



Functional Finishing of Polyester Fabric Using Bentonite Nano-Particles



Amira Abou el-Kheir*¹, Nahed A. Abd El-Ghany², Mona M. Fahmy², Sara E. Aboras³ and Lamiaa K. El-Gabry¹

¹Proteinic and Man-made Fibers Dept., Textile Industries Research Division, National Research Centre, 33 El Bohouth st., Dokki, Cairo, Egypt, P.O. 12622.

²Department of Chemistry, Faculty of Science, Cairo University, Cairo, Egypt.

³Master Student, Chemistry Department, Faculty of Science, Cairo University, Cairo, Egypt.

THIS work is devoted to increase the functional properties of polyester fabric such as UPF, moisture regain, tensile strength at break and dyeability towards basic dye. To achieve this purpose polyester fabric were modified with different concentrations bentonite nanoparticles (BNPs) using pad – dry curing technique and IR dyeing machine. The effect of the (BNPs) on the physical and mechanical properties of the treated fabrics such as moisture regain, tensile strength at break, elongation percentage and thickness were investigated. Topographical investigation of the said nano-particles was conducted using transmission electron microscopy (TEM). The surface morphology and surface chemical elements of the treated as well as the untreated fabrics were investigated using field emission scanning electron microscopy and dispersive X-ray spectroscopy, respectively. FTIR, thermogravimetric analysis and UPF of the treated polyester fabrics compared to the untreated one were assessed. The colour strength of dyed polyester fabrics with both disperse and cationic dyestuffs as well as the fastness properties were evaluated. Excellent results of the treated fabrics toward physical and coloration properties were obtained.

Keywords: Polyester fabrics, BNPs, Physical and Mechanical properties, UPF, Basic dyeability.

Introduction

Polyester fibre (PET), as a very important textile fibre, has been widely used in clothing, home furnishings, and some industrial areas due to its superiorities of high strength, mechanical stability and low cost [1, 2]. However, polyester fabric suffers from several drawbacks such as its hydrophobic nature, pilling problem and very difficult in dyeing which diminish their comfort attributes. Many attempts have been made to enhance and acquire desired properties to polyester fibres including utilization of low temperature atmospheric plasma to increase its wettability and dyeability. It was reported that the PVA treated polyester showed improved hydrophilic properties over intact and sodium

hydroxide treated polyester fibres.

Recently, it has been demonstrated that inorganic nanoparticles are promising fillers in the modification of polyester. A very small amount of modified nanoparticles can effectively improve the modulus, strength, toughness, and thermal deformation temperature of the polymers [3]. Nanoclays are one of the most important industrial nano particles as they are inexpensive, widely available in nature and environment friendly. Abou El-Kheir et al. was reported that the treatment of the viscose fabrics with sodium polyacrylate/kaolin nano-composite improved the functional properties such as, tear strength as well as antimicrobial activity and increasing its dyeability to a great extent [4]. Also, El-

*Corresponding author e-mail: amiraadell@yahoo.com

Received 2/12/2019; Accepted 23/12/2019

DOI: 10.21608/ejchem.2019.20404.2223

©2020 National Information and Documentation Center (NIDOC)

Gabry et al found that the biocidal activity was increased due to the treatment of viscose fabric with different types of organic and inorganic nanostructural materials [5].

Bentonite, highly plastic clay, is one of the nanoclay containing about 85% clay mineral, montmorillonite. The commercial importance of bentonite depended on its physicochemical properties rather than its chemical composition, such as excellent plasticity and lubricity, high dry-bonding strength, high shear and compressive strength, low permeability and low compressibility. Bentonite is an absorbent aluminium phyllosilicate, and have excellent rheological and absorbent properties [6].

In this work, BNPs was chosen to treat polyester fabric to investigate its effect on some physical and mechanical properties such as hydrophilicity, UV protection and dyeability towards disperse and cationic dyes. This work is devoted to incorporate BNPs into the polymer matrix of polyester using different techniques and study its effect on phsicomechanical properties like moisture regain, UPF, tensile strength, elongation % and dyeability to both disperse and basic dyes.

Experimental

Materials

Polyester fabrics was supplied from Misr Spinning and Weaving Co., El Mahalla El Kobra, Egypt. A plain woven polyester fabric weighing is 155 g/m².

Chemicals

Bentonite nano-particles (BNPs) was supplied by (purchased from) Sigma–Aldrich, Germany. All other chemicals were of laboratory grade and used without further purification.

Dyestuffs

Disperse dyestuff Samaron Pink HFG (C.I. Disperse Red 185) and Remacryl Red BRL (C.I. Basic Red 18) were used.

Methods

Scouring of polyester

Polyester fabrics were scoured with (2 g/L) nonionic detergent solution (Hostapal C V. from Clariant, Egypt) with a liquor ratio 1: 25, at 45 °C, for 30 min, then rinsed twice in cold tap water, and dried at room temperature.

Modification of polyester fibres with sodium hydroxide

Egypt. J. Chem. **63**, No. 1 (2020)

Polyester fabrics were treated with different concentrations of aqueous sodium hydroxide (2%, 4%, 6%, 8%, 10% v/v) with liquor ratio 1:100 at 80°C for an hour with occasional shaking. The treated samples were immediately and thoroughly washed with water for complete removal of alkali. The final washings were checked with a pH paper until neutral and the samples were air-dried. The loss in weight of the pretreated samples compared to the untreated according to the following equation:

$$\text{Loss of weight\%} = \frac{W_{tb} - W_{ta}}{W_{tb}} \times 100$$

W_{tb} weight of polyester fibres before treatment,
 W_{ta} weight of polyester fibres after treatment.

Base combining capacity

The base combining capacity was estimated by measuring the amount of alkali combined with polyester material as follows [7]:

- The sample was soaked in 2% hydrochloric acid for 3-4 hr with occasional shaking. The sample was filtered and washed several times with ethanol/water mixture (60–40) until chloride ions are free. Then the sample was filtered and dried.
- The dry sample (0.5 g) was precisely weighed and introduced in 250 ml Erlenmeyer flask, followed by 50 ml 0.1N sodium hydroxide solution containing 5% sodium chloride. The flask was Stoppard and allowed to stand overnight with occasional shaking. The content of the flask was back titrated with 0.05 N hydrochloric acid using phenolphthalein as indicator. Blank titration was carried out on the untreated sample, and the carboxyl content of the sample was determined as follows:

$$\text{Carboxyl content} = \frac{(X - Y)N_A}{W} \times 100 \text{ meq/100 g fabric}$$

Where; X is the volume of HCl solution used in titration of control sample,

Y is the volume of HCl solution used in back titration,

N_A is the normality of HCl solution, and W is the weight of fabric sample (g).

Treatment of polyester fibres with nano-materials

Alkali-treated (pretreated) and the untreated polyester fibres were treated with different concentrations of BNPs using pad-dry curing and IR dyeing machines.

Treatment of polyester fibres with BNPs using pad-dry cure technique

Different amounts of BNPs (1%, 3%, 5% wt./v) were dispersed in distilled water and homogenized for one hour using ultrasonic homogenizer (100 watt). Pretreated polyester fabrics as well as the untreated one were modified with the different concentrations of BNPs using pad dry cure technique. The treated fabrics were padded in two dips and two nips to a wet pick up of 100% followed by drying at 80°C for 5 min then cured at 160°C for 3 min.

Treatment of polyester fabrics with BNPs using IR dyeing machine

Untreated and pretreated polyester fabrics were treated with the said concentrations of BNPs dispersed solutions (1%, 3%, 5% wt/v) with liquor ratio 1:100 using (PYROTEC³ of IR its company of ROACHES made in the UK), the temperature of the IR device was adjusted at 130 °C. After one hour the treated samples were rinsed with running water and left to dry at ambient temperature.

Dyeing process

Dyeing with basic dye

The dye bath solution was prepared using required amount of dye C.I. Basic red 18 with some warm water to give the prescribed shade (1% (o. w. f.) and diluting with water to completely soluble dye. The dye solution was adjusted to pH 5. The dyeing process was carried out at 85°C for an hour with occasional shaking and the liquor ratio was 1:100. After that, all the dyed samples were withdrawn, rinsed thoroughly with warm water and air-dried.

Dyeing with disperse dye

The untreated, pretreated and BNPs treated polyester fabrics were dyed with disperse dyestuff (C.I. Disperse Red 185). Required amount of C.I. Disperse Red 185 was dissolved in 1% acetic acid, and then added 2g/l of the carrier to prepare (1% O.W.F.) dye solution. The pH of the bath was adjusted at pH 5, and then the temperature was gradually heated to 98°C. The sample were added to the bath and the dyeing continued for 60 min., with liquor ratio 1:100 Finally, The dyed samples

were thoroughly washed in warm and cold water and kept to dry at room temperature.

Characterizations & Measurements of Nano bentonite-treated polyester fibres

Transmission electron microscopy (TEM)

The morphology of the BNPs was investigated using TEM (JEOL, JEM-1230 Japan, with an acceleration voltage of 120 kV). The sample for TEM analysis was obtained by placing a drop of the colloid dispersion onto a carbon-coated copper grid. The sample was dried at room temperature and examined using a TEM without further modification or coating.

Field Emission Scanning electron microscopy (FE-SEM) and dispersive X-ray spectroscopy (EDX)

Quanta FEG 250 scanning electron microscopy (FE-SEM) with 30 kV scanning voltages was employed to observe the morphologies of untreated and treated fabrics. Quanta FEG 250 with Oxford Instruments EDX with INCA software system. EDX measurement conditions, 20 kV accelerating voltage, 21 mm working distance, 1 nA sample.

FTIR Analysis

Infrared Spectra were recorded on FT-IR Nicolet 5 DX Spectrophotometer. The samples were examined as 1.5% KBr pellets.

Thermogravimetric analysis (TGA)

From each sample of 4-5 mg were cut and used for thermo-gravimetric analysis experiments. ANETZSCH TGA 209 thermo-balance, airflow of 10.00 cm³ min⁻¹ and aluminum oxide crucibles were used for thermo-gravimetric measurements. The temperature range was 20 to 700°C, and the heating rates were 10°C min⁻¹.

UV-Protection

The UPF of untreated and finished fabrics (size 3 cm× 3 cm) was determined according to the Australian/New Zealand standard (AS/NZS 4366-1996) using UV-Shimadzu 3101 PC spectrophotometer at wavelength of 280 to 390 nm, which includes the UVB (280 to 320 nm) and the UVA (320 to 400 nm) according to the following equation:

$$UPF_t = \frac{\sum_{\lambda=280}^{400} E_{\lambda} \times S_{\lambda} \times \Delta\lambda}{\sum_{\lambda=280}^{400} E_{\lambda} \times S_{\lambda} \times T_{\lambda} \times \Delta\lambda}$$

Where, E_{λ} : relative erythema spectral effectiveness, S_{λ} : solar spectral irradiance, T_{λ} : average spectral transmission of the specimen, and $\Delta\lambda$: measured wavelength interval (nm).

Tensile strength and Elongation %

The tensile strength and elongation percentage of fabric before and after treatment were evaluated using an Instron Tensile Tester (USA) according to ASTM D 76 Standard Specification for Textile Testing Machines. The average dimensions of the used samples were (5×20 cm).

Moisture Regain %

Measurements of moisture regain of the fibres were performed using the standard ASTM method 2654-76 (West, 1981). Moisture regain of the samples was calculated according to the following equation:

$$\text{Moisture regain \%} = \frac{W_1 - W_2}{W_2} \times 100$$

Where W_1 is the weight of the sample (g) after saturation in the standard humidity atmosphere; W_2 is the constant weight (g) of dry sample.

Colour intensity (K/S)

Spectral reflectance measurements of the dyed samples were measured using UV/Vis spectrophotometer (Hunter lab, Ultra Scan Pro, USA). The color values expressed as K/S, values of the dyed samples were determined by applying Kubelka–Munk Eq. 1 [8].

$$K/S = \frac{(1-R)^2}{2R} - \frac{(1-R_0)^2}{2R_0}$$

Hydrolysis of polyester with sodium hydroxide

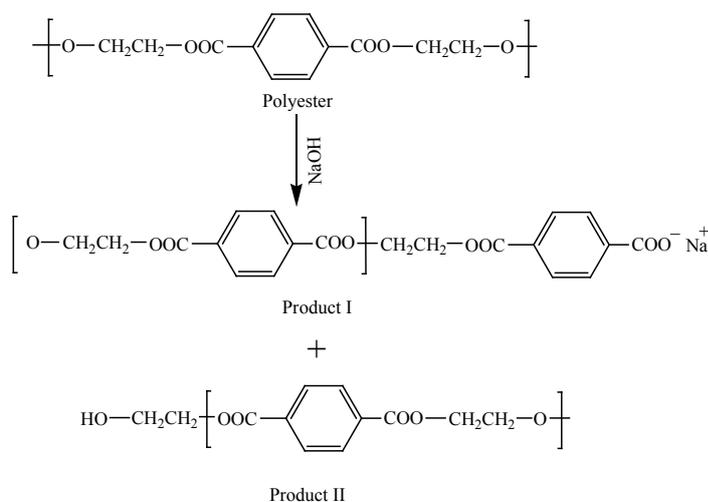


Fig.1: Hydrolysis of polyester fabrics using sodium hydroxide product I and II: active hydrolyzed polyester fabrics

Where R is the decimal fraction of the reflectance of the dyed substrate, R_0 is the decimal fraction of the reflectance of the undyed substrate, S is the scattering coefficient, K is the absorption coefficient.

Washing fastness

The color fastness to washing was determined according to the AATCC test method (AATCC Technical Manual, Method 36, (1972), 68, 23, (1993)) using Launder Ometer[9]. The colour was determined according to BS 1006: CO₂ test 2 with the use of soap solution (5 g/l, liquor ratio 1:50) for 45 minutes at 50°C ± 2°C[7].

Results and Discussions

In his work, polyester fibres were treated with different techniques and different concentration of BNPs. The untreated as well as pretreated polyester were submitted to measure the chemical composition, mechanical properties, physical properties and dyeability in addition to measuring particle size of the used nano materials. Results obtained along with appropriate discussion are given below.

Topographical study (TEM)

Figure 2 show the Transmission Electron Micrographs (TEM) of BNPs powder. This figure implies that the size of the BNPs (NB) powder is within the nano range (17–58 nm), which ensures the better dispersion of NB within the treated fabrics.

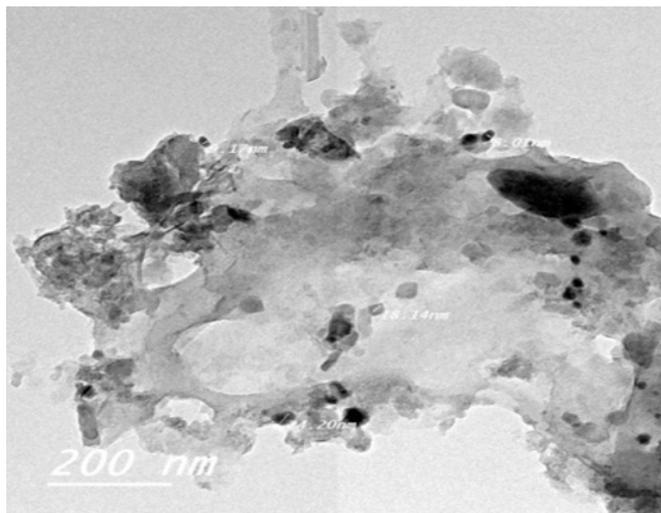


Fig. 2: TEM of BNPs

Field emission scanning electron microscopy (FE-SEM) and dispersive X-ray spectroscopy (EDX)

Morphological structure of untreated as well as treated polyester fibres were investigated using field emission scanning electron microscopy. The FE-SEM image for the untreated polyester fabric indicate a clean and smooth longitudinal fibril structure surface (Figure 3 a).

Figure 3(b-e) indicate the presence of BNPs on the surface and between the fibre filament of polyester fabrics treated with 8% sodium hydroxide followed by 5% BNPs using different techniques (IR machine, and pad dry cure).

It is obvious that the amount NB covers the surface of the samples treated with pad dry cure technique (fig. 3, c & e) is higher than that when using the IR dyeing machine (fig 3. b & d), This result may be referred to the high temperature used in the curing step (160 °C), results in increasing the swelling of the fabrics, which permitting considerable amount of NB to penetrate the fiber filament and deposit on their surface. Figure 3 shows also the sample treated with 8% NaOH followed by 5% bentonite is covered with large amount of bentonite which may be due to the opening of the polymeric structure due to the pretreatment of polyester fibres with NaOH.

Surface chemical elements of the treated fabrics were assessed by EDAX spectroscopy. Figure 4 shows EDX spectra for treated as well as untreated polyester fibres. Fig. 4 (a) show the carbon and oxygen peaks which are belonging to the native polyester fibres.

The peak appeared during the investigation of sample treated with 8% sodium hydroxide then 5% BNPs by IR service technique Fig. 4(b) indicating the presence of Si, Al, Mg, Ca, Fe, Ti and Mn elements of 1.97, 0.51, 0.07, 2.28, 13.37, 2.89 and 2.47% respectively. Fig.4(c) shows the spectra of the sample treated with 8% sodium hydroxide and 5% BNPs by pad dry cure technique which are attributed to Si, Al, Na, Mg, Fe and Ca of 3.69, 1.79, 0.37, 0.31, 1.61 and 0.39% respectively. Fig. 4(d) shows the spectra of sample treated with 5% BNPs by IR service technique, which are attributed to Si, Al, Mg, Fe and Ca of 1.63, 0.78, 0.13, 0.77 and 0.41% respectively. Fig. 4 (e5) shows the spectra of sample treated with 5% BNPs by pad dry cure technique, which are attributed to Si, Al, Mg, Fe and Ca of 7.62, 3.77, 0.64, 2.78 and 1.17% respectively. SEM and EDAX results indicated the presence of NB in the treated polyester fibres samples.

Base combining capacity

The base-combining capacity of polyester fabrics, hydrolyzed with sodium hydroxide, is a function of the free acidic groups found along with its macromolecules. In this study, polyester fabrics were partially hydrolysed with different concentrations of sodium hydroxide (4, 6, 8, and 10%) to increase the polar groups on the fibre surface, namely hydroxyl and carboxyl groups [7], which in turn increase the moisture regain of the treated fabrics to great extent. The base combining capacity content of the alkali-treated fabrics was determined and the obtained results were tabulated in Table 1. Data of this investigation illustrated that, the carboxylic content of the

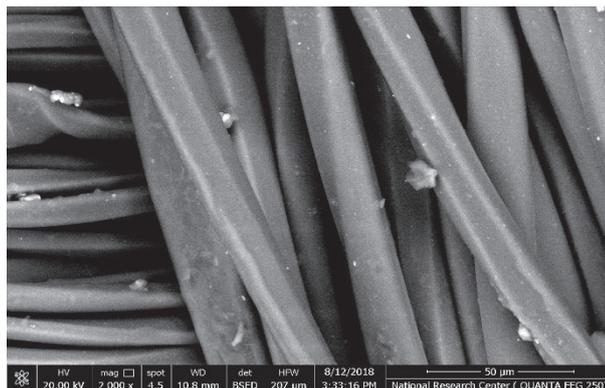


Fig. 3 (a): Untreated polyester fibres.

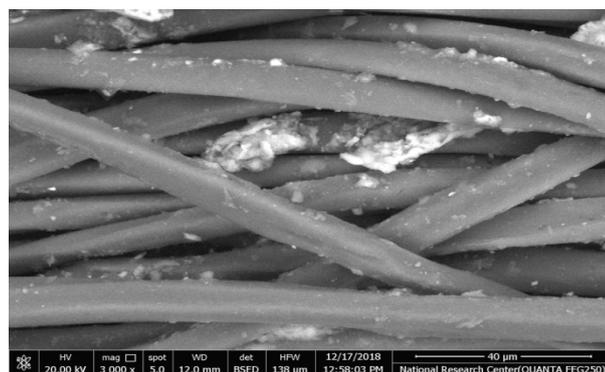


Fig. 3 (b): Treated sample with 8% sodium hydroxide followed by 5% BNPs by IR service technique

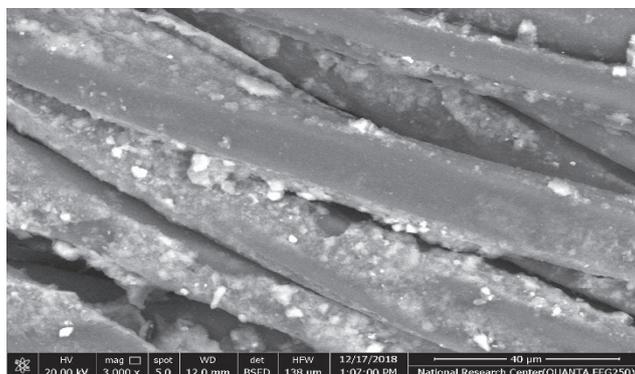


Fig. 3 (c): Treated sample with 8% sodium hydroxide followed by 5% BNPs by Pad dry cure technique.

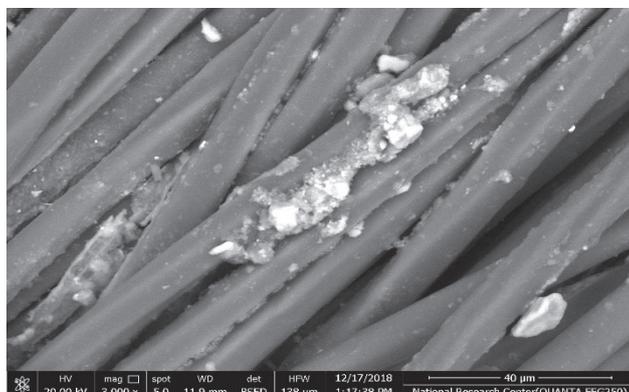


Fig. 3(d): Treated sample with 5% BNPs by IR service technique.

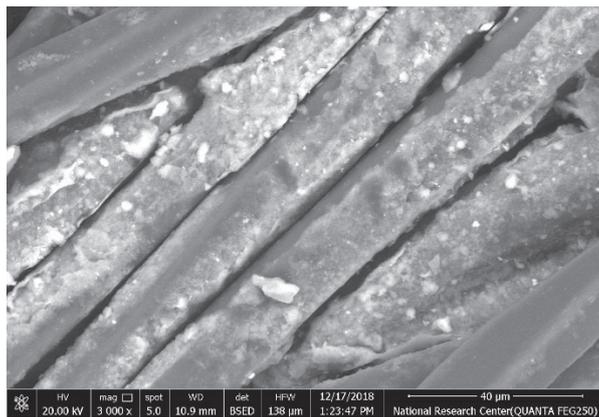
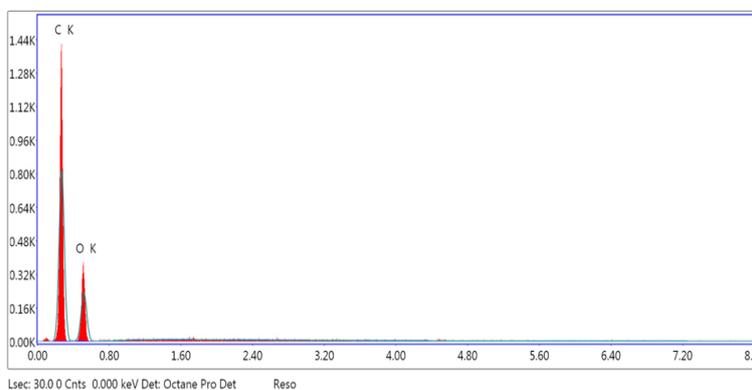


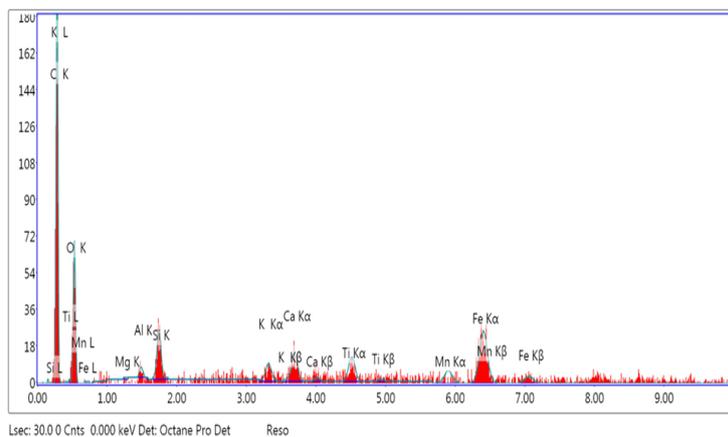
Fig. 3 (e): Treated Sample with 5% BNPs by Pad dry cure technique.

Fig. 3: (FE-SEM) Scanning electron microscopy of untreated and treated polyester fibres by different techniques (pad dry cure and IR dyeing machine).



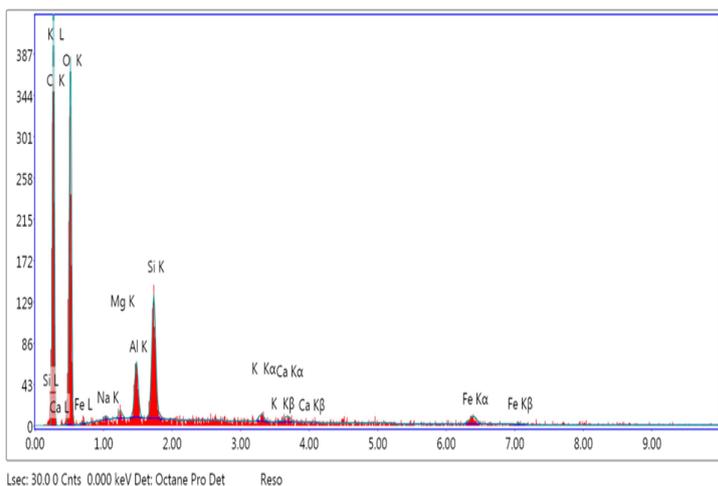
Element	Weight %	Atomic %
C K	63.9	70.22
O K	36.1	29.78

Fig.4 (a)



Element	Weight %	Atomic %
C K	49.66	66.07
O K	25.58	25.55
MgK	0.07	0.05
AlK	0.51	0.3
SiK	1.97	1.12
K K	1.21	0.49
CaK	2.28	0.91
TiK	2.89	0.96
MnK	2.47	0.72
FeK	13.37	3.83

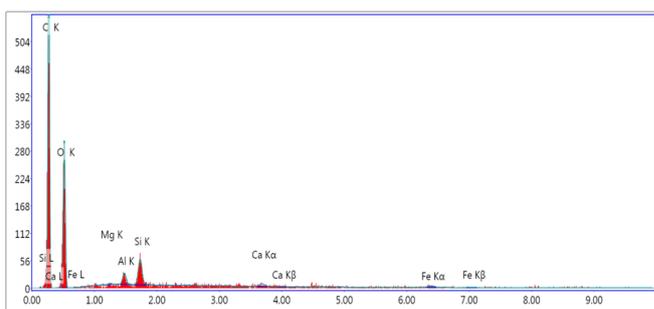
Fig.4 (b)



Lsec: 30.0 0 Cnts 0.000 keV Det: Octane Pro Det Reso

Element	Weight %	Atomic %
C K	48.32	57.52
O K	43.12	38.54
NaK	0.37	0.23
MgK	0.31	0.18
AlK	1.79	0.95
SiK	3.69	1.88
K K	0.39	0.14
CaK	0.39	0.14
FeK	1.61	0.41

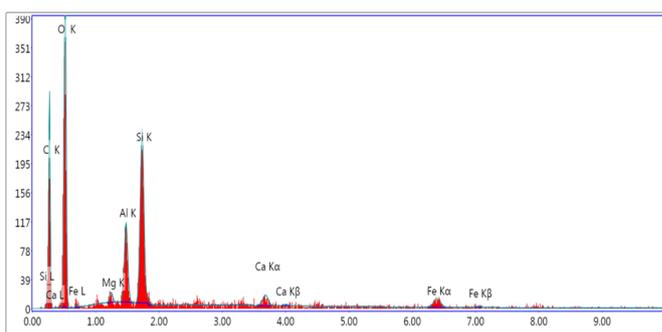
Fig. 4 (c)



Lsec: 30.0 0 Cnts 0.000 keV Det: Octane Pro Det Reso

Element	Weight %	Atomic %
C K	55.62	63.53
O K	40.66	34.87
MgK	0.13	0.07
AlK	0.78	0.4
SiK	1.63	0.79
CaK	0.41	0.14
FeK	0.77	0.19

Fig. 4 (d)



Lsec: 30.0 0 Cnts 0.000 keV Det: Octane Pro Det Reso

Element	Weight %	Atomic %
C K	37.26	47.43
O K	46.75	44.67
MgK	0.64	0.4
AlK	3.77	2.14
SiK	7.62	4.15
CaK	1.17	0.45
FeK	2.78	0.76

Fig.4 (e)

Fig. 4: EDX of untreated as well as treated polyester fibres (a) EDX of untreated polyester fibres, and (b) of the treated polyester fibres with 8% sodium hydroxide followed by 5% BNPs by IR service technique. (c) Treated polyester fibres with 8% sodium hydroxide followed by 5% BNPs by Pad dry cure technique. (d) Treated polyester fibres with 5% BNPs by IR service technique. (e) Treated polyester fibres with 5% BNPs by Pad dry cure technique.

alkali treated fabrics increased gradually as the concentration of sodium hydroxide increased. It was indicated that, the best concentration of sodium hydroxide used in the alkali treatment of polyester fabrics is 10% as it showed the higher value of carboxylic content (31%).

Moreover data of Table 1 illustrate the percentage loss in weight of the polyester fabrics after hydrolysis with different concentration of sodium hydroxide. It was found that as the concentration of sodium hydroxide increases the loss in weight increases. This result is due to the formation of disodium terephthalate, which is soluble in alkaline solution. It is produced as a result of the topo chemical reaction of sodium hydroxide in which the hydroxyl ions of NaOH attack the carbonyl group of surface PET fibre forming disodium terephthalate and ethylene glycol [10].

FTIR analysis

Figure 5 Showed the FTIR spectra of untreated PET, 8%NaOH pretreated PET and that treated with 5% BNPs. Untreated PET spectrum showed a characteristic peaks at 3622, 3427 cm^{-1} related to the stretching vibration of water and carboxylic acid OH groups respectively. While the peaks at 2961, 2917 and 2852 cm^{-1} are corresponding to CH_2 Stretching vibrations, the strong peaks at 1711 and 1468 cm^{-1} are related to the asymmetrical and symmetrical vibrations of the carboxylic ester $\text{C}=\text{O}$ groups respectively. The peaks at 1578, 1504 and 720 cm^{-1} are assigned to the $\text{C}=\text{C}$ of the benzene ring, while the peak at 1339 is due to bending and wagging vibrational modes of the ethylene glycol segment [11]. The characteristic peaks at 1241, 1094 and 1017 cm^{-1} are due to $\text{C}-\text{O}$, $\text{C}-\text{O}-\text{C}$ and $\text{C}-\text{OH}$ respectively, while the peaks appeared at 871 and 847 cm^{-1} are related to the p-substituted benzene ring Figure 5(a). Spectrum of PET treated with 8% NaOH showed in addition to the mentioned peaks, broad

peaks around 3539 and 1455 cm^{-1} related to the hydrogen bonded OH group and $-\text{COO}^-$ group resulted from the alkaline hydrolysis respectively Figure 5 (b). IR Spectrum of PET/5% Bentonite and PET/8% NaOH/5% Bentonite showed additional peaks at 3694 cm^{-1} related to hydroxyl groups of bentonite structure ($\text{Si}-\text{OH}$, $\text{Al}-\text{OH}$) and the peak around 3619 is attributed to the adsorbed water. The peaks in the range between 900 to 700 cm^{-1} is related to the $\text{Si}-\text{O}$ group while that at 522 and 465 cm^{-1} are attributed to $\text{Al}-\text{O}-\text{Si}$ and $\text{Si}-\text{O}-\text{Si}$ groups respectively Figure 5 (c, d). These data are in accordance to that in literature [12]. Moreover the ionic interaction between Bentonite and PET functional groups is supported from the shift of some vibrations to lower frequencies, as the peak at 3427 cm^{-1} (OH), and 1094 cm^{-1} ($\text{C}-\text{O}-\text{C}$) were shifted to 3720, 1086 cm^{-1} and to 3422, 1090 cm^{-1} in spectrum of PET/ 5% BNPs and PET/ 8%NaOH/ 5% BNPs, respectively.

Thermogravimetric analysis (TGA)

Figure 6 (a, b) showed the thermal stability behavior of untreated PET, treated PET/ 8%NaOH and treated PET/ NaOH/ 5%BNPs using thermogravimetric analysis (TG). From the diagram the TG/DTGA curves of all samples exhibited weight loss about 5% at temperature range between 50- 150 $^{\circ}\text{C}$ related to the evaporation of water and volatile substances [12]. The main degradation of untreated PET sample started at T_{on} set around at 387 $^{\circ}\text{C}$, might result from the removal of the vinyl ester and acid end groups and formation of cyclic oligomers. The DTG curve indicated T_{max} at 428 $^{\circ}\text{C}$ related to the degradation of the cyclic oligomer and release of acetaldehyde and anhydrides oligomers [13], the residual mass at maximum degradation was 22% of the original mass. Treated of PET with 8% NaOH resulted in a great number of OH and COOH groups on the polymeric chains and thus formation of an intermolecular hydrogen bonds which consumed much heat for degradation leading to increase in

TABLE 1. The base combining capacity of polyester fabrics hydrolyzed with sodium hydroxide

Concentration of sodium hydroxide (%)	Base combining capacity (meg./ 100g) woven sample	Loss in for pretreated polyester fibre (wt.%)
0 (untreated sample)	5	0
4	24	5.27
6	27	10.66
8	29	13.79
10	31	16.89

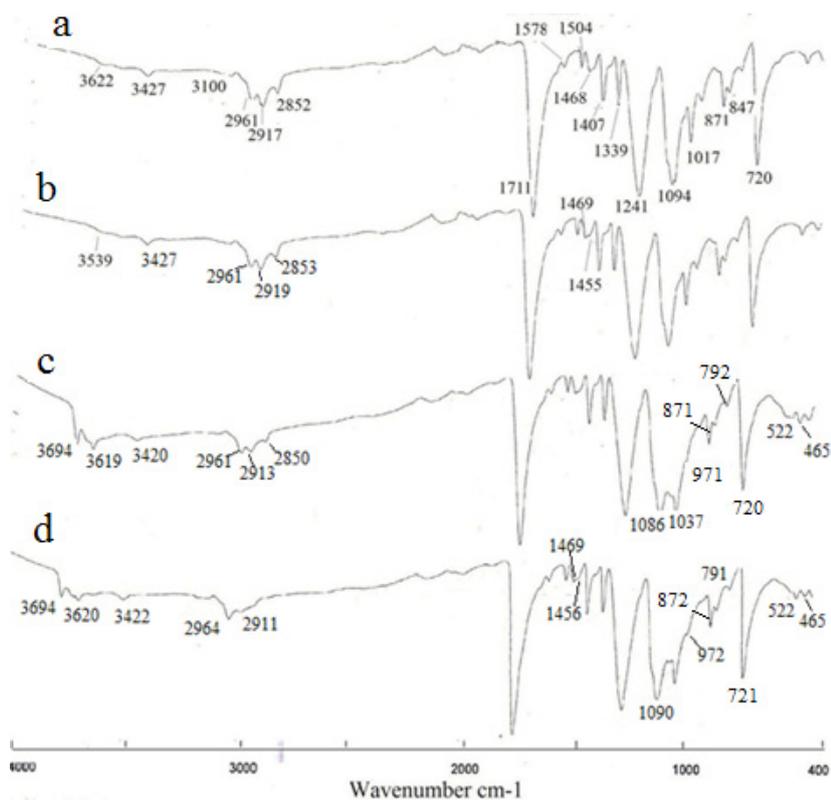


Fig. 5. FTIR spectra of (a) untreated PET, (b) PET/ 8%NaOH, (c) PET/ 5% Bentonite and (d) PET/ 8%NaOH/ 5% Bentonite.

the T onset to 410 °C and T max appeared at 444 °C with residual mass of 20%. The decrease in the residual mass might result from the effect of NaOH in solubilization of the short chains of PET matrix. Addition of bentonite in 5% concentration (based on PET sample weight) before and after treatment with NaOH increases the thermal stability of PET as indicated from the TG/ DTGA curves, since the residual mass increased from 22% to 24 % for PET / 5% Bentonite sample and from 20% to 26% for PET/ 8%NaOH/ 5% bentonite sample. It seemed that the stabilizing action of Bentonite on PET chains may result from; (1) the generated hydrogen bonds between BNPs constituents and the PET OH, COOH function groups; (2) the ionic interaction between the Si-O and Al-O of Bentonite with the PET function groups. The entire T onsets, T max, T ends and Residual masses were summarized in Table 1. *Ultraviolet Protection Factor (UPF)*

Ultraviolet protective factor (UPF) measures the effectiveness of textile fabrics in protecting the human skin from UV radiation (UVA and UVB radiations). Excessive exposure of the skin

to UV radiation might be carcinogenic resulting in chronic reactions and injury, accelerated ageing of the skin [14]. An overdose of UV radiation leads to acute and chronic reactions, skin reddening (erythema) or sunburn, increasing the risk factor of persons susceptible to melanoma and skin cancer [15].

Data in Table 3 illustrate the ultraviolet protection factors of the treated as well as untreated polyester samples. It is found that the treated samples have excellent resistance to ultraviolet radiation compared to the untreated one. Data in Table 3 clarify that the untreated and alkali treated samples have no protection for UVA and UVB radiations, on contrary the samples treated with 5% bentonite have a very good protection for both UVA and UVB radiations (UPF 80% and 68%) for both pad dry cure technique and IR SERVICE technique respectively.

These results may be due to the polyester fabrics were coated with a thin layer of the said nano-material and subsequent minimization of the porosity within the fibre vicinity, which acts as a barrier, thus prevents the UV radiation to penetrate

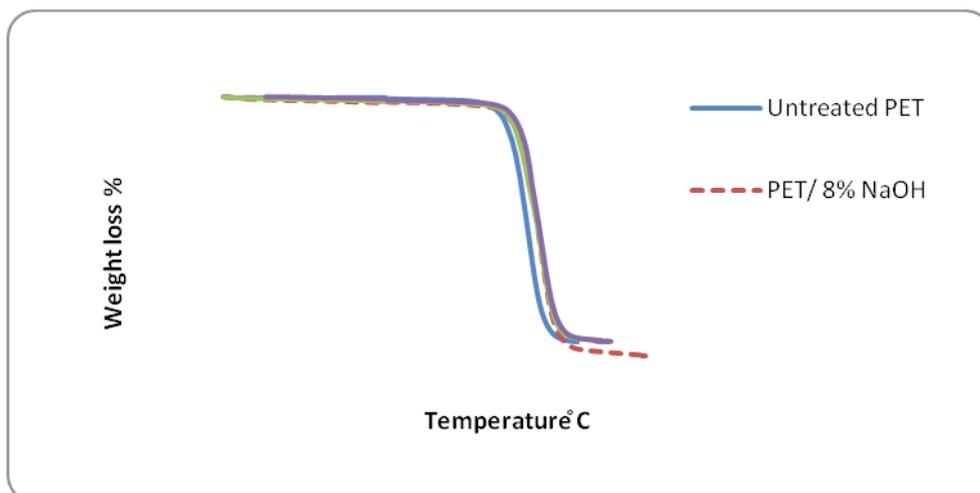


Fig. 6 (a):TGA diagram of PET, PET/ 8% NaOH, PET/ 5% BNPs and PET/ 8% NaOH/ 5% BNPs.

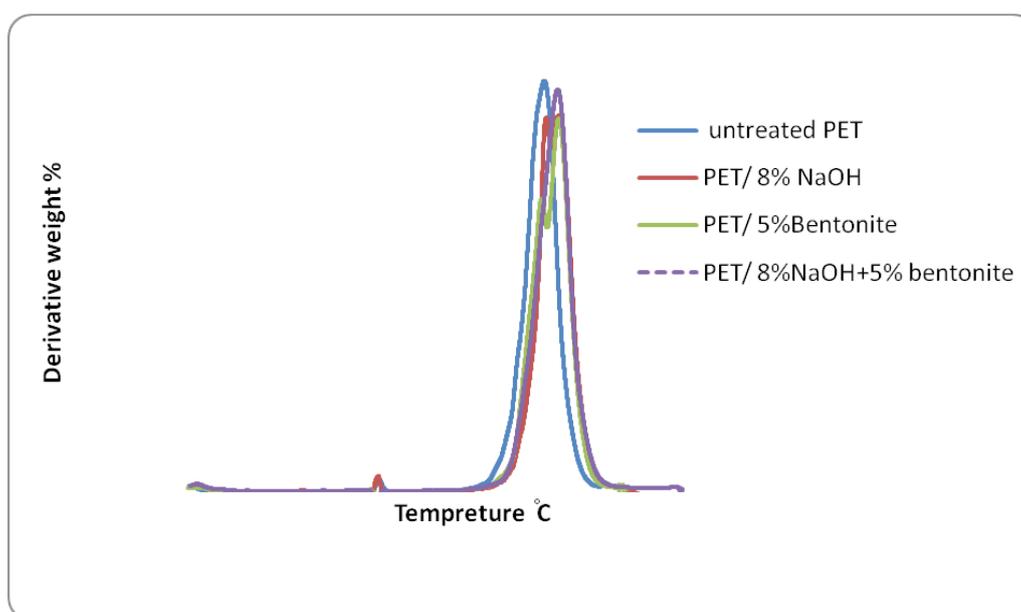


Fig. 6 (b):DTGA diagram of PET, PET/ 8% NaOH, PET/ 5%BNPs and PET/ 8%NaOH/ 5%BNPs.

TABLE 2: TG/DTG properties of the specimens

Samples Description	T _{onset} (°C)	T _{max} (°C)	T _{end} (°C)	Residual weight %
Untreated PET	387	428	450	22
PET/8%NaOH	410	444	470	20
PET/5%BNPs	400	446	465	24
PET/8%NaOH+5% BNPs	385	443	470	26

TABLE 3: percentage UPF of treated as well as untreated polyester fibres

Samples Description	% Increase in UPF			
	Pad Dry cure technique	Grade	IR service technique	Grade
Untreated polyester fibres	8	No protection	8	No protection
Treated with 8% NaOH	10	Poor protection	10	Poor protection
8%NaOH+5%BNPs	33	Very Good protection	19	Very good protection
PET+1% BNPs	45	Very good protection	38	Very good protection
PET+3% BNPs	58	Excellent protection	53	Excellent protection
PET +5% BNPs	80	Excellent protection	68	Excellent protection

the treated samples [16]. Besides, the presence of aluminum and silicon oxides increases the protection of treated fabrics for UV radiation [17]. Virtually, it can be concluded from these results that treatment of polyester fibres with BNPs resulted in enhancement of the % UPF of the said fibres.

Tensile strength & Elongation %, Thickness and moisture regain of untreated as well as treated polyester fibres.

Data of Table 4 reveal that the tensile strength and elongation percentage of the treated samples as well as the untreated one by different techniques (Pad dry cure technique & IR dyeing machine service technique). The results of the two techniques clarify that the tensile strength of the treated fabric with different concentrations of BNPs and with 8% NaOH/5%BNPs increased compared to the untreated one. Furthermore, Data of Table 4 illustrate that the samples treated with 1, 3 and 5%BNP enhance the tensile strength of about 7.4, 8.7 and 10.5%, respectively. This result may be attributed to the increase of the interfacial adhesion between the BNPs and the fibre filaments after the treatment due to the dispersion of NB throughout the polyester fibres. In addition, data of table 4 reveal that the polyester fibres treated with 8% sodium hydroxide decreases the tensile strength of about 15.4%. This result may be referred to the topochemical reaction of sodium hydroxide in which the hydroxyl ions of NaOH attack the carbonyl group of PET fibre forming disodium terephthalate and ethylene glycol, which are soluble in alkaline medium. Moreover, NaOH dissolved the short chain found on the fibre surface.

Data of Table 4 reveal also, that the thickness of treated samples as well as the untreated one

treated with different techniques. The results of this table showed that the thickness of all samples are comparable of each other.

Moisture regain of untreated as well as treated polyester fibres are given in Table 4. It is obvious that the pretreated and treated fabrics have moisture regains better than that comparing with the untreated one.

Data of table 4 clarify that fabrics treated with different concentration of BNPs have moisture regain values higher than that of the untreated fibres. Also, it was obvious from results in Table 4 that as the BNPs concentrations increase the value of moisture regain also increase. These results may be referred to the hydrophilic behavior of the BNPs, where the hydration causes the galleries to expand and the clay to swell, so increasing the moisture regain percentage values.

Dyeing Behaviour

Colour intensity (K/S) With Disperse Dyes

Table 5 revealed that the colour intensity of untreated and treated polyester fabrics with C.I. Disperse Pink dye using different techniques (Pad dry cure technique & IR dyeing machine). The result of table 5 showed remarkable increase of the colour intensity compared to the blank sample. It was found that the higher K/S value was obtained of the sample treated with 5% BNPs and the lowest value of the sample treated with sodium hydroxide. These results could be attributed to the swelling properties of BNPs where the fibre swell to more extent compared to the untreated one and enhances the dye solution to penetrate the fibre polymer system, besides the formation of hydrogen bond and van der Waals forces between the dye and polymeric chains of polyester fibres.

TABLE 4: Tensile strength & Elongation % Thickness and Moisture regain % of treated and untreated polyester fibres.

Samples Description	Tensile Strength (kg /cm ²)		Elongation %		Thickness (mm)		Moisture regain %	
	Pad. Dry cure	IR dyeing machine	Pad. Dry cure	IR service	Pad Dry cure	IR dyeing machine	Pad dry cure	IR dyeing machine
Untreated polyester fibres	0.78	0.78	26	26	0.24	0.24	0.4	0.4
Treated with 8% NaOH	0.66	0.66	35	35	0.19	0.19	1.6	1.6
8%NaOH+5%BNPs	0.7094	0.7159	24.165	32.512	0.24	0.25	2.9	2.5
PET +1% BNPs	0.8376	0.7764	28.235	22.353	0.24	0.28	1.9	1.7
PET +3% BNPs	0.8480	0.7990	25.294	20.882	0.24	0.29	2.7	2.3
PET +5% BNPs	0.8619	0.8071	22.353	30.688	0.24	0.28	3.5	3.1

TABLE 5: Colour strength with disperses dye of untreated and treated polyester fibres

Samples Description	Pad dry cure		IR(PYROTEC3) service	
	K/S value at $\lambda_{max.}= 540nm$	Increase relative K/S%	K/S value at $\lambda_{max.}=$ 540nm	Increase relative K/S%
Untreated polyester fibres	2.54	0	2.54	0
Treated with 8% NaOH	2.58	1.57	2.58	1.57
8% NaOH +5% BNPs	3.35	32	2.75	8.3
PET +1% BNPs	2.84	12	2.84	11.8
PET+3% BNPs	3.07	22	2.85	12.2
PET +5% BNPs	3.59	41	2.88	13.4

Colour intensity (K/S) With Red Basic Dyes

Table 6 revealed that the colour intensity of untreated and treated polyester fibres with C.I. Basic Red 18 dye at maximum wavelength 585nm and PH 5. Data in Table 6 illustrate that the K/S values of the treated fibres increase to a great extent compared to the untreated one. It was found that the sample pretreated with NaOH has K/S of 4.67 compared to the 2.1 for the untreated sample. This result is due to the formation of carboxylate ions after the treatment with sodium hydroxide which increases the dye uptake toward basic dyes. Data of table 6 clarify that the highest colour intensity were obtained for the samples treated with BNPs as the K/S values of 1, 3, and 5% BNPs concentration were 6.92, 7.88 and 7.93 respectively compared to 2.1 for untreated one. This outcome is attributed to the isomorphs substitution of Al³⁺ for Si⁴⁺ in the tetrahedral sheet and Mg²⁺, Fe²⁺ cations for Al³⁺ in the octahedral sheet of bentonite results in a net negative charge on its surface. So that, dye uptake was carried out through the electrostatic attraction between the

dye cations and the negatively charged surface of the treated polyester fibres with BNPs.

Washing fastness

Washing fastness of disperse dye

Data of Table 7 show that the washing fastness of the dyed fabrics with C.I. Disperse Red 185 of the untreated, pretreated and treated polyester fabrics with BNPs. The results show that the treatment with 8% NaOH/ 5% BNPs improved the washing fastness of polyester fabrics overall. It was found that the values of alteration improved with the treatments with both techniques, despite of the staining decreases for all treated fabrics with IR (PYROTEC3) service technique compared with untreated one.

Washing fastness of Red Basic dye

Untreated polyester fibres were not dyed with basic dye and therefore no contaminated fibres were expected to be stained. The results show that washing fastness of the polyester fibres give good to excellent with rating 3–4 and 5 and the best washing fastness rating was obtained on pretreated

TABLE 6: Colour strength with the red basic dye of untreated and treated polyester fibres.

Samples Description	K/S value at $\lambda_{\max} = 585\text{nm}$	
	Pad. Dry curing	IR (PYROTEC ³) service
Untreated polyester fibres	2.10	2.10
Treated with 8% NaOH	4.67	4.67
8% NaOH + 5%BNPs	6.20	4.79
PET+1% BNPs	6.92	7.16
PET +3% BNPs	7.88	7.51
PET+5% BNPs	7.93	7.59

TABLE 7: Washing fastness of untreated and treated polyester fabrics with disperse dyes

Samples Description	Pad. Dry curing		IR (PYROTEC ³) service	
	Alt	St _p	Alt	St _p
Untreated polyester fibres	3	4-5	3-4	4-5
Treated with 8% NaOH	3	4	3	4
8%NaOH+5%BNPs	4	4	4	5
PET +1% BNPs	4	4-5	4	4-5
PET +3%BNPs	3-4	4	4	4-5
PET +5% BNPs	4	4	3-4	4-5

Alt: alteration, St_p, staining on polyester fibers.

polyester fibres with BNPs and significant value with the treatment with IR (PYROTEC³) service technique. The value of alteration increased from 1 to 4 for untreated and treated fabrics respectively. The staining of both polyester and acrylic fibres was increased to be 4-5 for both techniques. These results may be due to the formation of hydrogen bonds within the fibers.

Conclusions

Polyester fibre were modified with different concentrations of BNPs (1, 3 and 5%) using two techniques namely, pad dry cure technique and IR dyeing machine. It was found that the results of treatment using pad dry cure technique showing slightly improvement in the fabric properties than that obtained using IR dyeing machine.

FE-SEM and data of dispersive X-ray spectroscopy (EDX) showed the effect of BNPs on the surface morphology of the treated polyester fabric compared to the untreated one, which gave a clue about the presence of BNPs through polyester macromolecule. The results of the TGA analysis of the treated samples with BNPs compared to untreated one indicated to improvement of the

thermal stability of the treated fibres compared to untreated one, which may be due to the hydrogen bonds between BNPs constituents and the PET OH, COOH function groups.

The values of UPF clarified that the untreated and alkali treated samples have no protection for UVA and UVB radiations, on contrary the samples treated with 5% bentonite have a very good protection for both UVA and UVB radiations (UPF increased to 80%) for pad dry cure technique. The results of the physical and mechanical properties such as tensile strength & elongation% and thickness and moisture regain of the treated polyester fabric with 5% BNPs were significant improved

The results of the colour intensity and fastness to wash of untreated and treated polyester fabric with C.I. Disperse Pink dye as well as C.I. Basic Red 18 dye showed that significant improved due to the treatment.

This study indicated that the treatment of polyester fabric with BNPs have a significant impact in developing and enhancing their properties.

TABLE 8: Washing fastness of untreated and treated polyester fabrics with basic dyes

Samples Description	Pad. Dry curing			IR (PYROTEC3) service		
	Alt	St _p	St _A	Alt	St _p	St _A
Untreated polyester fibres	1	4-5	4	1	4-5	4
Treated with 8%NaOH	2-3	4-5	4	2-3	4-5	4
8%NaOH+5%BNPs	3-4	4-5	4-5	4	4-5	4
PET +1% BNPs	4	4-5	4-5	4	4-5	4
PET+3% BNPs	4	4-5	4-5	4-5	4-5	4-5
PET+5% BNPs	4	4-5	4-5	4	4-5	4-5

Alt: alteration, St_p staining on the polyester fibres, St_A staining on the acrylic fibres.

References

- Varesano A.; Belluati A.; Ramirez D. O.; Carletto R. A.; Vineis C.; Tonetti C.; Songia M. B. and Mazzuchetti G. "A systematic study on the effects of doping agents on polypyrrole coating of fabrics" *J. Appl. Poly. Sci.* **133** (1), (2016).
- Yu J.; Zhou T.; Pang Z. and Wei Q. "Flame retardancy and conductive properties of polyester fabrics coated with polyaniline" *Text. Resear. J.* **86** (11), 1171-1179 (2016).
- Zhang M. Q.; Rong M. Z.; Zhang H. B. and Friedrick K. "Mechanical Properties of Low Nano-Silica Filled High- Density Polyester Composites" *Polym. Eng.Sci.* **43** (2) 490–500 (2003).
- Abou El-Kheir A.; Ezzat M.; Bassiouny F. and El-Gabry L. K. "Development of some functional properties on viscose fabrics using nano kaolin" *Cellulose* **25**, 4805- 4818 (2018).
- El-Gabry L. K.; Sharwy S.; Abou El-Kheir A. and Abd El-Megeide Z. M. "Multifunctional Viscose Fabric Through Loading with Organic and Inorganic Nanostructural Materials" *Egypt. J. Chem.* **60** (1), 555 -569 (2018).
- Adeyemo A. A.; Adeoye I. O.; Bello O. S. "Adsorption of dyes using different types of clay: a review" *Appl. Water Sci* **7**, 543–568 (2017).
- Dumecha, B., & Nalankilli, G. "Anionic dyeability of polyester fabric by chemical surfave modification" *IJMTEs*, **4** (9), 1-14 (2017).
- Judd D. and Wyszecski G. "Colour in Business, Science Industry" John Wiley and Sons, New York, (1975).
- AATCC Technical Manual, Method 36, (1972), 68, 23, (1993).
- Rakesh M. M. and Sanjeev R. S. "Weight reduction of polyester fabric using sodium hydroxide solutions with additives cetyltrimethyl ammonium bromide and [BMIM] Cl" *J. Text. Instit.* **108**(4), 467–471 (2017).
- Pereira A. P.; Silvaa M. H.; Júniora É. P.; Paulaa A. and Tommasinia F. J. "Processing and Characterization of PET Composites Reinforced With Geopolymer Concrete Waste" *Mater. Res.* **20** (2), 411-420 (2017).
- Yoleva A.; Djambazov S.; Michailov G. "Organic Modification of Bulgarian Bentonite By An Easy Low Cost Method" *J. Chem. Technol. Metall.* **51**(3), 275-280 (2016).
- Samperi F.; Puglisi C.; Alicata R. and Montaudo G. "Thermal degradation of poly (ethylene terephthalate) at the processing temperature. *Polym. Degrad. Stab.* **83** (1), 3-10 (2004).
- Mangesh D.; Bhagyashri N. "Superhydrophobic and ultraviolet protective nylon fabrics by modified nano silica coating" *J. TEXT. I.* **108** (3), 1-8 (2017)
- Riva A.; Algaba I.; Pepió M. "Action of a finishing product in the improvement of the ultraviolet protection provided by modal and modal sun fabrics: Modelisation of the effect" *Fibers and Polymers*, **8**, 205-211 (2007)
- El-Sayed H.; Abou El-Kheir A.; El-Gabry L. K. and Haggag K. "Imparting Durable Ultraviolet Protection to Polyamide 6 Fabric" *Fibers and Polymers* **20** (10), 2106-2113 (2019).
- Sankareswari M.; Vidhya R.; Malliga P. and Selvi B. K. "Influence of SiO₂ Concentration on TiO₂ Thin Films as Protective Layer to Chlorophyll in Medicinal Plants against UV Radiation" *Int. J. Thin. Fil. Sci. Tec.* **6**, 9-13 (2017).