

# ALKALINE TREATMENT OF POLYETHYLENE GLYCOL MODIFIED POLY (ETHYLENE TEREPHTHALATE) FABRICS

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*The present work aims at investigating the kinetic behaviour of the alkaline treatment of polyethyleneglycol modified poly (ethylene terephthalate) (PEG-M-PET) and regular poly (ethylene terephthalate) (R-PET) (for comparison) fabrics. Moreover, the effect of the introduction of polyethylene glycol (PEG) moieties in macromolecule on the hydrolysis behaviour of PET samples and some properties of treated fabrics were studied. Effect of reaction parameters such as treatment time, concentration of sodium hydroxide, and temperature on the extent of hydrolysis was examined. The mechanism of hydrolytic degradation of PEG-M-PET and R-PET fabrics, as determined by weight loss, had also been ascertained. At equivalent parameters of alkali concentration, time and temperature of treatment the weight loss levels obtained in the case of PEG-M-PET were higher than when the alkaline treatment was carried out in the presence of R-PET fabrics. Weight loss increases linearly with treatment time and nonlinearly with alkali concentration and reaction temperature. The extent of nonlinearity is lower for PEG-M-PET fabrics as compared to that of R-PET samples. Control and treated PEG-M-PET and R-PET fabrics were evaluated by scanning electron microscopy (SEM), mechanical and thermal (DSC, TGA) properties. It was found that alkaline treated PEG-M-PET fabrics are characterized with deeper cavities on the surface which ran parallel to the fibre axis, higher decrease in breaking strength and flexural rigidity, less surface roughness, lower  $T_g$  values and higher decomposition temperature ( $T_d$ ) as compared to the R-PET weight reduced samples.*

**Key words:** Alkaline hydrolysis, Fabrics, Poly (ethylene terephthalate) (PET), Mechanical and thermal properties, Surface morphology.

## INTRODUCTION

Alkaline treatment of polyethylene terephthalate (PET) is a well – known finishing process for fabrics<sup>1</sup>. It improves handle<sup>2</sup>, wettability<sup>3</sup>, resistance to abrasion damage<sup>4</sup>, soil resistance properties<sup>5</sup>, and drape of fabrics<sup>6</sup>. There have also been some reports on kinetics of alkaline hydrolysis, as follows. C.G.G. Namboori<sup>7</sup> found that treating polyester with different concentrations of aqueous NaOH solutions would cause a significant weight loss in the fibre. Moreover, at a constant temperature, the weight loss was proportional to hydrolysis time. This observation was consistent with the one given by E.Waters<sup>8</sup>. N.Kallay et al.<sup>10</sup> indicated that at a constant concentration of aqueous NaOH solution, the weight loss was

proportional to a specific area of the fibre. It was stated that the radius of the fibre decreased when the concentration of aqueous NaOH solution was increased<sup>11</sup>.

Though there is abundant literature available on weight reduction of regular polyester, less information is available about alkaline treatment of fibres produced from copolyesters. Recently, fibres were produced from polyethylene glycol modified polyethylene terephthalate (PEG-M-PET)<sup>12</sup>. The use of these fibres is increasing day by day and likely to achieve a significant position in the next few years due to their technological advantages as: a higher colour yield, shorter dyeing cycle, reduction in dyeing costs, environmental protection, and reduction of the oligomer problem during dyeing<sup>13</sup>.

Alkaline hydrolysis may be used for further modification of PEG-M-PET fibres to produce desirable properties in the consumer product. At present time there is almost no reference material on the extensive study on alkaline treatment of this type of fibres. It appears, therefore, of great scientific and practical interest to establish the behaviour of these fibres towards such alkaline treatment. In light of the above mentioned the present work aims at investigating the kinetic behaviour of the alkaline treatment of PEG-M-PET and regular polyethylene terephthalate (R-PET) (for comparison) fabrics. Moreover, the effect of the introduction of PEG moieties in macromolecule on the hydrolysis behaviour of PET samples and some properties of treated fabrics were studied.

## EXPERIMENTAL

### Materials

PEG-M-PET fabric was a plain weave (100/65)/inch in both weft and warp, weighing 152g/m<sup>2</sup>; R-PET fabric was a plain weave (97/64) / inch in both weft and warp, weighing 143g/m<sup>2</sup>. The fabrics used throughout this study were kindly supplied by Misr Polyester Co., Kafr El-Dwar, Egypt. The fabrics were scoured at 80°C for 45 min. with solution containing 2g/l nonionic detergent, washed with cooled water, squeezed and finally air dried.

Solutions of sodium hydroxide (A R grade) were made up in distilled water and stored in plastic bottles; concentrations were determined by titration.

### Production of PEG-M-PET fibres

Synthesis of polyethylene glycol modified polyethylene terephthalate (PEG-M-PET), and the production of oriented staple modified fibres were carried out according to the method describe in<sup>12</sup>. These staple fibres were used for manufacturing PEG-M-PET fabrics.

### Alkaline hydrolysis

The alkaline treatment of polyester fabrics was carried out using a high temperature, high pressure laboratory dyeing machine. Required amounts of alkali solutions were placed in stainless-steel bowls, fabric samples were immersed in the solutions, and the sealed bowls were rotated in a closed bath containing ethylene glycol at the desired temperature. The liquor- to- goods ratio (L: G) was 50:1. The bath temperature increased at rate of 2°C/min. After the predetermined durations, the samples were removed from the bath, rinsed repeatedly with distilled water, neutralized with a solution of 1% hydrochloric acid and rinsed. The samples were then dried at 100°C, cooled in a dessicator, and weighed.

### Characterization of products

The weight loss (WL) is expressed as relative WL according to the equation:

$$WL (\%) = [W_1 - W_2 / W_1] \times 100$$

Where  $W_1$  and  $W_2$  are the weights of the samples before and after alkaline treatments, respectively

The breaking strength and percentage elongation at break was determined using Instron universal testing machine with a 5x20cm guge length at ~ 30cm/min. constant rate of elongation at 65% relative humidity and 20±2°C. Results are the average of 5 tests.

Flexural rigidity, a measure of bending modulus, and hence, stiffness of the fabric, as it is experienced between fingers, was measured using a single Contilever test<sup>14</sup>. The overall effect of warp and weft is reported as the flexural rigidity value of the fabric.

Surface Roughness was measured using MEASURING INSTRUMENT model S E 1700a, Kosaka Laboratory Ltd., JAPAN, according to JIS 94.

### Thermal analysis

- The differential scanning calorimetry (DSC) for parent and alkaline treated polyester fabrics was carried out using DSC Perkin-elmer-7, USA thermal analyzer. The rate of heating was adjusted at 10°C/min. Thermograms were recorded from 25°C to 400°C under nitrogen atmosphere.
- The thermogravimetric analysis (TGA) for parent and alkaline treated polyester fabrics was carried out using TGA-50 Shimadzu thermal analyzer. The rate of heating was adjusted at 10°C/min. Thermograms were recorded from 25°C to 600°C under nitrogen atmosphere.

### Surface morphology

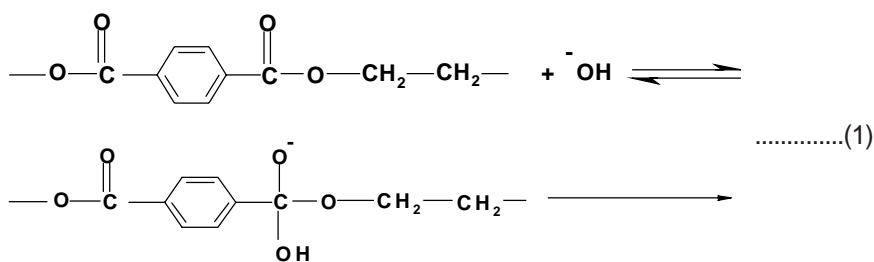
A JEOL-Model JSM-T20 Scanning Electron Microscope (SEM) operating at 19KV was used to obtain photomicrographs of fibres surfaces. Coating was done with gold in vacuum evaporator.

## RESULTS AND DISCUSSION

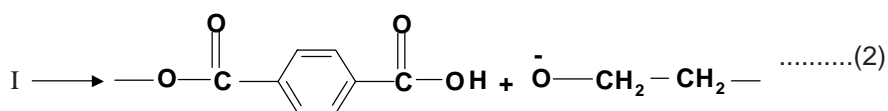
In this work, we report on the results of the alkaline treatment of PEG-M-PET fabrics by using aqueous NaOH solutions. Moreover, morphological observation, mechanical and thermal properties of parent and treated fabrics were evaluated.

It was observed that alkaline treatment of both PEG-M-PET and R-PET leads to a loss in weight of the fabrics. Hydrolysis was found to proceed, but with a low weight loss (R-PET = 1.8% , PEG-M-PET = 2.2% after 10 min. at 100°C) when fabrics were treated with aqueous NaOH solution having low concentration (0.25N). The weight loss increased with increasing NaOH concentration and treatment temperature, irrespective of the type of treated fabrics. However, a high level of weight reduction occurs in the case of PEG-M-PET fabric. This tendency may be the result of the introduction of polyethylene glycol (PEG) moities in PET macromolecule and preferred hydrolysis that the OH – group had towards PEG-M-PET rather than R-PET.

According to the present views of the hydrolysis of polyethylene terephthalate (PET) by alkali metal hydroxides<sup>1,2,15,16</sup> the hydroxide ions attack the electron - deficient carbons, resulting in the first step in the production of hydroxyl and carboxyl groups at the fibre surface. A low molecular segment of the chains is removed, resulting in weight loss and decreased fibre diameter.



In the second step, further reaction between OH<sup>-</sup> ions and -C- breaks the macromolecular chain and produces -COOH and -O-CH<sub>2</sub>-CH<sub>2</sub>-, the final product is -COONa in alkaline solution



Since the main difference between the two polymers PEG-M-PET and R-PET is existence of PEG moiety, one can conclude that the dissolution of PEG-M-PET resembles that of R-PET.

Given below are the different factors that affect the weight loss of treated samples.

#### Treatment Time

Fig. 1 illustrates the percentage weight loss of PEG-M-PET and R-PET fabrics respectively on treatment with 0.25, 0.5, 2.0 mol/l sodium hydroxide solution at 100°C for various time periods. According to this figure, alkaline hydrolysis proceeds linearly with time at constant temperature and depends on the initial concentration of the alkali. The slope of the straight lines obtained between weight loss and time increases with increasing concentration of the alkali. This was valid irrespective of the type of treated PET fabric. A linear dependence of weight loss on treatment time has also been observed by other investigators for R-PET round fibre<sup>7,9</sup>. The relationship between weight loss,

WL, and treatment time, t, can be expressed as:

$$\text{WL} = b.t \quad \text{.....(3)}$$

Where, b, is a constant dependent on temperature and alkali concentration. However, a high level of weight reduction was observed in the case of PET-M-PET fabric. The slopes of these linear curves (Table - 1) indicate a high rate of weight reduction in the case of PEG-M-PET fabric, than when the reaction was carried out in the presence of R-PET fabric. This may be due to the higher sensitivity of PEG-M-PET to alkaline attack, since it contains PEG moiety in the polymer chain itself. The difference in the internal structure of PEG-M-PET in comparison with the R-PET would also be responsible for this behaviour. Similar behaviour was observed in the case of alkaline hydrolysis of cationic dyeable polyester yarns<sup>17</sup>.

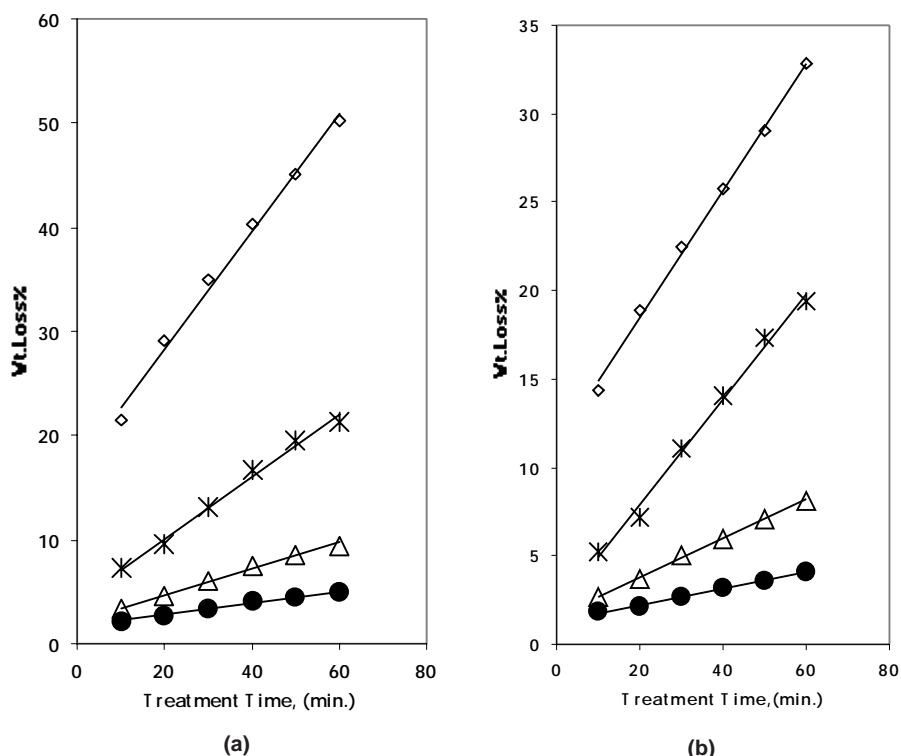


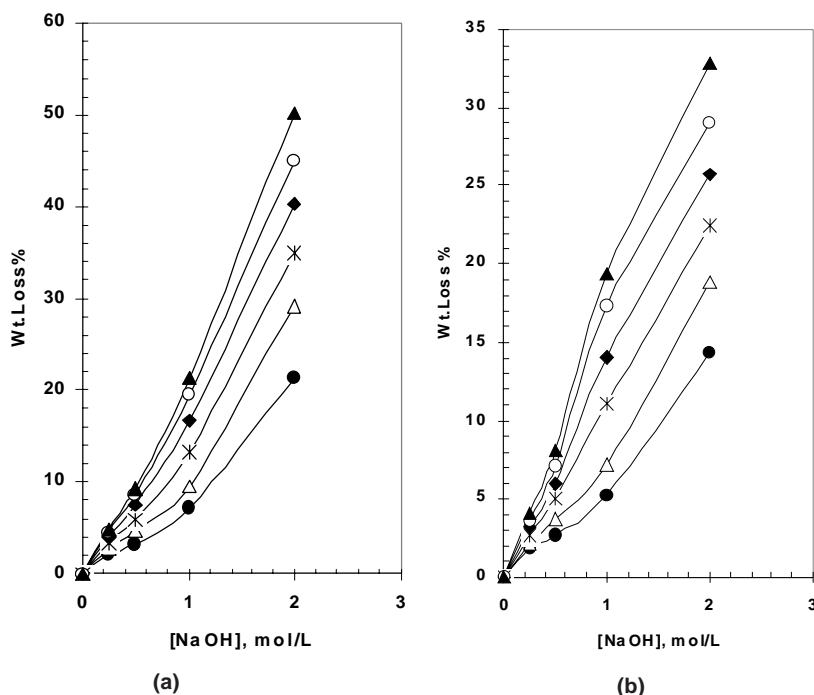
Fig. 1: Dependence of weight loss for PEG-M-PET (a) and R-PET (b) fabrics on treatment time

Reaction conditions:

[NaOH], mol/l : (●), 0.25; (△), 0.5; (\*), 1.0; (◇), 2.0;  
 Treatment temperature, 100°C;  
 Material: Liquor ratio (M:L), 1:50.

**Table - 1: Slope of Linear Relations Between Loss in Weight (%) and Time of Treatment (min.) and Time of Treatment (min.)**

Fabric	Slope at [NaOH], mol/l:			
	0.25	0.5	1.0	2.0
PEG-M-PET	0.054	0.122	0.282	0.576
R-PET	0.046	0.108	0.277	0.368



**Fig. 2: Dependence of weight loss for PEG-M-PET (a) and R-PET (b) fabrics on concentration of sodium hydroxide solutions.**

**Reaction Conditions:**

Treatment time (min.) : (●) ,10; (Δ) ,20; (✱) , 30;(●) ,40;(?) ,50;(▲) ,60;  
 Treatment temperature,100°C;  
 M: L, 1:50.

**NaOH Concentration**

Typical curves showing the dependence of weight loss for PEG-M-PET and R-PET fabrics on concentration of sodium hydroxide solution are given in Fig. 2. It can be seen that at constant treatment time and temperature the weight loss increases with increasing the concentration of NaOH solution, irrespective of the type of treated fabric. However, a high level of weight reduction occurs in the case of PEG-M-PET fabric, than in the case R-PET, especially at high concentration of NaOH solution. For instance, a weight loss of 32.6% was obtained in the case of R-PET fabric at NaOH concentration of 2.0

mol/l. This contrasts with a weight loss of 50.2% after alkaline treatment of PEG-M-PET under the same conditions. The findings listed on Fig. 2 also revealed that alkaline hydrolysis of PEG-M-PET and R-PET fabrics proceeds nonlinearly with the concentration of alkali. The

extent of nonlinearity increases with increasing time of treatment. There are two possible causes. The first is because the concentration of OH<sup>-</sup> ion decreases with the degree of reaction. The second cause is that when a large fraction of the sample has been hydrolyzed, the surface area decreases accordingly, thus decreasing the reaction rate. A second order relationship,

$$WL = a C + d C^2 \dots\dots\dots (4)$$

where, (a) and (d) are constants dependent upon treatment time and temperature, satisfactorily describes the experimental data. Validity of Eq.4 is verified in Fig. 3, where WL/C values have been plotted against C. All plots are straight lines as required by Eq. 4. Values of constants (a) and (d), obtained from the intercept and slope, respectively, of the straight lines, are summarized in Table - 2. It is clear from the table II that values of constant (d), which is a measure of the extent of nonlinearity, is lower for PEG-M-PET fabrics as compared to that of R-PET fabrics.

**Treatment Temperature**

Dependence of weight loss for PEG-M-PET and R-PET fabrics on treatment temperature is shown in Fig. 4. It can be seen from the figure that the percent weight loss increases with the temperature, irrespective of the type of fabric used. When the temperature is well beyond the T<sub>g</sub> of PET (~80°C for crystallized PET<sup>9</sup>), the slope is

**Table - 2: Values of Constants (a) and (d) for Polyester Fabrics at 100°C**

Treatment Time (min.)	PEG-M-PET fabrics		R-PET fabrics	
	(a), mol <sup>-1</sup>	(d), mol <sup>-2</sup>	(a), mol <sup>-1</sup>	(d), mol <sup>-2</sup>
10	8.30	1.10	4.40	1.30
40	13.8	2.60	11.5	2.70
50	16.0	3.10	12.9	4.20
60	16.9	4.10	14.8	4.30

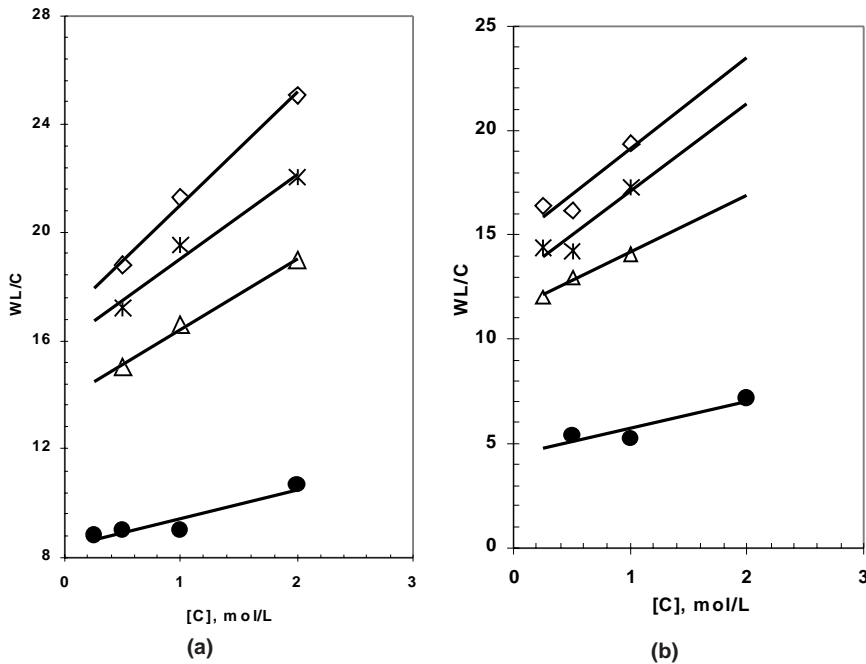


Fig. 3: Test of validity of second order relationship for the description of data indicating the dependence of weight loss for PEG-M-PET (a) and R-PET (b) fabrics on concentration of NaOH solutions for different intervals of treatment time (min.): (•),10;(Δ),40;(✱),50;(△),60.

steeper. The observed nonlinear dependence of weight loss on the concentration of NaOH and temperature is due to the enhanced dissolution of polymer chains with increasing severity of the treatment<sup>4</sup>. The steep rise in weight loss at treatment temperature

above the  $T_g$  is because above this temperature the molecular chains have more freedom to move and thus more easily interact with NaOH. The results obtained for PEG-M-PET and R-PET show the same trend, but the values are much smaller in the case of R-PET.

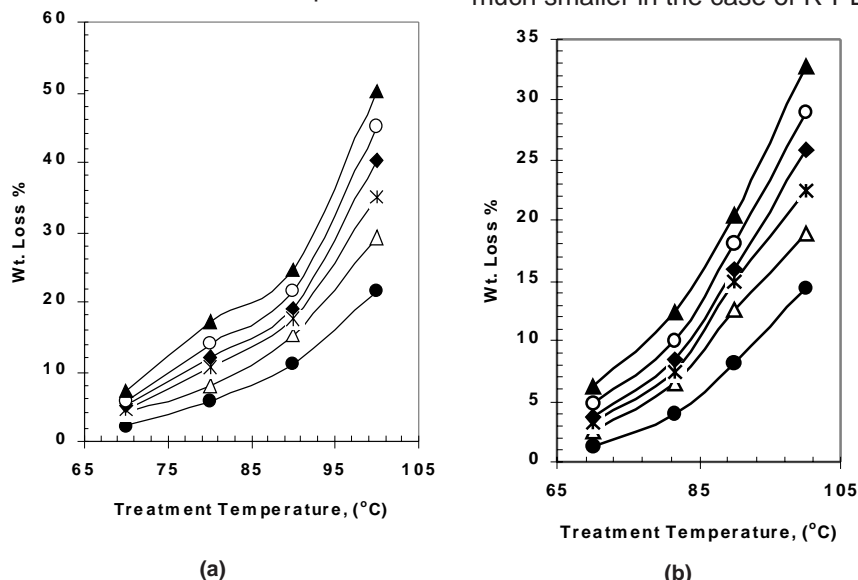


Figure 4: Dependence of weight loss for PEG-M-PET (a) and R-PET (b) fabrics on treatment temperature.

**Reaction Conditions:**

Treatment time (min.):(•), 10; (Δ),20;(✱),30; (◐),40;(o) ,50;(▲), 60; [NaOH], 2mol/l. ;

M: L, 1:50.

Based on the abovementioned discussion, it can be stated that in the process of alkaline hydrolysis polyethyleneglycol modified polyethylene terephthalate fabrics show higher sensitivity to alkaline attack. Under any alkaline treatment conditions, the values of weight loss for PEG-M-PET are always higher than those for regular PET; the point which paves the way for shortening the treatment time to achieve weight losses similar to those for regular PET.

**Fabric Characterization**

**Morphological Observation**

Fig. 5 presents a typical scanning electron microscopy (SEM) for untreated and treated with NaOH PEG-M-PET and R-PET fabrics. Before treatment the fibre was thick, round, and smooth. As the hydrolysis started a few pits appeared on the surfaces, and the latter become coarser. These pits probably serve as nucleation centers for crack formation. The pits increase in number and depth with increasing weight loss, irrespective of the type of treated fabric. But the surface ditches and pits differ for the two types of samples. It was observed that the pits on the surface of R-PET fibres seem to be flatter and less deep. In contrast, as the alkaline treatment of PEG-M-PET fabrics progressed deep cavities formed which ran parallel to the fibre axis. The scanning electron micrographs of the hydrolyzed PEG-M-PET and R-PET are similar to those presented elsewhere<sup>4,18,19</sup>.

**Mechanical Properties**

Table - 3 illustrates the effect of alkaline treatment on some mechanical properties of PEG-M-PET and R-PET fabrics. Based on the data listed in this table, one can conclude the following:

- Breaking strength decreased continuously with an increase in weight reduction irrespective of type of treated PET fabric.

**Table 3: Effect of Alkaline Treatment on Some Mechanical Properties of PEG-M-PET and R-PET fabrics**

Fabric	WL (%)	Breaking strength (kg)	Breaking elongation (%)	Decrease in breaking strength (%)	Flexural rigidity (mgr)	Surface roughness (µm)
PEG-M-PET	0	54.0	43.0	-	668	16.3
	6.80	48.1	35.0	9.60	418	14.3
	24.0	35.5	30.0	34.2	267	13.0
	35.0	30.0	25.0	44.4	258	12.2
	50.0	21.0	22.0	61.1	231	11.3
R-PET	0	59.0	38.0	-	694	21.6
	5.20	46.0	28.5	22.0	516	18.0
	9.20	42.0	24.0	29.0	498	15.1
	21.6	-	-	-	302	15.1
	37.6	35.5	18.0	39.8	-	-

**Table 4: Effect of Alkaline Treatment on Thermal Properties of PEG-M-PET and R-PET Fabrics**

Fabric	Weight Loss % after alkaline treatment	T <sub>g</sub> (°C)	T <sub>m</sub> (°C)	Heat of fusion ΔH <sub>f</sub> (J/g)	Initial decomposition temperature, IDT (°C)	Temperature (°C) at Maximum decomposition rate dw/dt (mg/min.)	Decomposition temperature at various % weight loss (°C):			
							10	20	40	80
PEG-M-PET	Zero	63.0	249	50.9	353	447	414	428	442	482
	14.0	56.0	247	51.5	340	445	415	424	439	-
	31.5	53.0	245	45.8	327	441	401	416	431	471
	50.0	52.0	250	39.9	325	428	393	401	418	449
R-PET	Zero	69.0	251	50.4	352	449	410	423	438	473
	8.70	58.0	251	49.5	347	448	410	423	438	472
	17.2	55.0	252	47.7	337	428	394	408	420	452
	40.2	54.0	252	44.2	329	424	379	397	414	449

The behaviour of elongation is the same as that of breaking strength. The alkaline treated PEG-M-PET samples showed a higher decrease in breaking strength as compared to the R-PET weight reduced fabrics. The decrease in breaking strength at higher weight losses may be due to the formation of pits on the surface which probably act as weak points when fabric is elongated

under stress<sup>4</sup>. In addition, with an increase in weight reduction the internal structure gets affected as reflected in changes in crystallinity and the breaking down of the interchain H-bondings<sup>17</sup>.

- In general, the alkaline treatment of both PEG-M-PET and R-PET fabrics leads to a decrease of flexural rigidity and imparts the fabrics a soft feel. This decrease becomes more

pronouncing with increasing weight loss treatment. But a comparison between the effect of weight loss on decreasing flexural rigidity would indicate that the decreasing percent for PEG-M-PET fabrics is higher than for R-PET irrespective of the weight loss. Rate of lowering flexural rigidity, which is relatively fast in the beginning, becomes progressively slower with

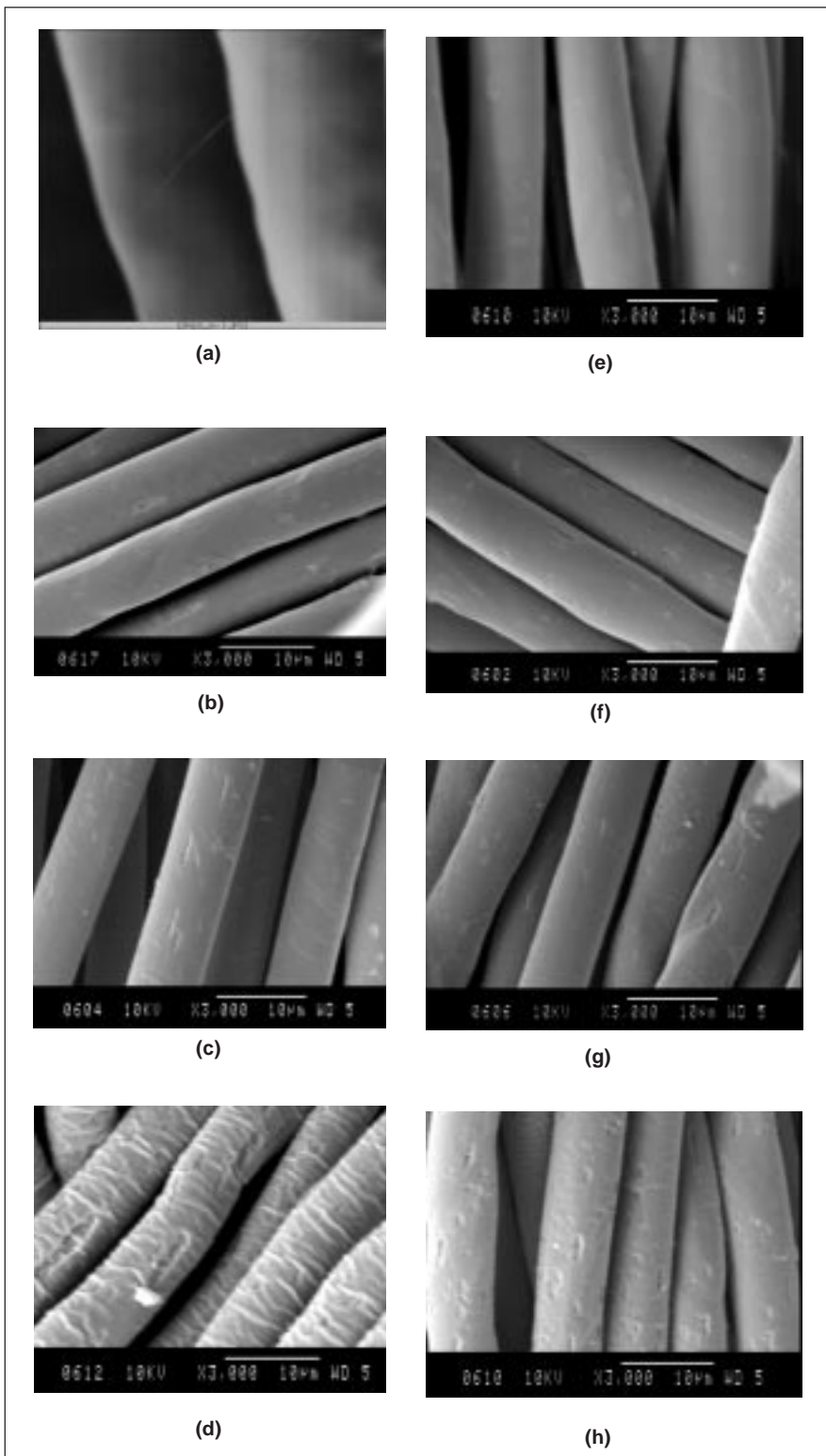


Figure 5: Scanning electron micrographs for untreated and alkaline treated PEG-M-PET and R-PET fabrics

PEG -M- PET	R-PET
(a) PEG - M-PET	(e) R-PET
(b) PEG - M-PET (WL=5.2%)	(f) R-PET (WL=8.7%)
(c) PEG - M-PET (WL=31.5%)	(g) R-PET (WL=17.2%)
(d) PEG - M-PET (WL=50.0%)	(h) R-PET (WL=40.0%)

increasing weight loss in the treatment. For instance, in the case of PEG-M-PET fabrics, a decrease of 37% in the value of flexural rigidity is obtained at a weight loss of 6.3%. Increasing weight loss up to 24% is accompanied with a significant decrease (60%) in flexural rigidity. Further increase in weight loss up to 35% and 50% causes no significant decrease in flexural rigidity.

- In general alkaline treatment decreases surface roughness. Data recorded in Table- 3 show a decreasing trend with increasing weight loss of treatment. The untreated and treated PEG-M-PET fabrics are characterized by surface roughness less than that for R-PET fabrics.

#### Thermal Properties

The thermal properties of untreated and treated with NaOH solutions PEG-M-PET and R-PET fabrics were investigated by studying their DSC and TGA thermograms. The results are presented in Table - 4.

It may be seen that in case of PEG-M-PET fabrics up to a weight loss of about 14% after alkaline treatment the glass transition temperature ( $T_g$ ) values decreased as compared to that of untreated PEG-M-PET sample. However, beyond that value the depression is small or not very significant. At the same time, in the case of R-PET fabric, the degree of depression in  $T_g$  values is more prominent and with the increase in the weight loss % after alkaline treatment there is a continuous decrease in the  $T_g$  values. The same trend appears also in the case of melting temperature ( $T_m$ ) and heat of fusion ( $\Delta H_f$ ) values. It appears in these cases that the change in structural parameters (crystallinity and orientation) changed as compared to that of

parent samples to a much wider extent. This may lead to a change in crystallinity/amorphous ratio, thus leading to a slight change in the melting temperatures.

From the results, it may be seen also, that with an increase in weight loss % after alkaline treatment, the initial decomposition temperature (IDT) and the decomposition temperature ( $T_d$ ) decrease continuously in all the cases of % weight loss as compared to that of parent PEG-M-PET and R-PET fabrics. But at comparable loss weight % after alkaline treatment and equal decomposition % weight loss, the  $T_d$  for PEG-M-PET fabrics is always higher than that for R-PET ones; a point which signifies the role of PEG moieties in PET macromolecule. It is worth-mentioning that the increased value of  $T_d$  in case PEG-M-PET as compared with R-PET could be due to the presence of the ether link in PEG-M-PET, which is characterized with more thermostability than the ester group.

## CONCLUSION

- Alkali treatment revealed that Polyethylene glycol modified poly ethylene terephthalate

fabrics are more sensitive to alkaline treatment than regular poly ethylene terephthalate fabrics. At any alkaline hydrolysis conditions, the values of weight loss for PEG-M-PET are always higher than those for R-PET; the point which paves the way for shortening the treatment time to achieve weight losses similar to those for R-PET.

- Alkaline treated PEG-M-PET fabrics are characterized with deeper cavities on the surface which ran parallel to the fibre axis, higher decrease in breaking strength, higher decrease of flexural rigidity, less surface roughness, lower glass transition temperature ( $T_g$ ), and higher decomposition temperature ( $T_d$ ) as compared to the R-PET weight reduced samples.

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