



Self Cleaning PET Fabrics Treated with Nano TiO₂ Chemically Cross-Linked with Xanthenes Gum or Cyclodextrin

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To cite this article:

Amr Atef Elsayed, Omaima Gaber Allam, Sahar Hassan Salah Mohamed, Hussain Murad. Self Cleaning PET Fabrics Treated with Nano TiO₂ Chemically Cross-Linked with Xanthenes Gum or Cyclodextrin. *American Journal of Nano Research and Applications*.

Vol. 3, No. 3, 2015, pp. 46-52. doi: 10.11648/j.nano.20150303.13

Abstract: This paper would like to compare the ability of two cellulosic polymers to bind nano titania to polyester fabrics, in order to provide the fabric a self-cleaning property. The fixation of the nano titania on the polyester fabric was explored using Cyclodextrin or Xanthan gum. The photocatalytic activity of TiO₂ nanoparticles deposited on the polyester fabric was followed by the degradation of methylene blue as a model of an organic stain on the polyester fabric surface. The XRD patterns and SEM photographs of polyester fabric coated with nano titania were recorded. The different factors affecting the self-cleaning property as well as the fixation of nano titania was investigated.

Keywords: Nano Titania, Xanthan Gum, Cyclodextrin, Polyester Fabric

1. Introduction

Recent developments of nanotechnology directed to applications in textile areas including fibres are considered. Nanotechnology can provide high durability for fabrics, because nanoparticles have a large surface area-to-volume ratio and high surface energy, thus presenting better affinity for fabrics and leading to an increase in durability of the function. Some of the applications of nanoparticles to textiles are considered [1]. TiO₂ is one of the most popular and promising materials in photo catalytic application due to its strong oxidizing power. TiO₂ is commercially available and easy to prepare in the laboratory [2].

Several recent studies reported the promising potentials of nontoxic and inexpensive TiO₂ nanoparticles (TiO₂ NPs) for imparting multifunctional properties to different textile materials [3-8]. The compatibility of TiO₂ NPs surface with fiber surface chemical functionalities is one of the most important prerequisites for obtaining stable composite system and long-term durability effects. The tailoring of desirable fiber surface from the standpoint of its chemical functionality for improvement of TiO₂ attachment to the fabric surface is very important. NPs binding efficiency is recently gained much scientific interest, generally hydroxyl and particularly

carboxylic groups are the potential sites for binding of TiO₂ NPs [9, 10]. The alginate was applied as a fiber surface modifier for improvement of binding efficiency between colloidal TiO₂ NPs and polyester fabric. The results imply good laundering durability of the fabric [11]. Cationization is a novel treatment on cotton to produce fabric with new characteristics. The nano titanin particles were stabilized on the cotton surface using butane tetra carboxylic acid [12].

Xanthan gum is a high molecular weight polysaccharide. It is used as a rheology control agent in aqueous systems and as a stabilizer for emulsions and suspensions. In textile printing, common thickeners such as guar gum, xanthan gum and sodium alginate are used to control the rheology of the dye pastes; Alginates, guar gum, and xanthan gum are often the thickeners of choice because they are pure non-reactive hydrodynamic thickeners unlike starches and other thickeners. In addition to fashion and comfort demands, the garments today must simultaneously provide self-cleaning properties, antimicrobial and UV protection. Utilizing these advantages is the ideal material for a wide range of uses, including filter materials in industrial applications [13]. The use of cyclodextrins and their derivatives (CDs) in the textile domain is a challenge that rose in the early 80's. The grafting of CDs onto cellulose fibers by using epichlorohydrin as crosslinking agent was reported [14], also it was covalently

linked to the fabrics by the intermediate of the poly carboxylic acid (PCA) that esterified (or amidified) at the same time with the OH (or NH₂) groups of fibers and those of CDs [15]. Recently, several studies have reported the presence of nanocrystalline TiO₂ layers on textiles that were prepared from sol–gel at relatively low temperatures [16]. Keeping in mind, the polyester fabrics is one of the most consumed fabrics, so the present paper consists to report the results obtained in a study that aimed to apply the above mentioned chemical path onto polyester made fabrics. We describe the study of the parameters involved in this particular textile processing. These parameters include the curing conditions (temperature, time) and also the nature of the reactants. Citric acid (CTA) used as crosslinking agents; sodium dihydrogen hypophosphite (NaH₂PO₂) was used as catalyst. The methylene blue was used as a model for stain on the fabric surface.

2. Materials and Methods

2.1. Materials

Poly (ethyleneterephthalate) (PET) nonwoven fabrics (surface weight = 55 g/m²) were supplied by Abou El-Ola for Spinning and Weaving, 10th of Ramadan, Egypt. B-Cyclodextrin (CD), Acros organic, USA. Titanium tetraisopropoxide {Ti (OCH(CH₃)₂)₄} 97% from Sigma-Aldrich. Citric acid hydrate (CTA), sodium, hypophosphite (NaH₂PO₂) and other chemicals were of a laboratory grade.

2.2. Synthesis of Xanthan Gum

Xanthan gum obtained from milk permeates by *Xanthomonas campestris* LIS-4 isolated from infected cabbage seeds [17]. The obtained xanthan gum has the following characteristics: Production yield of xanthan gum from milk permeate is 43.9 g/l, pyruvic acid content is 2.3 mg/100g and the viscosity of the aqueous solutions of xanthan gum is 4.8 CP.

2.3. Preparation of Nano Titanium

Single-phase anatase sol was prepared according to previous methods [18, 19]. Ten milliliter titanium tetraisopropoxide was added dropwise to 100ml acidic aqueous solution containing 1ml nitric acid (70%) and 10 ml acetic acid (97%) under vigorous stirring. The mixtures were heated at 60°C and maintained at that temperature while stirring for 16h.

2.4. Finishing of Polyester (PET) Fabrics

The polyester fabrics were impregnated in an aqueous bath containing citric acid (5% w/w), the catalyst (NaH₂PO₂) (0.5% w/w) and the CD or gum) (5% w/w). Polyester fabrics were padded at wet pick up 100%. The fabrics were then dried at 90 °C, and cured at 180 °C for 30 min in case of CD [20], while the curing conditions were studied in case of gum. Samples were washed with water in soxhlet in order to

remove unreacted products. The percentage grafting (% wt) of the samples were calculated from the following equation.

$$\% \text{ wt} = \frac{wt_2 - wt_1}{wt_1} \times 100$$

Where wt₂ and wt₁ are the weight of sample after and before treatment, respectively.

2.5. Treatment of Grafted Polyester Fabrics with TiO₂ Sol

Virgin polyester as well as pretreated polyester fabrics were subjected to a dip-pad–dry-cure process. The substrates were dipped in TiO₂ sol (containing 1.6% Ti) for 1 min, and padded at wet pick up 100%. The substrates were oven dried, and finally cured at 120°C for different periods of time. The fabrics were then washed in an ultrasonic bath to remove the unbound nano titania from the fabric surface.

2.6. Characterization of the Prepared Nano Titania

2.6.1. Transmission Electron Microscopy (TEM) Characterization

The surface morphology of the prepared nano titania was investigated using transmission electron microscope JEM - 1230 JEOL Co. Japan, maximum magnification power is 600kX.

2.6.2. X-ray Diffraction (XRD) Analysis

The XRD patterns of the prepared nano titania were recorded by using PAN Analytical X Pert PRO X-ray diffractometer and Cu Ka radiation ($\lambda=1.5406 \text{ \AA}$) as X-ray source. The scanning rate were carried out at 8 °/min in 2 θ range of 20 °-70 ° (θ being the angle of Braggs diffraction), using a wavelength of ($\lambda=1.5406 \text{ \AA}$). The crystal diameter has been determined according to Deby-Scherrer formula:

$$D=K \lambda / (\beta \cos \theta)$$

Where K is the Scherrer constant (0.9), the radiation wavelength $\lambda=0.15406 \text{ nm}$, β is the peak width of half maximum; and θ is the Braggs diffraction angle and D is the particle size.

2.7. Characterization of the Modified Polyester Fabrics

Virgin polyester as well as modified polyester fabrics were mounted on aluminum stubs, and sputter coated with gold in a 150 Å sputter (coated Edwards), and examined by Jeol (JXA-840A) Electron Probe Microanalysis (Japan), magnification range 35 – 10,000, accelerating voltage 19kV.

The amount of nano titania absorbed by the PET fabrics was determined by boiling 1 g fabrics in 100 ml concentrated sulfuric acid for 5 hours, the eluted titanium was estimated using atomic absorption spectrometer (VARIAN AA220).

2.8. Photocatalytic Activity of the Nano Titania Estimation

The photocatalytic activity of the nano titanium oxide coated PET fabric was investigated by treating the fabric with 2 ml methylene blue (MB) solution (1%) followed by drying

at ambient temperature. One half of each methylene blue stain on the fabric was exposed to 400W high pressure mercury lamp for different periods of time. The other half was enclosed with a black paper to avoid its irradiation from the lamp. K/S (absorption to scattering coefficient where, K is the absorption; S, the scattering coefficient.) was assessed to each stain on the fabric (the enclosed and the unenclosed parts) after the exposure to the UV irradiation, using Hunter Lab Universal Software MiniScan TM XE. The efficiency of nano titanium to remove the methylene blue (MB) stain was calculated according to the following equation.

$$\% \text{ Removal of (MB)} = \frac{(K/S) \text{ before irradiation} - (K/S) \text{ after irradiation}}{(K/S) \text{ before irradiation}} \times 100$$

2.9. Washing Durability Test

Adopting the AATCC test method 61 – 2003, the washing fastness of the treated fabrics was assessed. Washing was performed using a home laundering machine. The fabrics were washed in 0.15% aqueous solution of sodium lauryl sulphate as detergent and in the presence of 50 steel balls for 45 min. The fabrics were then rinsed with water, and dried at room temperature.

3. Results and Discussions

3.1. Characterization of Nano Titania

XRD data (Fig. 1) show distinct diffraction peaks at 2θ of 25.28, 27.41, 46.9, and 54.6. The sharp peaks confirmed the crystallinity of the prepared nano titanium dioxide. The data also indicates that the majority of the prepared nano titanium dioxide is in the anatase form, and according to Scherrer's equation the average nano titanium dioxide crystal size is 20-25 nm. Figure 2 shows the TEM photograph of the prepared nano titania, and the average particle size calculation showed that the prepared titania is in the size of 25 nm, which is in consistence with the value obtained from the XRD analysis.

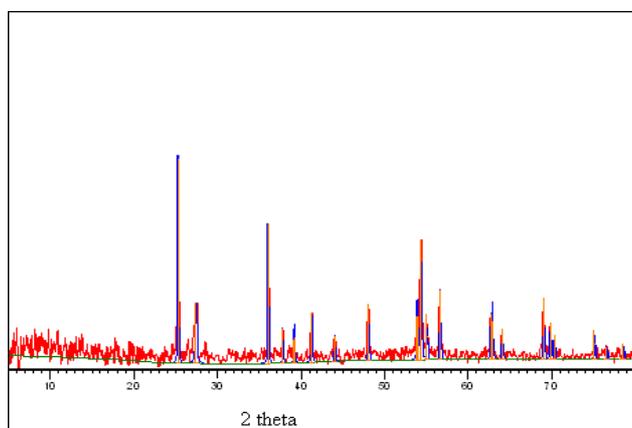


Figure 1. XRD pattern of the prepared nano titania.

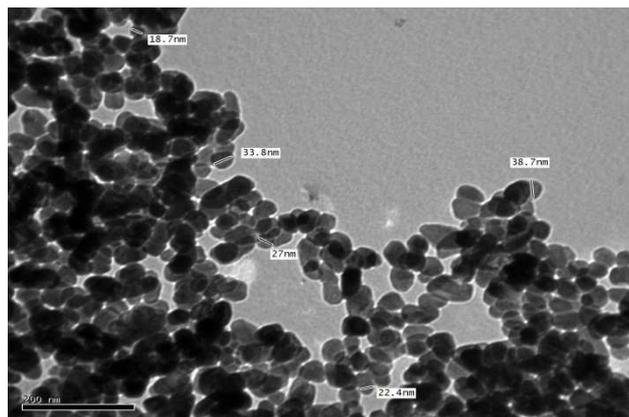


Figure 2. SEM photograph of the prepared nano titania.

3.2. Effect of Curing Conditions

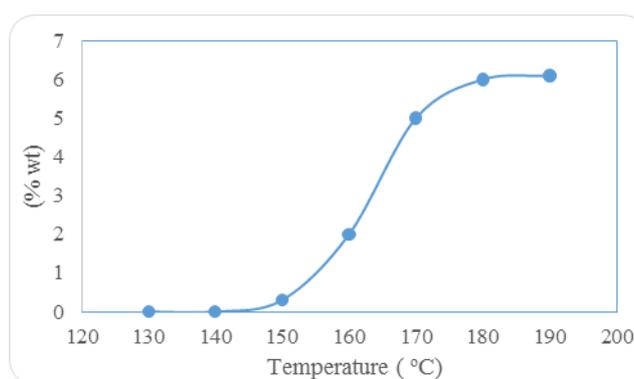


Figure 3. Effect of curing temperature on the % wt of gum added to PET fabrics. (Curing time 10 min).

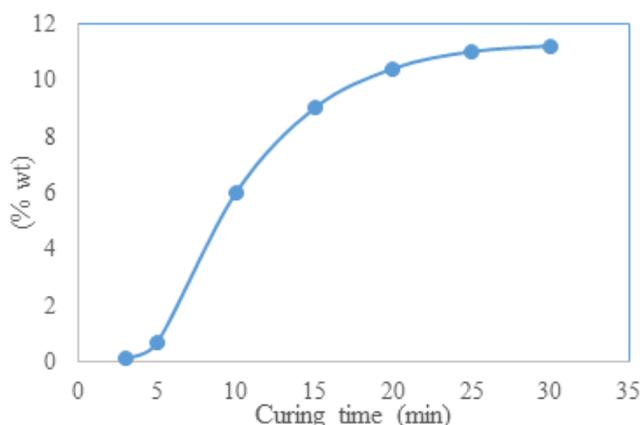


Figure 4. Effect of curing time on the % wt of gum added to PET fabrics. (Curing temperature 180 °C).

The curing conditions between gum, CTA and PET fabrics were studied at variable temperatures and time. It is observed that, for ten minutes curing, as the curing temperature increase, the (%wt) increases, to reach its maximum at 180 °C (Figure 3) after which no appreciable increase in the (%wt) was observed. Figure 4 shows that, the maximum (%) grafting (11%wt) could be attained after 25 min curing at 180 °C. It is worth mentioning that, the %wt of CTA and CD added to PET fabrics was 8.1% at curing conditions of 180

°C for 30 min. The PET fabrics treated by a mixture of CTA and CD or gum resulted in a weight increase of the samples. We could confirm that a permanent grafting occurred as this value did not vary upon the successive washings. The direct reaction between CD and PET fibers has been proved to be

impossible. It is concluded that the reaction occurred through the polyesterification between CTA and CD and the formed copolymer is physically adhered or was even entangled into the fibrous network so that grafting was resistant to washings and was thus permanent [15].

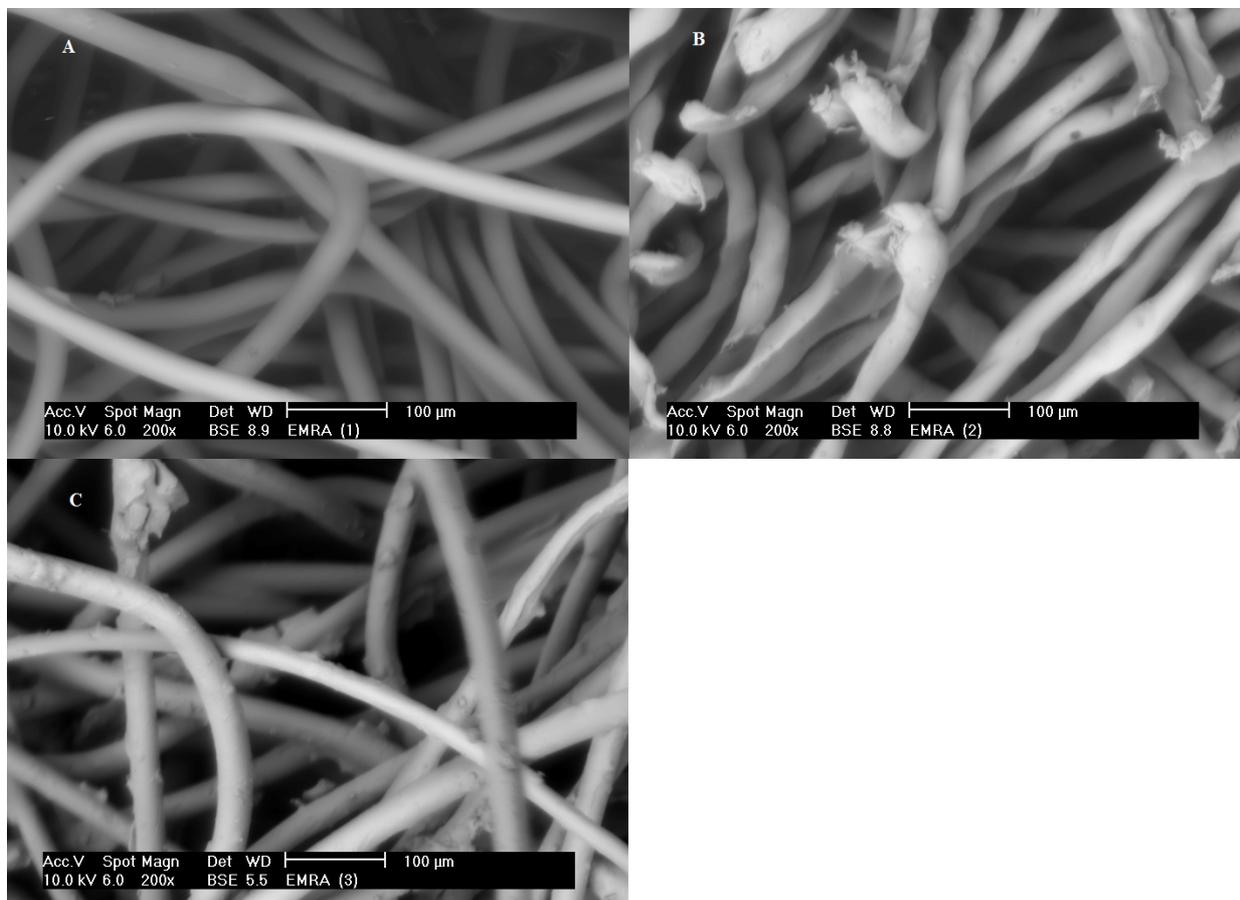


Figure 5. SEM images of PET fibers (A) untreated PET, (B) PET treated with CD & CTA and (C) PET treated with gum & CTA).

3.3. Characterization of PET Fabrics Treated with CD or Gum

The presence of CD or gum on the PET fibers was confirmed by SEM analysis Fig 5. The SEM photographs also show the formation of homogeneous layer of CD or gum on the BET fibers surface, as well as the presence of small aggregates in case of gum.

3.4. Influence of TiO_2 Concentration and Curing Time on the % MB Removal

The self cleaning property of PET fabrics treated with CTA and CD and after treated with nano titania was studied. Values of % MB dye removal were presented against the nano titania concentration and curing time in Table 1.

Data of Table 1 shows the inability of virgin PET fabric to remove the MB dye. While in both cases of unmodified as well as modified PET fabrics with CTA or CD and after treated with nano titania, it was found that, as the concentration of titanium increased from 0.3% to 0.9%, the % dye removal increased; it is also observed that, higher

titanium concentration leads to a decrease in % dye removal. This observation could be explained as a result of the coagulation of the nano particles at higher concentrations on the fabric surface, and therefore losing the preferential of the nano size. The data also clarify the % dye removal form PET fabric treated with CTA-CD and after treated with nano titania is much higher than that of PET fabrics only treated with nano titania, which could be referred to the ability of the unreacted carboxyl groups of CTA to attract larger amount of nano titania to the fabric surface [13].

Maximum dye removal of (91.7%) was attained when nano titania cured with the modified PET fabrics for 25 min, and that seems to be the required time for the formation of a stable coordination between Ti^{4+} and the created functionalities on PET fabric.

The aforementioned study was repeated for the PET fabrics treated with CTA and gum (Table 2). It should be underlined that PET fabrics treated with CTA-gym and after treated with nano titania shows higher ability to remove MB dye in comparison with those of CD-CTA. Additionally curing for 15 min is quite enough to reach the maximum %

of dye release.

Table 1. Effect of nano TiO₂ and curing time on the% of MB dye removal from PET fabrics pretreated with CTA-CD.

Type of modification of PET fabrics	Conc. Of nano titania (%)	Curing time of nano titania and fabrics (min)	% MB dye removal after UV irradiation for		
			30 min	60 min	120 min
MB dye solution only			2.23	7.38	9.34
Unmodified PET and without nano titania			3.22	8.63	10.29
Unmodified PET	0.3	25	25.46	30.95	56.84
	0.6		28.59	39.19	61.36
	0.9		31.73	59.19	71.91
	1.2		29.38	44.68	62.93
	1.5		25.85	42.72	62.14
	0.3		47.03	63.51	72.14
	0.6	15	52.53	65.08	77.63
	0.9		56.06	67.04	87.56
	1.2		44.29	64.29	83.13
	1.5		44.68	63.51	77.63
	0.3		52.13	67.04	72.93
	0.6		54.09	75.67	79.99
PET modified with CTA - CD	0.9	20	59.19	79.99	90.23
	1.2		56.84	74.89	78.42
	1.5		53.31	72.93	75.67
	0.3		60.37	69.79	79.99
	0.6		61.16	73.32	81.56
	0.9		25	63.12	83.52
	1.2	50.56		80.77	86.66
	1.5	41.54		77.24	81.56

Table 2. Effect of nano TiO₂ and curing time on the% of MB dye removal from PET fabrics pretreated with CTA-gum.

Type of modification of PET fabrics	Conc. Of nano titania (%)	Curing time of nano titania and fabrics (min)	% MB dye removal after UV irradiation for		
			30 min	60 min	120 min
PET modified with CTA - gum	0.3	15	56.45	64.29	87.44
	0.6		61.16	71.75	89.40
	0.9		61.94	76.06	95.51
	1.2		61.16	73.32	92.5
	1.5		60.37	72.53	91.36
	0.3		20	63.90	74.10
	0.6	65.47		84.30	90.19
	0.9	67.04		85.48	96.07
	1.2	65.08		84.69	94.50
	1.5	64.29		83.52	94.50
	0.3	25		61.94	73.71
	0.6		67.83	79.99	92.15
	0.9		69.79	87.83	96.50
	1.2		67.04	86.66	92.54
	1.5		65.47	84.69	92.15

The superior ability of PET fabrics treated with gum and after treated with nano TiO₂ over CD even at lower curing time, could be explained on the bases of the ability of gum to absorb higher amount of nano titania. In order to elucidate that assumption, the titanium existed on PET fabric modified either with gum or CD was leached out; and the obtained amount of titanium was presented in Table 3. It is clear that CTA-gum is able to bind higher amount of titanium than CTA-CD, which could be referred to the availability of unreacted carboxyl groups of CTA [21] in addition to readily existed carboxyl groups in gum macromolecules (pyruvic

acid content 2.3 mg/100g), which is known for its affinity toward nano titania. Similar results was observed in previous study about the affinity of PET fabrics treated with alginates to bond to nano titania [11]. Namely, when diameter of nanocrystalline anatase TiO₂ particles becomes around 20 nm or smaller, the surface of Ti atoms adjust their coordination environment from octahedral to more reactive pentacoordinated (square pyramidal) [22,23]. These undercoordinated defect sites are likely the sources of enhanced binding between carboxyl functionalities and Ti atoms. Bearing in mind, CTA-CD only counts on the

unreacted carboxyl groups of CTA for the bonding to TiO₂.

Table 3. Concentration of TiO₂ extracted from PET fabrics.

Modification	Initial conc. of TiO ₂ sol (%)	Conc. of TiO ₂ extracted (g/100g fabric)
PET unmodified (25 min)		0.01
PET+ CTA+CD (15 min)		0.05
PET+ CTA+CD (20 min)		0.09
PET+ CTA+CD (25 min)		0.10
PET+ CTA+CD (30 min)	1.6	0.11
PET+ CTA+ gum (15 min)		0.33
PET+ CTA+ gum (20 min)		0.35
PET+ CTA+ gum (25 min)		0.35
PET+ CTA+ gum (30 min)		0.37

3.5. Washing Durability Evaluation

In order to elucidate the durability of photocatalytic activity of nano TiO₂ deposited on the PET fabrics treated with CTA-gum or CTA-CD, the photo degradation process under the UV irradiation was repeated after different washing cycles as presented in Figure 3. It can be noticed that the xanthan gum is able to maintain the bonded nano titania without noticeable decrease in the self cleaning property of the PET fabrics, even after 10 washing cycles. It is worth mentioning that the PET fabrics treated with CTA-CD and after treated with nano titania can sustain the self cleaning property after the repeated washing cycles but in a manner less than that of gum. On the contrary, unmodified PET fabric and treated with nano titania loses all of its self cleaning ability after 5 washing times. The overall results confirm the formation of strong and stable bond between carboxyl groups of gum with Ti⁴⁺.

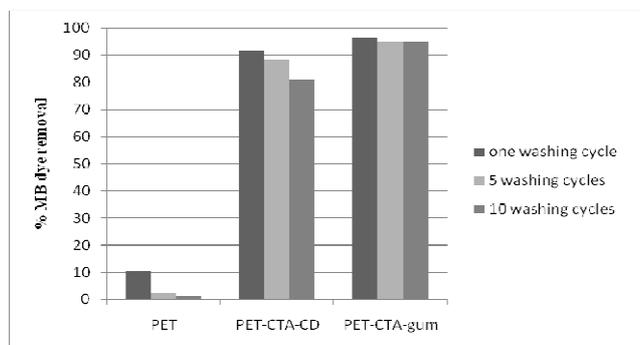


Figure 6. Washing durability test (TiO₂ conc. 0.9% cured at 20min, irradiation time 120 min).

4. Conclusions

The purpose of this study was to investigate the self-cleaning properties of modified PET fabrics with CD or gum and after treated with nano TiO₂. Regarding to the curing conditions, the % wt of CTA and gum added to PET fabrics maximum % grafting (11%wt) could be attained after 25 min curing at 180 °C, while the % wt of CTA and CD added to PET fabrics was 8.1% at curing conditions of 180 °C for 30 min. The XRD data and TEM photograph of the prepared nano titania indicate that the majority of the prepared nano

titanium dioxide was in the anatase form with crystal size is 20-25 nm. Examination of the self-cleaning property of the modified PET fabrics with CTA and CD or gum and after treated with nano titania showed maximum dye removal of using CD was attained when nano titania cured with the modified PET fabrics for 25 min, while curing for 15 min is quite enough to reach the maximum % of dye release in case of gum. The PET fabrics treated with CTA-gym and after treated with nano titania shows higher ability to remove MB dye (95.5%) in comparison with those of CD-CTA (91.7%). The xanthan gum is able to maintain the bonded nano titania without noticeable decrease in the self cleaning property of the PET fabrics, even after 10 washing cycles.

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