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Improvement of Burning Properties on the Cotton/polyester/lacra Blend Fabric with Nano Silicone Material in Nano Silicone

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Abstract: In this research, we established a modified 45 flammability test for cotton blend material to measure the flammability of nanosilicone softener treated-cotton blend material. The wet pick-up was about 150±10%. b) The specimens were tested in the cross direction and nine specimens were measured. The average percent weight loss of the untreated cotton blend nonwoven is only 0.9%. The percent weight loss of treated sample is much higher than that of the untreated and this increased with the softener concentration. The treated samples show much higher weight loss and burning area than those that are untreated.There are few publications elucidating the antagonistic defeat of the FR process for cotton blend observed with commercial finishes. TGA shows that the treatment with nanosilicone softener increases the thermal stability of cotton blend in air before the maximum decomposition rate temperature and increases the residue of cotton blend. This study will both quantify the effect and fully characterize the mechanisms by which siloxanes affect the chemical and physical processes that occur during the degradation and burning of cotton blend and then will investigate approaches to mitigating these effects.

Key words: Cotton . nano silicone . lacra . burning.

INTRODUCTION

Nanosilicones are a class of polymers having the formula Rims (O) 4-m/2]n, m=1-3 and 1 n" [1]. Nanosilicone products have been widely applied on textiles for various end uses. The primary applications of nanosilicones in textile are fabric softening agents, fiber and thread lubricants, antifoaming agents and antistatic agents. The treatment of nanosilicone products also achieves some additional benefits such as antimicrobial effect, water repellence and elasticity [2]. The increased flammability of nanosilicone treated-cotton blend was primarily attributed to the alteration in the melt characteristics of the cotton blend However, the effect of nanosilicone softener on the flammability of cotton blend materials has not been clearly defined yet. No testing method has been developed to test the flammability of cotton blend materials. The mechanisms of the increased flammability of nanosilicone softener treated-cotton blend are not clear as well. In this paper, we established a modified 45 flammability test for cotton blend material to measure the flammability of nanosilicone softener treated-cotton blend material. The mechanism of the increased flammability of nanosilicone softener treated-cotton blend was investigated. The thermo gravimetric analysis of the cotton blend mat was conducted as well. The most commonly used nanosilicone softener is polydimethylsiloxane (PDMS). Reactive polydimethyl siloxanes can achieve more durable effect than PDMS, such as hydrogen siloxanes, terminal silanols, epoxy reactive polydimethylsiloxanes and amino functional siloxanes [3]. However, nanosilicones have an antagonistic effect on the flame retardant properties of cotton blend. Cotton blend normally melts and flows at above 260°C without nanosilicone finish, cotton blend material shrinks away from ignition flame, melts and self extinguishes when subjected to a flammability test [4]. Weil discussed the antagonistic effect of nanosilicone oil on the flame retardant properties of cotton blend. There are several possible mechanisms for the increased flammability caused by nanosilicones: the scaffold effect attributed to thermal degradation of nanosilicones to form a three dimensional, cross linked, inorganic Si-O-Si or silica itself and the reduced tendency to shrink away from an ignition source caused by the lowered surface energy [5-7].

MATERIALS AND METHODS

We established a modified 45 flammability test for cotton blend material (Lab Tex Co.) to measure the flammability of nanosilicone softener treated-cotton blend material. The mechanism of the increased flammability of nanosilicone softener treated-cotton blend was investigated. The thermo gravimetric

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analysis of the cotton blend mat was conducted as well. The most commonly used nanosilicone softener is polydimethylsiloxane (e.g. PDMS (Bayer Co.)). Reactive polydimethyl siloxanes can achieve more durable effect than PDMS, such as hydrogen siloxanes, terminal silanols, epoxy reactive polydimethylsiloxanes and amino functional siloxanes.However, nanosilicones have an antagonistic effect on the flame retardant properties of cotton blend. Cotton blend normally melts and flows at above 260°C without nanosilicone finish, cotton blend material shrinks away from ignition flame, melts and self extinguishes when subjected to a flammability test.

RESULTS AND DISCUSSION

Effect of siloxanes on the burning behavior of cotton blend: An amino functional hydrophilic self-dispersible nanosilicone softener and a reactive nanosilicone softener were investigated for their effect on the burning behavior of nonwoven cotton blend. One cotton blend highly consolidated nonwoven with a density of 136.5 g/m^2 and a second lightly needle punched (to give fabric some integrity) cotton blend mat material with a density of 144.1 g/m^2.

Flammability test method for cotton blend material treated with an amino functional hydrophilic nanosilicone softener: The flame test procedure, ASTM D1230-94 was employed. A dried specimen was inserted in a frame and held in a special apparatus at an angle of 45°, a standardized flame was applied to the surface near the lower end for 5 seconds and the time required for the flame to proceed up the fabric a distance of 127 mm (5 in) recorded. The weight loss after the flammability test was recorded and the percentage weight loss was calculated based on the weight in the tested area. The arithmetic mean percentage weight loss of 9 or 10 specimens was used to determine the flammability of the cotton blend materials.

- The fabric was padded and dried in a laboratory oven at 140°C for 3 minutes. The wet pick-up was about 150±10%.
- The specimens were tested in the cross direction and nine specimens were measured.

The average percent weight loss of the untreated cotton blend nonwoven is only 0.9%, Table 1 The percent weight loss of treated sample is much higher than that of the untreated and this increased with the softener concentration.

Though the standard deviations of the percent weight loss are quite big, the t-values of the percent weight loss paired with the control are all larger than the critical t-value at 95% confidence level. Thus, there is significant difference in the percent weight loss between the control and the treated sample. We can determine whether the treatment of nanosilicone softener increases the flammability of cotton blend materials. Figure 1 shows images of the cotton blend nonwoven after flammability testing compared to that of the untreated control sample. The untreated sample, Fig. 1a, shows a small empty area primarily due to melt and shrinkage of cotton blend fiber. The treated samples have a much larger void and chars on the edge of the specimen holder due to fiber combustion, melt and shrinkage. The specimens show larger burned area at higher concentrations.

The flammability of cotton blend nonwoven and mat material finished with a reactive nanosilicone softener: The average percent weight loss, standard deviation of percent weight loss, t-values paired with the control, t-values paired with two concentrations of cotton blend nonwoven material (136.5g/m^2) are shown in Table 2. The treatment with the reactive nanosilicone softener has an obvious impact on the sample flammability. When the concentration is 0.1%, the percent weight loss is 38.0% after the flammability test. When the concentration is increased, no increase in the percent weight loss is observed. The t-values of the percent weight loss paired with control are all larger than the critical t-value at 95% confidence level. Therefore, there is significant difference in the percent weight loss between the treated and untreated samples. When the concentration is further increased, the flammability of the sample does not change much. Images of the cotton blend nonwoven after flammability testing are shown in Fig. 2.

The effect of reactive nanosilicone softener on the flammability of cotton blend mat material (144.1 g/m^2) was also studied. In comparison to the nonwoven material, the structure of the mat material is very fluffy and contains a considerable amount of air. The average percent weight loss, standard deviation of percent weight loss, t-values paired with the control, t-values paired with two concentrations of cotton blend mat material are shown in Table 3. When the concentration is 0.1%, the average percent weight loss is 26.6%, which is much higher than the control. When the concentration was 0.5%, the weight loss increased to 55.8%. The whole tested area is burned. Char and some small black fibers formed at the edge and across the sample holder (Fig. 3). However, the untreated cotton blend mat does not burn and has almost no weight loss

Table 1: The average percent weight loss, standard deviation of the percent weight loss, t-value and critical t-value of cotton blend nonwoven finished with amino functional hydrophilic self-dispersible nanosilicone softener after the flammability test

Concentration (%)	Average percent weight loss (%)	Standard deviation of percent weight loss	t-value (Paired with control)	Critical t-value (a = 0.05)
Control	0.89	1.28	-	-
0.5	12.94	9.10	4.1	2.02
1.0	21.00	9.20	6.3	2.02
1.5	23.98	12.50	5.5	2.02

Table 2: The average percent weight loss, standard deviation of percent weight loss, t-values paired with the control, t-values paired with two concentrations of cotton blend nonwoven materials treated with reactive nanosilicone softener

Concentration (%)	Ave. percent weight loss (%)	Standard deviation	t-Value (Paired with control)	t-Value (Two concentrations paired)	Critical t t-Value (a = 0.05)
Control	1.4	1.8	-	-	-
0.1	38.0	12.8	9.0	1.3 (0.1%&0.5%)	2.1
0.5	28.9	17.5	4.9	1.3 (0.1% and 1.0%)	2.1
1.0	29.1	17.2	5.1	0.03 (0.5%&1.0%)	2.1

a) The fabric was sprayed with a nebulizer with reactive nanosilicone softener solution and dried in an oven at 160°C for 10 min. The wet pick-up was about 110±10%; b). The specimens were tested in the machine direction and ten specimens were measured

Table 3: The average percent weight loss, standard deviation of percent weight loss, t-values paired with the control, t-values paired with two concentrations of cotton blend mat material treated with reactive nanosilicone softener

Concentration (%)	Ave. percent weight loss (%)	Standard deviation	t-Value (Paired with control)	t-Value (Two concentrations paired)	Critical t-Value
Control	0.8	0.8	-	-	-
0.1	26.6	16.6	4.9	5.0 (0.1%&0.5%)	2.1
0.5	55.8	7.8	22.2	3.0 (0.1%&1.0%)	2.1
1.0	47.3	13.9	10.6	1.7 (0.5%&1.0%)	2.1

a) Control b) 0.5% c) 1% d) 1.5%

Fig. 1a: Images of cotton blend nonwoven treated with nanosilicone softener after the flammability test

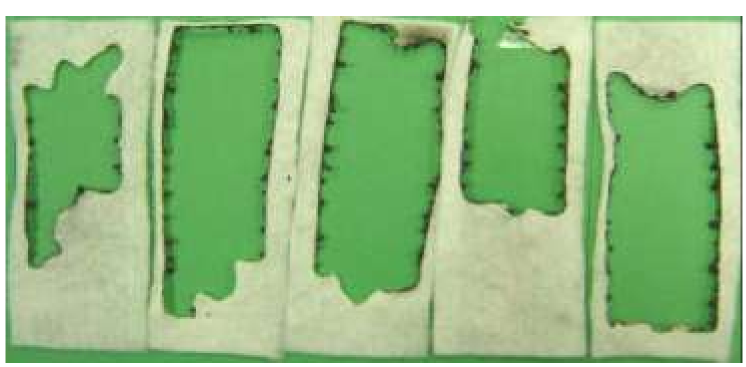
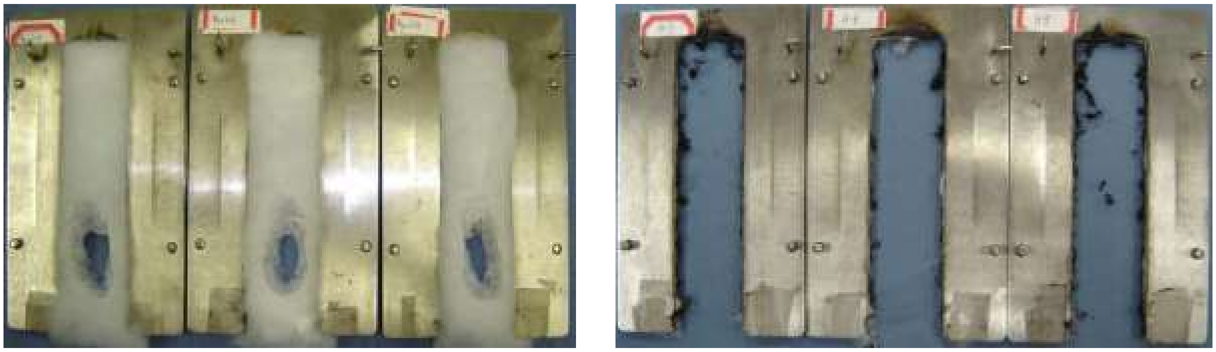
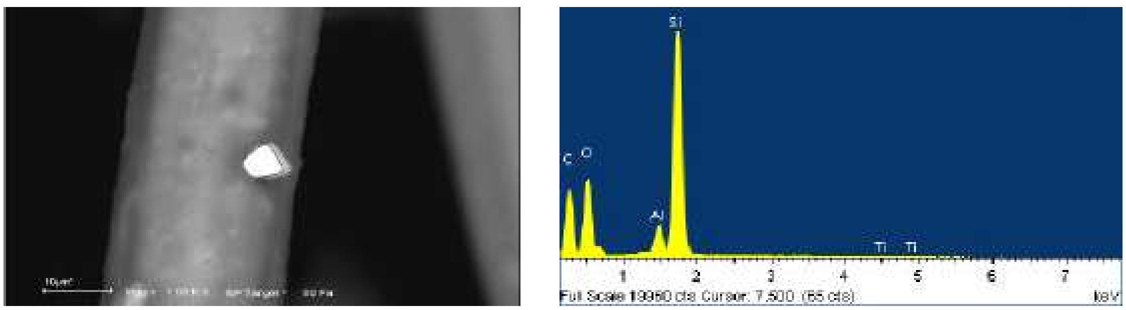


Fig. 2: Cotton blend nonwoven treated with 0.1% reactive nanosilicone softener after the flammability test



(a) Cotton blend mat, control (b) Cotton blend mat, 0.5%

Fig. 3: Cotton blend mat after the flammability test



a) SEM micrograph of cotton blend mat with nanosilicone particle b) EDX of spectrum of the nanosilicone particle

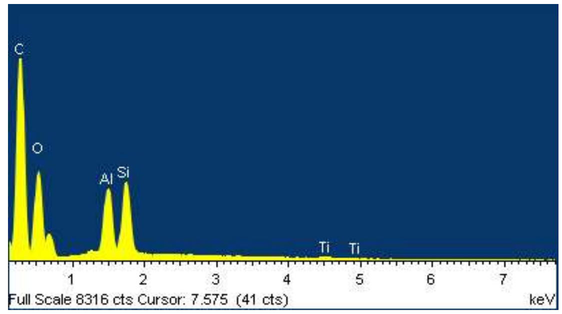
Fig 4: EDX spectrum of the nanosilicone particle on cotton blend mat treated with 0.5% reactive nanosilicone softener

after the flammability test. When the concentration of reactive nanosilicone softener is 1.0%, the weight loss is 47.3%. Most of the tested area is burned. All the t-values of the treated cotton blend mat are larger than the critical t-value at 95% confidence level. Thus, the flammability of the cotton blend mat treated with nanosilicone softener even at very low concentrations is significantly higher than the untreated cotton blend mat. The flammability of cotton blend mat treated with 0.5% and 1.0% reactive nanosilicone softener is significantly higher than that treated with 0.1% since the t-values paired with 0.1% and 0.5% and 0.1% with 1.0% are larger than the critical t-value (Table 3). The treatment of reactive nanosilicone softener seriously increases the flammability of cotton blend mat.

SEM and Energy Dispersive X-ray (EDX) analysis: EDX analysis is a semi-quantitative method providing relative element compositions in the scanned area. The quantity of the elements depends largely on the penetration ability of the X-ray. The SEM micrograph and EDX spectrum of the cotton blend mat treated with 0.5% reactive nanosilicone softener are shown in Fig. 4 and 5. Some nanosilicone softener particles are observed on the fiber surface in Fig. 4a. The EDX spectrum shows that the major compositions in the scanned area are carbon, oxygen, nanosilicon, aluminum and titanium (Fig. 4b). The aluminum comes from the SEM aluminum sample stub. The titanium comes from the delustrant TiO2 added during the cotton blend spinning process. The atomic compositions of carbon, oxygen and nanosilicon are 55.0, 35.2 and 8.6%, respectively (Table 4), which indicates that the particle is a nanosilicone softener particle. Hydrogen cannot be measured by the EDX. The EDX analysis of the area other than the particle is shown in Fig. 5b. Other than carbon and oxygen, the major components of cotton blend, nanosilicon is also observed on the fiber surface. Again, the aluminum comes from the aluminum sample stub. The element compositions of the scanned area other than the nanosilicone particle are shown in Table 5. The atomic composition of nanosilicon is 2.0%, much lower than that in the particle area. From the above discussion, nanosilicone softener accumulates on the cotton blend fiber surface as big particles and spreadsout on the fiber surface as invisible small particles.

The SEM micrographs of the melt surface of cotton blend mat after burning are shown in Fig. 6. Melted untreatedcotton blend has very high surface energy and intends to shrink away from the ignition source. Thus, the untreated cotton blend mat melts

(a) SEM micrograph indicating selected area for EDX,



b) EDX spectrum of the selected area

Fig 5: SEM micrograph and EDX spectrum of the selected area on cotton blend fiber of the mat treated with 0.5%reactive nanosilicone softener

Table 4:Element component of the particle on cotton blend mat treated with 0.5% reactive nanosilicone softener

Element	Weight (%)	Atomic (%)
C K	59.22	67.92
O K	32.55	28.02
Al K	3.79	1.93
Si K	4.18	2.05
Ti K	0.26	0.08
Totals	100.00	

Table 5: Element component of the selected area on cotton blend fiber of the mat treated with 0.5% reactive nanosilicone softener

Element	Weight (%)	Atomic (%)
C K	44.12	55.02
O K	37.58	35.18
Al K	2.02	1.12
Si K	16.22	8.65
Ti K	0.07	0.02
Totals	100.00	

Table 6: Element composition of the selected areaon the charred fiber btained from cotton blend mat treated with 0.5% reactive nanosilicone softener

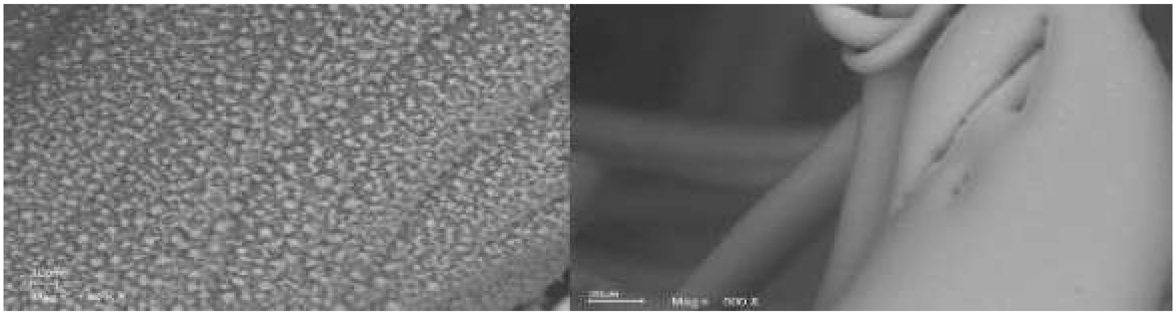
Element	Weight (%)	Atomic (%)
C K	66.91	74.98
O K	25.98	21.86
Al K	0.21	0.11
Si K	5.63	2.70
Ti K	1.27	0.36
Totals	100.00	

Table 7: Element composition of the selected areaon charred area of cotton blend mat treated with 0.5%reactive nanosilicone softener

Element	Weight (%)	Atomic (%)
O K	46.04	60.77
Al K	1.05	0.82
Si K	49.43	37.17
Ti K	2.62	1.15
Totals	100.00	

away from the flame and shows a very smooth melting surface (Fig. 6a). The melted surface of cotton blend mat treated with 1.0% reactive nanosilicone softener shows a rough surface with a large number of visible particles (Fig. 6b). After treatment with nanosilicone softener, the surface energy of cotton blend is lowered and cotton blend mat burns vigorously dropping flaming droplets instead of melting and shrinking away from the flame.

A number of small fibers form at the edge of the specimen holder and on the char after burning, Figure 7. A lot of bright charred fibers distinguished from cotton blend char are found in the SEM pictures. The bright charred fibers contain higher atomic weight elements since higher atomic weight elements have higher signal under back scattering detector. The EDX spectrum of the charred fiber obtained from cotton blend mat treated with 0.5% reactive nanosilicone softener after burning is shown in Figure 8. Nanosilicon shows a very intensive band in the EDX spectrum. The element composition of the charred fibers is shown in Table 6. The major components of the charred fiber are oxygen and nanosilicon and the mole ratio of oxygen to nanosilicon is around 1.67. Therefore, the most possible compositions of the charred fiber are nanosilicon dioxide and nanosilicon. The small amount of aluminum comes from the aluminum SEM sample stub. Nanosilicone softener decomposes to produce nanosilicon dioxide during the burning. Nanosilicon dioxide crystallizes to form nanosilicon dioxide fiber as the burning continues. It reduces the surface energy of cotton blend, inhibits the melting shrinkage of cotton blend and serves as supporting layer for cotton blend. The melted cotton blend served as fuel for continuing burning.



a) Untreated cotton blend mat   b) Cotton blend mat treated with 1.0% reactive nanosilicone softener

Fig. 6: SEM micrographs of the melt surface of cotton blend mat after the flammability test

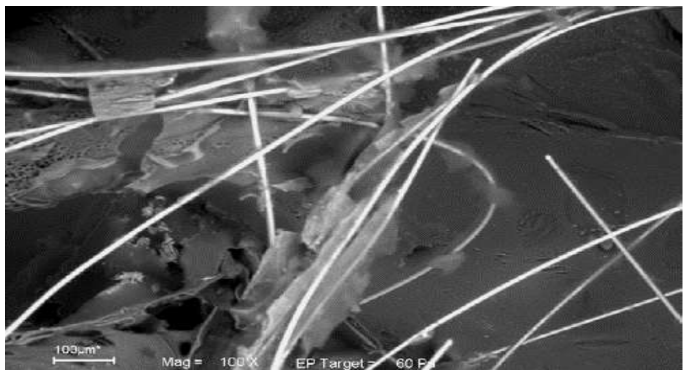
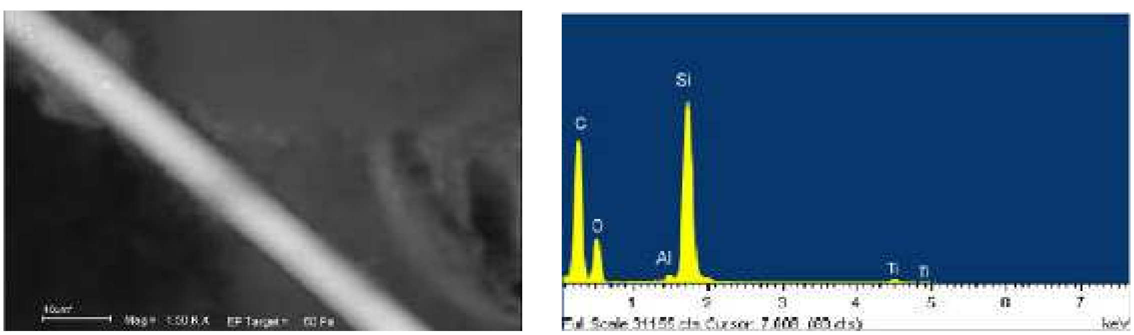
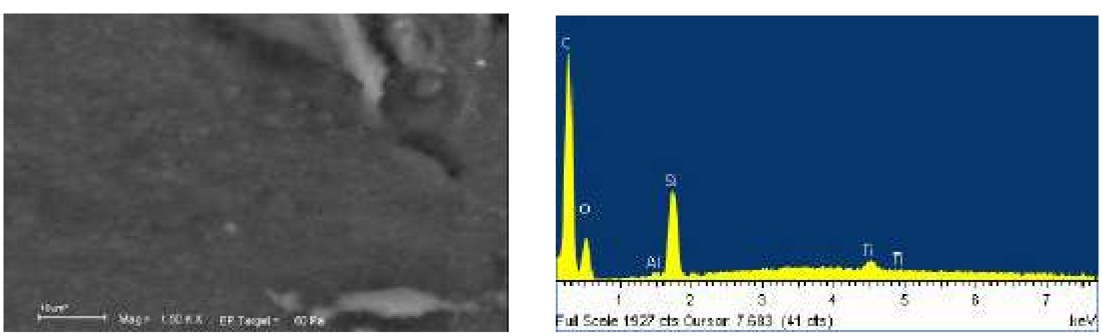


Fig. 7: SEM micrograph of char from cotton blend mat treated with 0.5% reactive nanosilicone softener



(a) SEM micrograph of the charred fiber   (b) The EDX spectrum of the charred fiber

Fig. 8: SEM micrograph and EDX spectrum of the charred fiber obtained from cotton blend mat treated with 0.5% reactive nanosilicone softener



(a) SEM picture of the char area   (b) EDX spectrum of the selected char area

Fig 9: SEM micrograph and EDX spectrum of the char area of cotton blend mat treated with 0.5% reactive nanosilicone softener

Table 8: T_{di} and T_{max} of untreated and reactive nanosilicone softener treated cotton blend mat

Sample	T_{di} (°C)	T_{max} (°C)
Control	170	440
0.1%	260	435
0.5%	385	440

T_{di}: Initial decomposing rate; T_{max}: Maximum decomposing rate.

of nanosilicon in the char did not have much of an effect compared to that on the cotton blend fiber before flammability test.

The EDX spectrum of the char of cotton blend mat treated with 0.5% reactive nanosilicone softener is shown in Figure 9. The surface of the char is sand like and the composition of the char area is shown in Table 7. The atomic percentage of carbon, oxygen and nanosilicon in the char area is 75.0, 21.9 and 2.7%, respectively. Compared with the element compositions of treated cotton blend mat before the flammability test, the char area contains higher carbon, lower oxygen and a little bit higher nanosilicon (Table 5). Most of the nanosilicone softener decomposes to produce nanosilicon dioxide fiber. Therefore, the composition

Thermogravimetric analysis (TGA): Figure 10 shows the TGA curves of untreated cotton blend mat and cotton blend mat treated with 0.1 and 0.5% reactive nanosilicone softener at a heating rate of 10°C/min in air. The characteristic decomposition temperatures derived from Figure 10 are presented in Table 8. The initial decomposition temperature (T_{di}) is the temperature at which the weight loss of the samples reaches 5 wt%. The T_{di} of untreated cotton blend mat is 170°C. The T_{di} of cotton blend mat treated with 0.1% and 0.5% reactive nanosilicone softener is 260 and 385°C, respectively, much higher than the untreated cotton blend mat. Therefore, the treatment of cotton blend mat with nanosilicone softener increases the stability of cotton blend and the stability increases with the nanosilicone concentration. The treated cotton blend mats decompose slower and show a smaller weight loss before the maximum decomposition rate temperature (T_{max}) than untreated cotton blend mat (Figure 10). Before T_{max}, the reactive nanosilicone softener self-condenses on the surface of cotton blend and inhibits the evaporation of cotton blend oligomer. Therefore, treated cotton blend mats have higher stability before T_{max}. The T_{max} of untreated and treated cotton blend mat does not show much difference and is at about 435°C. Therefore, the treatment of nanosilicone softener does not increase the thermal oxidative stability. However, the residues

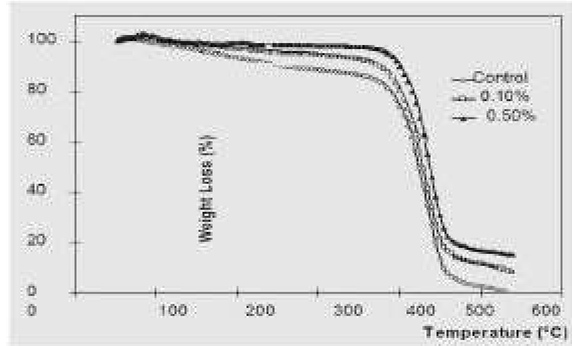


Fig. 10: TGA curves of cotton blend mat treated with 0.1 and 0.5% reactive nanosilicone softener

of thermal oxidative degradation of untreated cotton blend mat and cotton blend mats treated with 0.1% and 0.5% reactive nanosilicone softener are quite different (Figure 10). Treatment with nanosilicone softener increases the mass of residue significantly. The residue of untreated cotton blend mat is 0.45% at 530°C. The residues of cotton blend mat treated with 0.1% and 0.5% reactive nanosilicone softener are 8.2 and 15.1% at 540°C, respectively, much higher than that of the untreated cotton blend mat. The low levels of reactive nanosilicone softener do not account for the 7.8 and 14.7% increase in the char residue for cotton blend treated with 0.1% and 0.5% reactive nanosilicone softener, respectively. This result can be explained by the charring effect of nanosilicone softener on cotton blend in air at high temperature. Nanosilicone softener decomposes to produce inorganic nanosilicon dioxide as shown in SEM micrographs of the char. The inert nanosilicon dioxide protects the cotton blend underneath and facilitates the charring of cotton blend. Thus, the cotton blend mat treated with nanosilicone softener has higher residue.

CONCLUSIONS

The 45° flammability test established in this work combined with the Student t-test can be used as a semi-quantitative method to determine the flammability of cotton blend materials. The treatment with reactive nanosilicone softener significantly increases the flammability of cotton blend nonwoven and cotton blend mat materials. The treated samples show much higher weight loss and burning area than those that are untreated. One theory is that the mechanism for the increased flammability of nanosilicone treated cotton blend material is that the softener decomposes to produce inert inorganic nanosilicon dioxide fiber during the burning process, which serves as an inert support for the melted cotton blend. The molten cotton blend is the fuel supply for continued burning. Nanosilicone softener may also lower the surface energy inhibiting the melt shrinkage of cotton blend fiber when subjected to an ignition source. TGA shows that the treatment with nanosilicone softener increases the thermal stability of cotton blend in air before the maximum decomposition rate temperature and increases the residue of cotton blend.

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