/ 41.67	0.00/ 70.00	0.00/ 80.56	0.00/ 76.39
PES-SMPU-CNW5	15.00/ 34.00	25.00/ 20.00	24.17/ 41.90	43.26/ 72.31	33.43/ 100.00	53.03/ 75.00
PES-SMPU-CNW10	33.53/ 45.00	36.49/ 35.00	37.00/ 74.04	40.04/ 73.23	38.03/ 100.00	61.54/ 80.56
PES-SMPU-CNW20	35.00/ 54.17	50.00/ 35.83	42.86/ 100.00	50.89/ 87.50	55.00/ 100.00	75.00/ 92.36

the maximum values, like 75% in dry air and 92.36% in water at 65°C, which are significantly higher than the other treated ones. According to the results, both R_r and R_f ratios for all samples at all temperatures are higher in water than in dry air media. This may be attributed to the higher heat transmission coefficient of water compared to air. The dynamic bagging recovery performances of SMPU and SMPU-CNW treated samples can be explained with the effect of temperature on viscoelastic behaviour and molecular mobility of shape memory polymer and dimensional change of the polymer film on fibre surface[44]. Also, as seen in DMA results, the higher E' at the rubbery plateau for PES-SMPU-CNW20 enabled higher dynamic bagging recovery performances owing to an increase in releasing of the stored entropic energy above the T_{trans} (T_g) in stress-free mode[45]. In addition to these, the abrupt increase in bagging recovery ratio with CNW concentration, maybe attributed to two phenomena, viz probable stretching and distorting of hydrogen bonds of CNWs with temperature[46] and plasticization of the SMPU-CNW nanocomposite as result of water responsive shape memory effect[31, 34-36]. Moreover, higher bagging fixity of all the treated fabrics may result from an increase in fabric elasticity as well as lower fibre-fibre friction by a thicker polymer film layer among fibres and yarns (Fig. 2).

Bagging recovery performances tested under different environmental RH values at a fixed temperature of 20°C are given in Table 4. According to MANOVA results, finishing treatment type, RH, and their interactions has significant effects on bagging recovery performance of the polyester fabrics ($p=0.00<0.05$). Reference sample has the lowest R_r and also R_f ratio (0.00%), meaning that this sample is not affected by RH change of environment. This might be related to the hydrophobic nature of the polyester fibre. On the other hand, bagging recovery performance significantly increased for SMPU and especially SMPU-CNW nanocomposite treated samples with CNW concentration at all RH values. Also, both R_r and R_f ratios of SMPU-CNW nanocomposite treated samples increased abruptly for 65% RH which reached up to 55% and 75% for PES-SMPU-CNW20. According to the results, the SMPU-CNW nanocomposite finishing process results in high bagging recovery and stability performance proportional to the CNW concentration and RH. The dynamic bagging recovery performances of SMPU-CNW treated samples especially PES-SMPU-CNW20 ones can be explained with the effect of plasticization and humidity responsive shape memory effect originated from reversible hydrogen bonding formed within CNWs[31, 34-36].

Table 4 — Bagging recovery (R_r) and fixity (R_f) (%) performances of polyester fabrics according to RH at 20°C

Sample	20% RH		40% RH		65% RH	
	R_r	R_f	R_r	R_f	R_r	R_f
Reference polyester fabric	0.00	0.00	0.00	0.00	0.00	0.00
PES-SMPU	16.50	26.00	28.50	27.50	33.43	53.03
PES-SMPU-CNW5	21.87	27.78	32.50	30.00	38.91	58.71
PES-SMPU-CNW10	25.93	32.59	33.33	33.33	38.03	61.54
PES-SMPU-CNW20	27.50	40.00	35.00	37.29	55.00	75.00

4 Conclusion

This study has been focused on the production of a smart polyester fabric having a dynamic shape memory effect based on dimensional recovery. For this aim, the polyester fabric is treated with dual responsive nanocomposite finishing material consisting of temperature responsive SMPU and CNWs, providing additional water/humidity responsiveness. According to the results, SMPU-CNW nanocomposite treated polyester fabrics could return to their original dimensions dynamically (flat) from undesired deformed form (bagging) as a result of simultaneous temperature and water/moisture responsive shape memory effect. FTIR results confirm bagging recovery results that SMPU and SMPU-CNW nanocomposites with different nanoparticle concentration is grafted chemically onto fibre/fabric surface. Besides, SEM images show that SMPU-CNW nanocomposite is deposited on polyester fabric effectively with a thicker film formation providing higher strength as well as elasticity and bagging fixity values. Meanwhile, thermomechanical properties of the polyester fabric improve with SMPU-CNW nanocomposite finishing treatment according to DMA.

Hence, smart garments having the shape recovery ability to their original flat appearances during standard home washing, drying or wearing conditions at temperatures above T_g of SMPU can be produced with dual responsive SMPU-CNW nanocomposite treatments. Another advantage of the mentioned treatments is their lower side effects on fabric hand and other mechanical properties.

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