Coating of Macroemulsion and Microemulsion Silicones on Poly(ethylene terephthalate) Fibers: Evaluation of the Thermal Properties and Flammability

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ABSTRACT: Silicone softeners are widely used in the textile industry to improve the soft handling and aesthetic feel of textiles. These compounds can influence the thermal characteristics and flammability of polyester fabrics, which need to be addressed as important issues of human safety. To do this, the thermal degradation and flammability of macroemulsion and microemulsion silicone coated polyester fibers were studied with various analytical techniques, namely, differential scanning calorimetry, thermogravimetric analysis, dynamical mechanical thermal analysis, and limited oxygen index testing. It has been stated that the electrostatic and hydrophobic interactions of silicone emulsions with polyester can affect their thermal stability. On the other hand, the silicone emulsions present on the surface of poly(ethylene terephthalate) provide a three-dimensional scaffold, which produces faster combustion than pyrolysis. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 125: 1430–1438, 2012

Key words: coatings; degradation; silicones

INTRODUCTION

Polyester is the most important man-made fiber, with major advantages of a high modulus and strength, stiffness, stretch, wrinkle and abrasion resistance, relatively low cost, convenient processability, tailorable performance, and easy recycling.[1–4]

Silicone emulsions are a separate class of man-made softeners, which are widely used in the textile industry with outstanding specifications, including in the soft handling and aesthetic feel of textiles.[5–8] The improved lubricity of fibers, yarns, and fabrics inhibit abrasion and enhance soiling and wrinkle resistance, stretch recovery, tear strength, static protection, ironing, water repellency, and antifoaming during fabric finishing.[9–11] These compounds also reduce pilling, shrinkage, and sewing thread breakage.[12–14] Silicone emulsions are available as nanoemulsions, microemulsions, and macroemulsions, depending on the polymer architecture and functionality selected. Microemulsions are able to penetrate into the yarns and fabric with a very thin coating of silicone, bringing a soft and dry feel to fabric. Macroemulsions deposit on the external surface of the fabric, causing superior lubrication through a reduction of the dynamic coefficient of friction. These compounds can influence the flammability of such fabrics. In this regard, the flammability of silicone-softener-finished polyesters used in garments needs to be addressed as an important issue of human safety.[15–19]

All previous studies on the application of softener emulsions on textiles have focused on their effects on different physical, mechanical, and chemical properties, and only two research programs have explained the flammability of treated poly(ethylene terephthalate) (PET).[20–32] The effects of cationic household fabric softeners on the flammability of PET fabrics were studied by Gue et al.[33] They suggested that the rinse-cycle softener increased the flammability of polyester, but the dryer-sheet softener had no significant effect on the flammability of PET. Drews et al.[34] focused on the influence of silicone finishes on the burning behavior of PET. PET is suggested to be a moderately flammable fiber that melts, shrinks, and drips on contact with a flame. Researchers have found that silicone oils coated on PET may provide wicks for burning and accelerate the burning speed significantly. This phenomenon is stated as the scaffold effect of inorganic Si—O—Si, which forms a three-dimensional structure that reduces the tendency to of a fabric to shrink away from an ignition source. In other words, the increased flammability of the samples was attributed to the alteration in the melting behavior of the polyester.[34,35]

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TABLE I
Properties of the Polyester Fabric Used

Fabric type	Area density (g/m^2)	Number of wraps (1 cm)	Number of wefts (1 cm)	Wrap count (Nec)	Weft count (Nec)	Structure
Polyester	156	25	20	40	40	Plain

The influence of macroemulsion and microemulsion silicones as commercial textile finishing materials on the physical and mechanical properties of different fibers is well known, but in this respect, we wanted to study the effects of the type and amount of emulsion on the thermal stability and flammability of 100% polyester fabrics.

EXPERIMENTAL

Materials

Polyester fabric was used, and its properties are shown in Table I.

Nonionic detergent from Shirley Development, Ltd. (Stockport Cheshire, UK) was used for scouring. The macroemulsion and microemulsion silicone softeners were Siligen MS and Supersoft T_6, supplied by two companies, BASF, Germany and Arman Chimi Afrooz, Tehran, Iran respectively. Siligen MS was a nonionic amino-modified macroemulsion silicone fluid. It appeared opaque white with a total emulsion content of 20% and a silicone content of 15%. Its viscosity was 680 mPa s, and the average particle size was 148 nm. The other softener used was Supersoft T_6 as a nonionic microemulsion silicone softener. Its total emulsion and silicone contents were 45 and 24%, respectively. The internal phase viscosity was 200 mPa s, and its average particle size was 60 nm. Acetic acid from Merck was used for the softener bath.

Preparation and coating of the silicone emulsions on the polyester fabrics

The polyester fabrics were first scoured with 1% nonionic detergent for 30 min at 50°C with a liquor-to-fabric ratio of 40 : 1. Samples were then treated with two concentrations, 10 and 30 g/L, of macroemulsion and microemulsion silicones in water at 30°C by the padding method and 70% wet pickup. The pH was maintained at 5 with 0.5% acetic acid. The treated fabrics were then dried and cured in an oven at 130°C for 40 s. All of the treated fabrics were washed separately at 50°C for 10 min with a 1% nonionic detergent. It was found useful for removing the loosely bonded softeners.

All measurements were repeated five times, together with calculation of the standard deviation (the coefficient of variation was below 5% in all cases).

Calorimetric analysis

Differential scanning calorimetry (DSC) of the samples was carried out with a PerkinElmer Pyris 6 DSC model integrated with an IBM personal computer. The samples were heated from 30°C up to 350°C at a rate of 5°C/min in a nitrogen atmosphere. To gain insight into the effects of the macroemulsion and microemulsion on the degree of crystallinity (X_c) of the PET fiber, the normalized X_c values of the samples were determined with eq. (1):

$$X_c = (\Delta H_m / \Delta H_m^0) \times 100 \qquad (1)$$

where ΔH_m^0 is the melting enthalpy of 100% crystalline PET ($\Delta H_m^0 = 140.1$ J/g[5,6]) and ΔH_m is the normalized melting enthalpy of the sample.

Thermogravimetric analysis (TGA)

The thermal degradation analysis (TGA) of the samples was performed on a PerkinElmer thermoanalyzer (Pyris Diamond SII). In each case, a 5-mg sample was examined under N_2 at a heating rate of 5°C/min from room temperature to 600°C.

Dynamic mechanical thermal analysis (DMTA)

The tensile storage modulus and tensile loss factors of the coated fibers were measured by a dynamic mechanical thermal analyzer (DMA-TRITON Tritec 2000 DMA model) from Netzsch Co. The scans were made in single-cantilever mode at a constant heating rate of 5°C/min and at a frequency of 1 Hz from 25 to 300°C for 55 min.

Determination of the flammability by limited oxygen index (LOI) testing

LOI measurements were conducted on FTA LOI apparatus (Stanton Redcroft, The Netherlands) according to ASTM D 2863 (Fig. 1). The LOI measured the minimum oxygen concentration (in a flowing mixture of oxygen and nitrogen gas) required to support candlelike downward flame combustion. All measurements were the LOI mean values for five samples.

TABLE II
Thermal Behavior of the Nontreated PET Fibers and PET Fibers Treated with 10 and 30 g/L of Macroemulsion and Microemulsion Silicone Softeners

Sample	Quantity of softener in the solution (g/L)	T_g region (°C)	T_m (°C)	ΔH_m	X_c (%)	T_i^D (°C)	T_5^D (°C)	$Wt_{600°R}$ (%)
Nontreated	—	81.7 (0.12)	258.1 (2.1)	41.6 (0.11)	29.6 (0.05)	373.5 (3.2)	395.5 (3.5)	16.0 (0.01)
Treated with macro-	10	83.5 (0.11)	255.4 (3.4)	30.9 (0.09)	22.0 (0.03)	369.5 (3.7)	395.3 (2.7)	9.6 (0.01)
emulsion silicone	30	83.6 (0.10)	255.5 (1.5)	34.1 (0.10)	24.3 (0.05)	369.1 (1.2)	396.0 (1.2)	13.1 (0.01)
Treated withmicro-	10	99.6 (0.11)	249.1 (1.3)	34.7 (0.07)	24.0 (0.04)	365.9 (2.5)	372.8 (2.2)	12.3 (0.01)
emulsion silicone	30	99.9 (0.13)	248.4 (1.7)	35.8 (0.06)	25.5 (0.03)	363.6 (0.9)	387.5 (3.1)	4.0 (0.01)

Standard deviations are given in parentheses.

RESULTS AND DISCUSSION

DSC studies

Figures 2–4 show the DSC results of the nontreated PET fiber and those treated with 10 and 30 g/L macroemulsion and microemulsion silicones. The thermal parameters estimated from the DSC and TGA graphs are also given in Table II. All DSC curves related to PET and those treated with macroemulsion and microemulsion silicones did not show an identical pattern at different temperatures from 40 to 350°C. The DSC curve for the nontreated PET indicated a relatively endothermic region around 80–137°C. This was further followed by a sharp endothermic peak at about 258°C. The first endothermic region could have been a result of the glass-transition temperature (T_g) of the nontreated polymer. The second sharp endothermic peak at 260°C indicated the melting temperature (T_m) of the PET polymer.

As shown in Figures 2 and 3, a 10 g/L concentration of macroemulsion and microemulsion softeners in solution increased the T_g values for the fabrics to 83.5 and 99.6°C, respectively. An increase in the softener concentration to 30 g/L caused little effect on T_g for both softener-treated samples. It can be seen from Table II that the T_g, enthalpy, and crystallinity

percentage for the microemulsion-treated fabrics were also greater than those of the macroemulsion-treated ones. The results obtained from Figures 4 and 5 show that 10 and 30 g/L concentrations of the microemulsion silicones caused a greater increase in T_g of the polyester compared with those of the macroemulsion silicone. The DSC results also show that T_m ranged from 248 to 255°C for softener-treated fabrics, and they were lower than that of the nontreated fabric (T_m = 258°C). There were also decreases in the enthalpy and crystallinity percentage for all of the softener-treated samples compared with nontreated PET, as illustrated in Table II.

The T_g value was due to the molecular movement of PET. The results obtained from the T_g values of macroemulsion- and microemulsion-treated samples indicated that the amorphous molecules of PET became mobile at higher temperatures than the ones in the nontreated PET. Softener chains bonded to the PET surface and/or oriented on the surface by means of their electronic charges and their hydrophobicity. Both electrostatic and hydrophobic attractions were driving forces that affected the thermal characteristics of PET. The deposition of silicone softeners on polyester was believed to be a result of the two following effects: (1) electrostatic attractions

Figure 1 Flame chamber used in this study with horizontal position for samples.
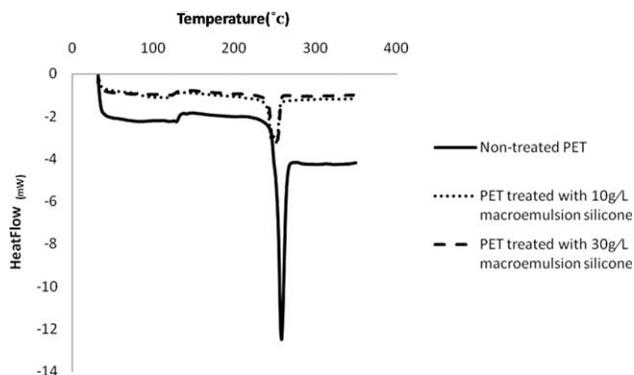
Figure 2 DSC curve of non-treated PET fiber and those treated with 10 g/L and 30 g/L macroemulsion silicone softener.
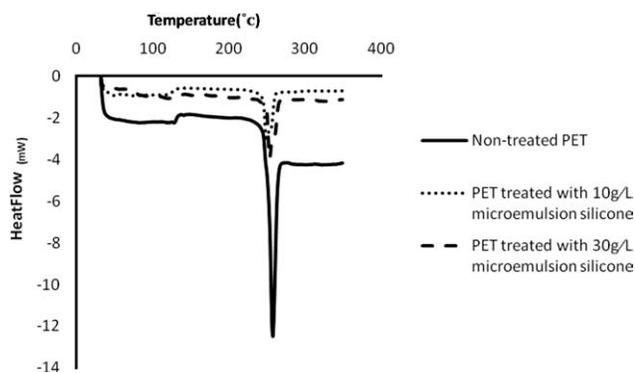
Figure 3 DSC curve of non-treated PET fiber and those treated with 10 g/L and 30 g/L microemulsion silicone softener.

between the positive center of the softener and the weakly negative charges generated by the polyester in water and (2) van der Waals interactions between the softener hydrophobic structure and the aromatic π-electron systems of the benzene rings in the PET chains. It can be stated that a microemulsion silicone consists of more functional groups on their polymer chains compared with a macroemulsion softener, which increases the interactions between the silicone polymer and PET, as can be seen from the T_g values.[16,17]

Both softeners also caused some characteristic decreases in the T_m, enthalpy, and crystallinity percentage of PET. The softener-treated samples consisted of a complex system of polyester and softener; therefore, their thermal parameters could have been influenced by interactions between these two systems. Polyester fiber has a semicrystalline structure, and it consists of highly organized crystals and randomized amorphous regions with high-molecular-weight chains.[36–38] On the other hand, softeners have generally shorter chain length molecules, which can penetrate easily into the yarn center or deposit on the surface of the yarns; this results in an internal plasticization of the fiber with decreasing thermal characteristics.[24,25,29,30]
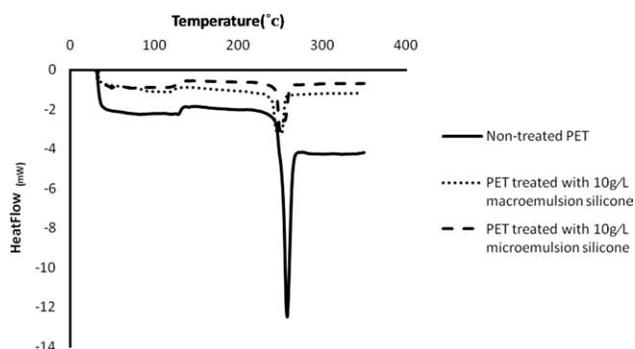
Figure 4 DSC curve of non-treated PET fiber and those treated with 10 g/L macro- and microemulsion silicone softeners.
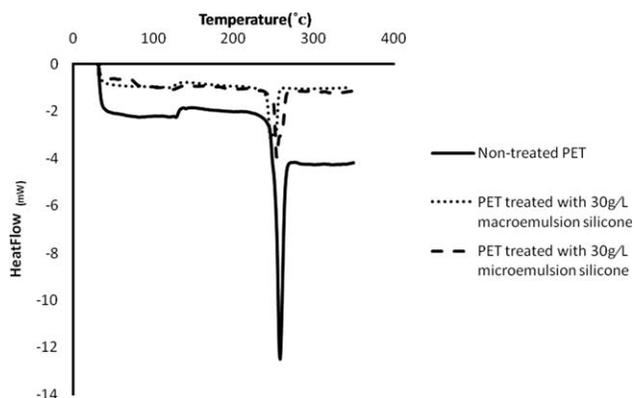
Figure 5 DSC curve of non-treated PET fiber and those treated with 30 g/L macro- and microemulsion silicone softeners.

The main factors affecting the T_m, enthalpy, and crystallinity percentage of different polymers include intermolecular bonding, structure, and chain flexibility. Silicone softeners with flexible chains would be expected to have lower melting points, enthalpies, and crystallinity percentages than PET; this would decrease the thermal characteristics of treated fibers.[32]

Thermogravimetric characterization

Figures 6–9 summarize the thermal degradation of the nontreated polyester and those treated with 10 and 30 g/L macroemulsion and microemulsion silicone softeners at different temperatures. The characteristic decomposition temperatures derived from these figures are presented in Table II. The softeners showed lower thermal degradation than the PET and softener-treated samples. These curves indicate that the softeners consisted of short-chain polymers compared with long chains of fibers. The weight losses due to the decompositions of nontreated PET and those treated with softeners were nearly the
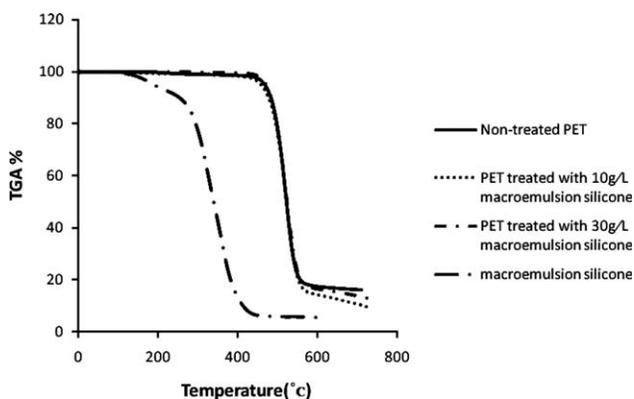
Figure 6 TGA thermograms of macroemulsion silicone, non-treated PET fiber and those treated with 10 g/L and 30 g/L macroemulsion silicone softener.
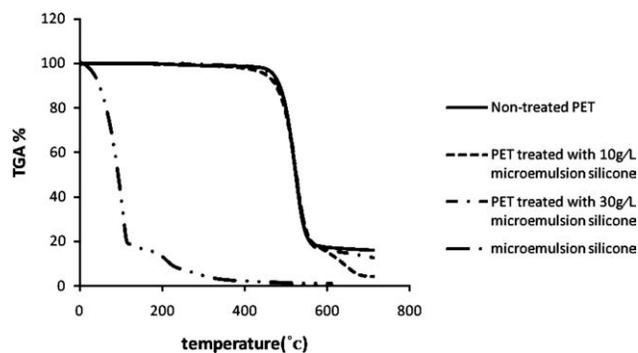
Figure 7 TGA thermograms of microemulsion silicone, non-treated PET fiber and those treated with 10 g/L and 30 g/L microemulsion silicone softener.

same up to a temperature of about 363°C. After this point, the initial thermal degradation (T_i^D) was influenced by the softener type and concentration. As can be seen, differences of the T_i^D values of the samples treated with 10 and 30 g/L macroemulsion softener were negligible. Both macroemulsion and microemulsion silicones decreased T_i^D of PET, and a greater decrease in T_i^D was observed with increasing microemulsion silicone from 10 to 30 g/L. The temperatures at a weight reduction of 5% (T_5^D's) for the 10 g/L macroemulsion and microemulsion silicone softener-treated samples were observed at 395 and 372°C, respectively; this indicated that the type of softener played a pivotal role in further degradation of the samples. The viscosity (chain length), silicone content (%), and particle size (nm) were three parameters of the silicone emulsions that influenced the thermal degradation of the polyester. The particles of the microemulsion silicones could penetrate more easily into the yarn center because of their size; this resulted in an inner three-dimensional structure of Si—O—Si, whereas macros deposited on the surface of the yarns. In other words, the thermal properties of the fibers depended to a large extent on the silicone content and chain length. Microemulsions with higher silicone contents produce an
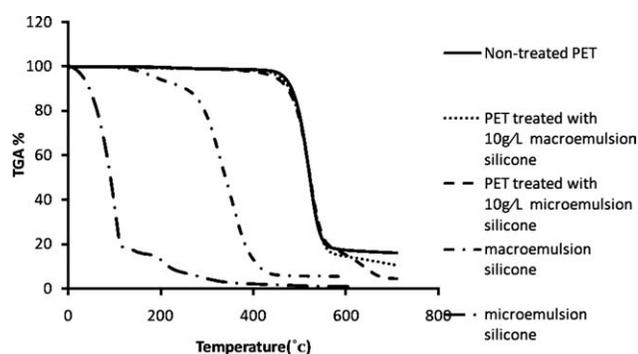
Figure 8 TGA thermograms of macro- and microemulsion silicones, non-treated PET fiber and those treated with 10 g/L macro- and microemulsion silicone softener.
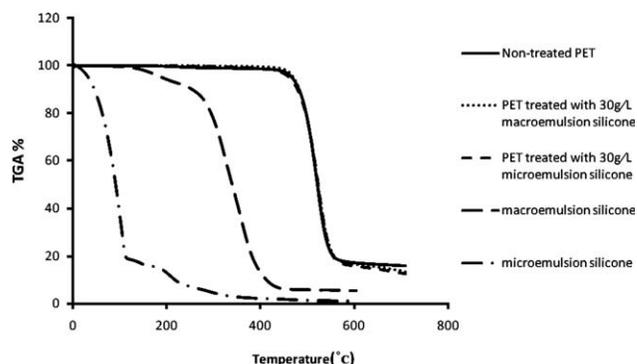
Figure 9 TGA thermograms of macro- and microemulsion silicones, non-treated PET fiber and those treated with 30 g/L macro- and microemulsion silicone softener.

increased silicone concentration inside the textile structure and tend to have less thermal stability.[26,27] The weight of the residue at 600°C (Wt$_{600°R}$) decreased for all of the treated fabrics and ranged from 13 to 4%. The results demonstrate that the amount of char and the thermal stability were lowered for samples treated with the silicone softeners. When one considers the thermal degradation results, it was consistently believable that the introduction of silicone emulsion onto the PET textile could have reduced its thermal stability and char formation on the basis of the low heat resistance of the softener at a high temperature of 600°C.

Because softeners are mostly on the surface of fibers and yarns, they are more accessible to heat; this, thereby, decreases the thermal decomposition temperature of PET. When more microemulsion softener aggregates were present on the PET fabrics, less char was produced so that more volatile combustible materials were available to maintain the heat and decrease the thermal resistance. This result was also confirmed by other authors with regard to the combustion behavior of flame-
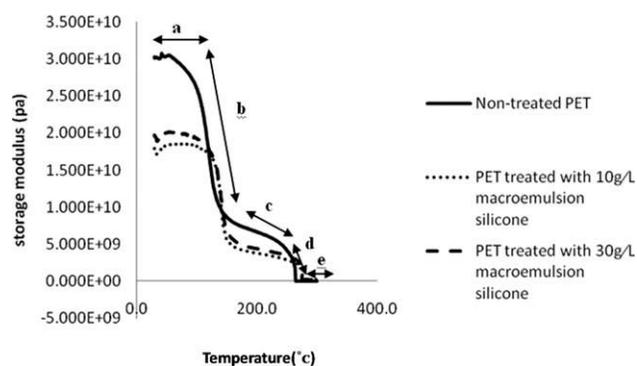
Figure 10 The storage modulus versus temperature for non-treated PET fiber and those treated with 10 g/L and 30 g/L macroemulsion silicone softener- (a) glassy region, (b) glass-transition region, (c) rubbery region, (d) rubbery flow region, (e) liquid flow region.
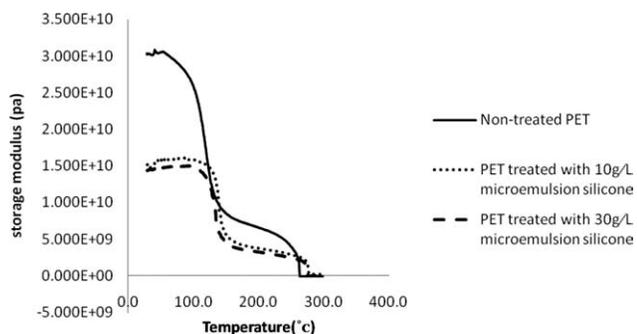
Figure 11 The storage modulus versus temperature for non-treated PET fiber and those treated with 10 g/L and 30 g/L microemulsion silicone softener.

resistant thermally protective textiles in the presence of oily contaminants. The TGA results were in agreement with the results obtained from the DSC measurements.[32,36,37,39]

DMTA

The dynamic mechanical thermal parameters, including storage modulus and tan δ, indicate a polymer's stiffness, molecular motion, relaxation process, and structural hetero groups and the morphology of the polymer blend systems.[36–38] This process was used to identify information on molecular changes within the polymer and changes in fiber displacement due to surface lubricity; these results are presented in Figures 10–13. The storage modulus graph of polyester could be divided into five parts, including glassy, glass-transition, rubbery, rubbery flow, and liquid flow regions (i.e., Fig. 10). The glass-transition range was characterized by a sharp decrease in the storage modulus of the polyester and the emulsion-treated fibers. As can be seen in Figures 10 and 11, the storage modulus curves for the sample treated with 10 g/L microemulsion silicone were started at lower values compared with those of
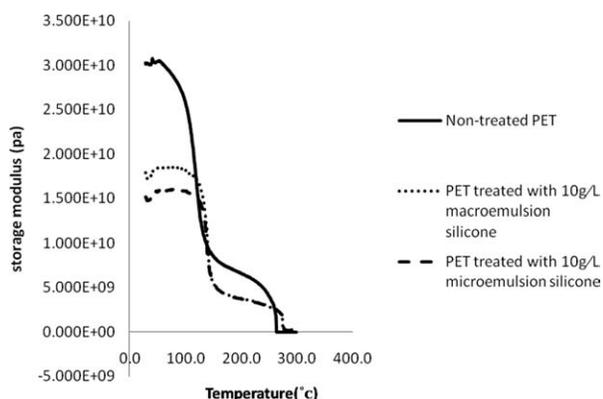
Figure 12 The storage modulus versus temperature for non-treated PET fiber and those treated with 10 g/L macro- and microemulsion silicone softener.
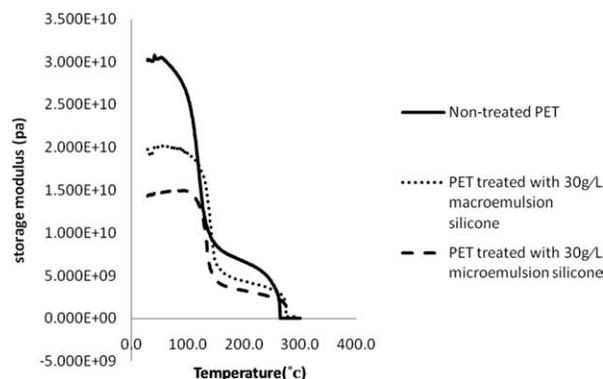
Figure 13 The storage modulus versus temperature for non-treated PET fiber and those treated with 30 g/L macro- and microemulsion silicone softener.

the nontreated one, and an increase in the softener concentration to 30 g/L caused a greater decrease in the modulus values. This behavior could be seen in the glassy and rubbery regions for both softeners. On the other hand, samples treated with the macroemulsion softener showed higher modulus values compared with the microemulsion-treated PET, as illustrated in Figures 12 and 13. As shown by the TGA figures, the macrosilicone softener increased the modulus values through the formation of a high-molecular-weight network on the surface of the textile. In the case of microemulsions, elastic silicone polymeric chains formed a low-molecular-weight network in the inner parts of the yarns, thus improving the fabric's modulus values. As the results show, macroemulsion silicones are more useful for the thermal stability of polyester fibers compared with microemulsions. This was shown by the values obtained from the DMTA figures.[26,27] A decrease in the modulus values for all of the samples at higher temperatures of 250°C was relevant to the premelting process. However, there was a different behavior in the modulus values for the macroemulsion- and microemulsion-treated samples at higher
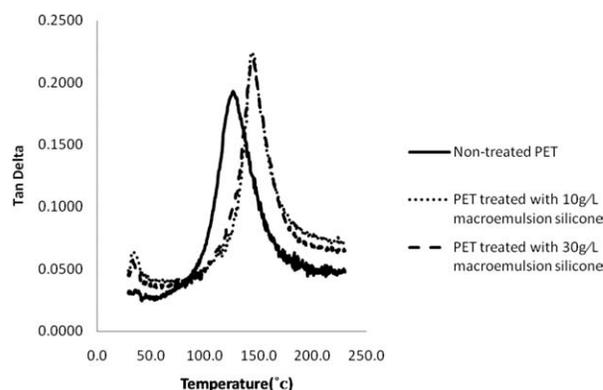
Figure 14 The tan delta versus temperature for non-treated PET fiber and those treated with 10 g/L and 30 g/L macroemulsion silicone softener.
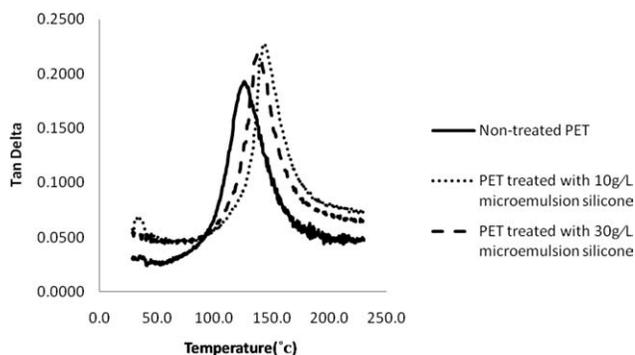
Figure 15 The Tan delta versus temperature for non-treated PET fiber and those treated with 10 g/L and 30 g/L microemulsion silicone softener.

temperatures of 250°C; this showed the electrostatic and van der Waals interactions of silicone emulsions with PET chains. In other words, the ionic forces of silicone softeners played a great role in the thermal behavior of the coated fibers. This result was incidentally consistent with our results obtained from DSC.

The dependencies of tan δ on the temperature for the nontreated PET and those treated with softeners are shown in Figures 14–17. The T_g values of the coated fibers were obtained from the tan δ peaks. The tan δ values for all of the samples increased sharply up to their maximum values because of the increase in viscosity and then decreased as the temperature was further increased. Finally, the tan δ values were permanent at higher temperatures below the melting point. The maximum tan δ at about 115°C for the nontreated PET corresponded to segmental relaxation related to the glass–rubber transition in the fiber. The tan δ values were higher for the PETs treated with 10 and 30 g/L macroemulsion and microemulsion silicone softeners. It could also be seen that the tan δ peak was shifted to higher temperatures for all of the softener-treated samples. These changes were attributed to the elastic nature and surface properties of the macrosilicone and
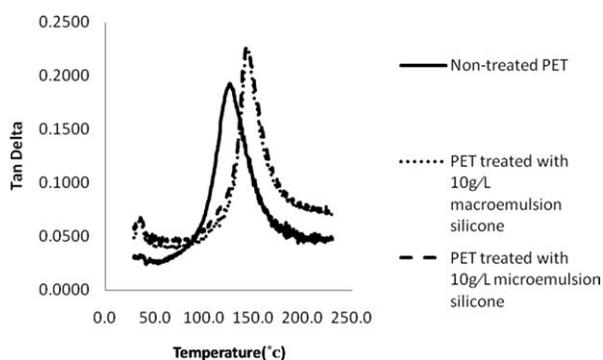
Figure 16 The Tan delta versus temperature for non-treated PET fiber and those treated with 10 g/L macro- and microemulsion silicone softener.
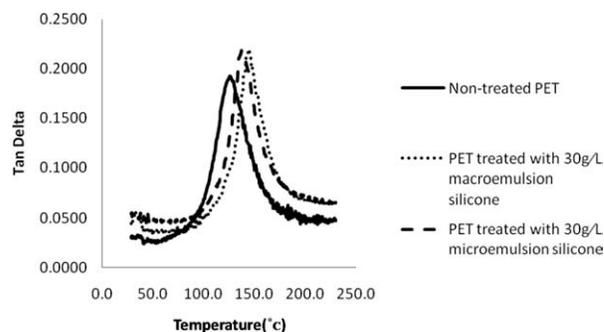
Figure 17 The Tan delta versus temperature for non-treated PET fiber and those treated with 30 g/L macro- and microemulsion silicone softene.

microsilicone softeners, which affected the interactions of the PET–softener system, and the lower segmental motions of the PET chains, which resulted in an increase in the tan δ value. This result was confirmed by other authors for fibers treated with various lubricating agents.[32,34,39]

Flammability

Table III shows the LOI (%) values for the nontreated and softener-treated polyester textiles. A lower LOI percentage indicated a greater flammability.

When a polyester fiber is exposed to flame, it does not ignite easily because of its tendency to shrink away. However, from Table III, it is clear that the LOI percentage for the samples treated with 10 g/L softener solutions decreased compared with that of the nontreated fabric. An increase in the softener concentration caused a greater decrease in the LOI percentage. Results obtained from the flammability test revealed that the microemulsion silicone caused a greater increment in the flammability compared with the macroemulsion one. This result confirmed previous results obtained from thermal studies of the samples. As was stated earlier, microemulsions can generate a network inside the fabric structure,

TABLE III
LOI (%) Values for the Nontreated PET Fibers and PET Fibers Treated with Macroemulsion and Microemulsion Softeners

Sample	Quantity of softener in the solution (g/L)	LOI (%)
Nontreated	—	19.7 (0.8)
Treated with macro-emulsion silicone	10	19.3 (0.6)
	30	19.1 (0.6)
Treated with micro-emulsion silicone	10	18.9 (0.2)
	30	18.5 (0.5)

All measurements are the LOI mean values for five samples.

whereas macros deposit on the surface of the fiber by the formation of a high-molecular-weight network because of their high viscosity. A microemulsion silicone network may decompose inside the fiber structure by forming silicone dioxide. This can serve as a fuel for melted fibers inside the textile. On the other hand, macroemulsion silicone can increase the volatile fuel at the surface of a fiber with less flammability.[26,27,39]

Flame is a gas-phase phenomenon that cannot convert a textile as a nonvolatile material into a gaseous form by combustion. The fiber is decomposed initially by the flame to yield low-molecular-weight products that can volatilize from the surface and enter the flame. Softeners mainly consist of fatty acid compositions and lubricants, which are derived from tallow. They are more absorbed on the surface of a PET textile, which is more accessible to the flame. The flammability results obtained in this research were confirmed by the work of other researchers. They suggest that the rate of decomposition of the softener-treated textiles were higher for the softener than for the fiber. It can be stated that this process underwent considerable volatilization before burning, and the pyrolytic gasification process was simply evaporation. Others also claimed that softeners deposited on the fabric increased the amount of volatile fuel released for the combustion process and, thereby, increased the vigor of the combustion process.[9–11,32,39–48]

CONCLUSIONS

This study showed that type and amount of softener affected the thermal decomposition and flammability of polyester. The softeners affected the thermal behavior of PET in terms of the T_g, T_m, enthalpy, crystallinity percentage, initiation thermal degradation, and weight of the residue at high temperatures. PET decomposed at lower temperatures in the presence of softener, and it was highly affected by the amount of softener. The results show that electrostatic and hydrophobic forces affected the thermal characteristics of the softener-treated fabrics. All coated polyester fabrics also showed lower LOI percentages and slightly higher flammabilities compared with the nontreated fabric, and to a lesser extent, the quantity of softener applied to the fiber slightly influenced the combustion behavior. The softener present in the fabric surface added volatile products to the combustion process, increasing the amount of heat produced. Thus, it produced faster combustion by pyrolysis and decomposed the textile further until it was completely decomposed and formed char. We concluded that with respect to the concentrations used for this research, a 10 g/L macrosilicone softener is preferred for textiles that will be used in public spaces.

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