

# Poly(styrene-*co*-butyl acrylate) Latex-Reinforced Polyester Nonwoven Fabric Composites: Thermal and Morphological Studies

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**ABSTRACT:** A series of thermoplastic composites were fabricated by impregnating the polyester nonwoven fabric in poly(styrene-*co*-butyl acrylate) latex having different monomer compositions of styrene and butyl acrylate viz., 100/0, 90/10, 80/20, 70/30, 60/40, and 50/50 weight by weight. Thermogravimetric analysis (TGA) of the composites was performed to establish the thermal stability and their mode of thermal degradation. From TGA thermograms, a slight improvement in thermal stability of the composites was noticed compared to polyester nonwoven fabric. Degradation kinetic parameters were obtained for

the composites using Broido and Coats–Redfern methods. The activation energy ( $E_a$ ) of the composites for the thermal degradation process lies in the range 7.1–261 and 60–264 kJ/mol for Broido and Coats–Redfern methods respectively. Morphology of the tensile-fractured composites was studied using scanning electron microscopic technique. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 114: 467–474, 2009

**Key words:** polyester nonwoven fabric; styrene/butyl acrylate; composites; TGA; kinetic parameter

## INTRODUCTION

Because the thermal and thermal-oxidative stability of the composites determine the upper service temperature and the environmental conditions under which the composite can be used, it is very essential to understand the thermal stability of the composites. Because thermal stability is related to both the initial degradation temperature and the degradation rate of a polymer, the determination of activation energy is very important.<sup>1,2</sup> Thermogravimetric analysis (TGA) is an established technique for determining the weight change of a sample as a function of temperature or time to investigate the thermal stability of polymeric systems. Knowledge on degradation and the mode of thermal decomposition is highly recommended in the process optimization. The threshold decomposition temperature gives an indication of the highest processing temperature that can be used, whereas the study of the kinetics of the different decomposition processes

may help in the identification of the degradation mechanisms.

Styrene (St)-butyl acrylate (BA) emulsion copolymers are important class of materials used in many industrial applications because of the possibility of obtaining a large number of polymer products through the variation of the copolymer composition and the polymerization process.<sup>3</sup> Because acrylate elastomers do not have double bonds or the degree of unsaturation is very low, the aging resistance and processing properties of St/BA copolymers are very good.<sup>4</sup>

The industrial importance of St/BA copolymers has been revealed by many research scientists.<sup>5–8</sup> The kinetics of St/BA copolymers has been dealt by many researchers.<sup>9–17</sup> Yang and Yang<sup>7</sup> studied the influence of the comonomer ratio, initiator, emulsifier, and temperature on the polymerization rate and molecular weight. Cruz-Rivera et al.<sup>3</sup> obtained higher molecular weight by batch emulsion polymerization than in semi-continuous emulsion polymerization. The copolymerization of St/BA for various applications like pressure-sensitive adhesive, coating, and for nonwoven fabrics is mainly carried by emulsion polymerization.<sup>18,19</sup> The latex obtained through this technique can be directly applied.

Nonwoven fabric as a material may find its use in the fabrication of light-weight composites. The

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utilization of light-weight composites in the automobile sector may provide a positive impact on fuel economy. The phenolic resin-bonded nonwoven fabric composites have been used as a material for supporting and covered parts in automobile industry.<sup>20</sup> The mechanical properties of nonwoven fabric are insufficient even to some nonstructural applications and hence, polymeric binders/resins<sup>21–27</sup> are incorporated to enhance the same. In the previous communication,<sup>28</sup> we have studied the effect of different weight ratio of St/BA latex on the mechanical performance of polyester nonwoven fabric impregnated poly(styrene-*co*-butyl acrylate) composites. It was found that the incorporation of either PS alone or poly(styrene-*co*-butyl acrylate) latex into polyester nonwoven fibrous network enhanced the mechanical performance of the composites. In the present study, the fabricated composites were characterized by TGA and scanning electron microscopy (SEM) to understand the thermal stability and morphology.

## EXPERIMENTAL

### Materials

Styrene (St) and butyl acrylate (BA) monomers of pure reagent grade were obtained from Aldrich chemical and used after removing inhibitor. The inhibitor was removed by washing with 10% aqueous NaOH solution. Linear alkyl benzene sulfonate, potassium persulphate, sodium hydroxide, and tapioca starch were obtained from S. d. fine chemicals.

Randomly oriented, polyester-based, needle-punched nonwoven (150 g/m<sup>2</sup>) fabric having density of 0.2 g/cc, air permeability of 27 m<sup>3</sup>/m<sup>2</sup>/min at 20 WG and burst strength of 8 kg/cm<sup>2</sup> was procured from the local supplier. The fiber used to make the fabric has 3 denier × 64 mm length.

### Fabrication of composites

Poly(styrene-*co*-butyl acrylate) having different monomer compositions of St and BA were synthesized as per the procedure described elsewhere.<sup>28</sup> Polyester nonwoven fabric having a weight of 150 g/m<sup>2</sup> was impregnated into a bath containing poly(styrene-*co*-butyl acrylate) latex. The dipped fabric was squeezed in a two-roll squeezer to remove the excess latex. The optimized pickup ratio of polymer latex to nonwoven fabric was to 3.2 : 1. After the desired latex pickup was adjusted, the impregnated fabric was dried in a hot air oven at 150°C for 20 min to evaporate the residual water and to facilitate the film formation. The temperature distribution during drying was monitored and controlled using a thermometer with a PT-100 sensor. The dried com-

posites were initially soft and at this stage they were calendared to improve the surface finish, then the composites were cooled to ambient temperature to get the good stiffness. A series of composites were fabricated with poly(styrene-*co*-butyl acrylate) latex having different monomer compositions of St/BA viz., 100/0, 90/10, 80/20, 70/30, 60/40, and 50/50 weight by weight.

### Techniques

The TGA thermograms were recorded using Waters TA thermal analyzer 2950 TGA at a heating rate of 20°C/min in nitrogen media. The TGA profiles were recorded in the temperature range 30–600°C. The weight of the sample used was about 6–8 mg in all the cases.

The integral procedural decomposition temperature (IPDT) is defined as a means of summing up the whole shape of the normalized data curve. IPDT is an index of thermal stability determined from the thermogram area using the method reported in the literature.<sup>29</sup> The oxidation index (OI) was calculated<sup>30</sup> based on the weight of carbonaceous char (CR) as related by the empirical equation

$$OI = \frac{17.5 \times 0.4 CR}{100} \quad (1)$$

The thermal degradation kinetic parameters for polyester nonwoven fabric and their composites with poly(styrene-*co*-butyl acrylate) latex were measured from the TGA thermograms using Broido<sup>31</sup> and Coats–Redfern<sup>32</sup> methods, which provide overall kinetic data. For the sake of calculations and to know the nature of the decomposition, the complete thermogram was divided into two distinct sections. The activation energy for the thermal degradation process was evaluated using Broido equation

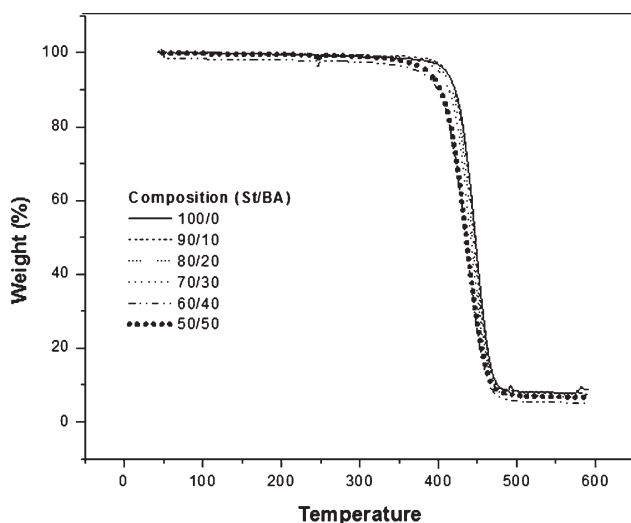
$$\ln[-\ln(1 - \alpha)] = -E_a/RT + \text{const} \quad (2)$$

where  $\alpha = (w_0 - w_t)/(w_0 - w_\infty)$  in which  $w_0, w_t$ , and  $w_\infty$  are the weights of the sample before degradation, at time  $t$  and after total conversion, respectively.  $R$  is gas constant,  $T$  the temperature (in K), and  $E_a$  the energy of activation can be obtained from the plot of  $\ln[-\ln(1 - \alpha)]$  verses  $1/T$ .

Coats–Redfern equation is as follows

$$\ln[-\ln(1 - \alpha)/T^2] = \log ZR/\beta E_a(1 - 2RT/E_a) - E_a/RT \quad (3)$$

where  $Z$  is the frequency factor,  $\beta$  the heating rate, and  $E_a$  the activation energy. A plot of  $\ln[-\ln(1 - \alpha)/T^2]$  verses  $1/T$  gives the slope for the evaluation of activation energy most appropriately.



**Figure 1** TGA thermograms of polyester nonwoven fabric-poly(styrene-*co*-butyl acrylate) latex composites.

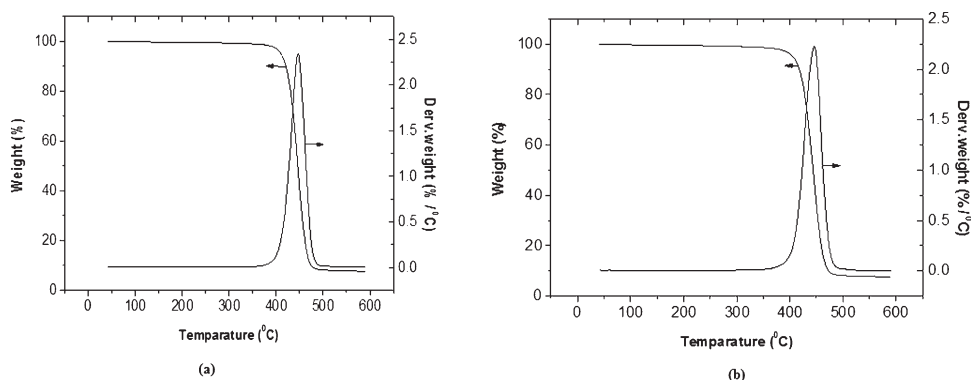
Scanning electron microscopy (SEM) studies were conducted with Jeol JSM-840A, Japan, to study the morphology of the tensile-fractured composites.

## RESULTS AND DISCUSSION

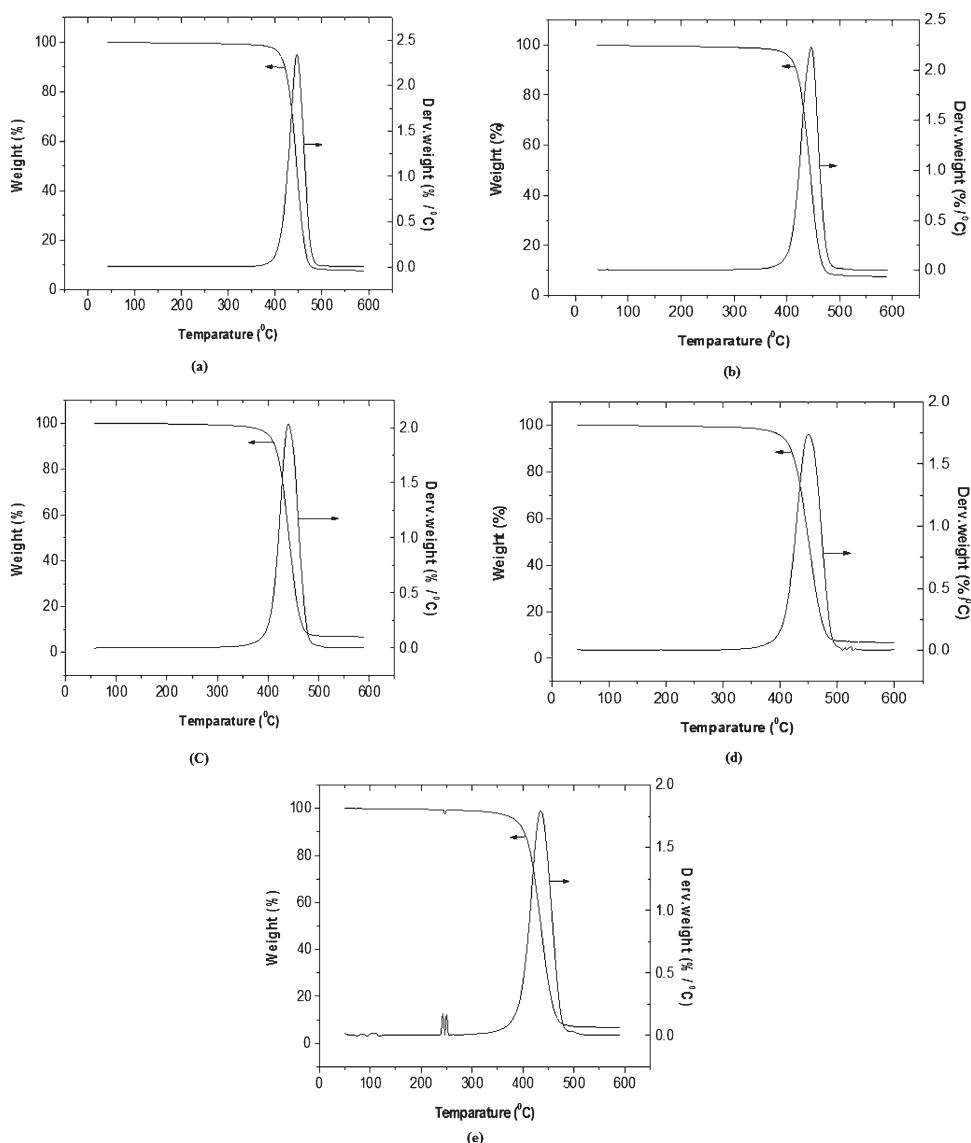
TGA curves and their typical derivative thermograms of polyester nonwoven fabric and their composites with poly(styrene-*co*-butyl acrylate) having different weight ratio of St/BA are given in Figures 1–3. The initial degradation of all the samples occurred above 380°C except the composites with 50/50 weight ratio of St/BA. This temperature range is significantly higher than the composite fabrication temperature. The initial degradation may be due to the evaporation of low-molecular weight impurities and thermal decomposition of organic emulsifier present in the PS and poly(styrene-*co*-butyl acrylate) latex.<sup>33</sup> From the thermograms, it is found that the

polyester nonwoven fabric is stable up to 400°C and the observed maximum degradation was at around 550°C. Schwenker et al.<sup>34</sup> made a similar observation on polyester fiber (Dacron) textile and other high polymeric materials using differential thermal analysis technique. Rana et al.<sup>35</sup> reported that the degradation of poly(styrene-*co*-butyl acrylate) starts at 310–320°C. Jang and Wilkie<sup>36</sup> noticed the degradation of neat polystyrene around 300–330°C. In the present study, the composites of polyester nonwoven fabric with PS and poly(styrene-*co*-butyl acrylate) latex found to start thermal degradation at 350–389°C. A slight increase in the degradation temperature of the composites compared with neat PS and poly(styrene-*co*-butyl acrylate) could be due to the presence of polyester nonwoven fabric. The polyester nonwoven fabric and their composites with poly(styrene-*co*-butyl acrylate) latex exhibited one-step degradation behavior. The one-step degradation behavior of neat PS and poly(styrene-*co*-butyl acrylate) were reported in the literature.<sup>33,37</sup> As polyester nonwoven fabric, PS and poly(styrene-*co*-butyl acrylate) exhibited one-step degradation, the TGA thermograms of all the composites resembled the same pattern.

Soundarajan et al.<sup>38</sup> showed that the thermal decomposition of copolymers of St/BA starts at much lower temperature compared with pure PS due to the free movement of copolymer chains caused by BA. In the current study, the incorporation of polyester nonwoven fabric increased the thermal stability of poly(styrene-*co*-butyl acrylate) composites. The observed thermal stability was found to decline with increasing the BA content in the copolymer composition. The observed reduction is drastic for the composites fabricated using 60/40 and 50/50 (St/BA) copolymer latex. This reduction in thermal stability of the composites with increasing the BA could be due to the reduction in glass transition temperature of copolymer, which allows the



**Figure 2** Typical TGA and its deravative thermograms of (a) polyester nonwoven fabric and poly(styrene-*co*-butyl acrylate)-polyester nonwoven fabric composites with (b) 100/0, weight ratio of St/BA.



**Figure 3** Typical TGA and its derivative thermograms of poly(styrene-*co*-butyl acrylate) – polyester nonwoven fabric composites with (a) 90/10, (b) 80/20, (c) 70/30, (d) 60/40, and (e) 50/50 weight ratio of St/BA.

free movement of copolymer chains. A similar observation was made by Zhang et al.<sup>39</sup> with respect to graphite oxide filled poly(styrene-*co*-butyl acrylate). The authors also explained that the addition of graphite oxide into copolymer increases the thermal stability by restricting the movement of copolymer chains.

The thermograms obtained during the TGA scans were analyzed to obtain the percentage weight loss as a function of temperature.  $T_0$  (temperature of onset decomposition),  $T_{10}$  (temperature for 10% weight loss), and  $T_{max}$  (temperature for maximum weight loss) were the main criteria to indicate the thermal stability of the composites. Higher values of  $T_0$ ,  $T_{10}$ , and  $T_{max}$  indicate higher thermal stability of polymer. The relative thermal stability of composites was evaluated by comparing the decomposition tem-

peratures at various percentage weight loss and IPDT values (Table I). IPDT values represent the overall nature of the thermogram over the entire range of the TGA curves. IPDT values of the composites are slightly higher than that of polyester nonwoven fabric. The calculated OI values of the composites are lower compared with neat polyester nonwoven fabric due to the low char content, suggesting that the composite. The increased char content is expected to provide an increased thermal stability due to the fact that the char has the ability and acts as barrier to prevent the oxygen diffusion into the combustion zone.<sup>40</sup>

The temperature range of decomposition, the percentage weight loss, and the percentage ash content obtained from TGA thermograms are given in Table II for polyester nonwoven fabric and their

**TABLE I**  
Data Obtained Form TGA of Polyester Nonwoven Fabric Composites With Different Composition of Styrene and Butyl Acrylate

Composition (St/BA)	Temperature at different weight loss + 2 (°C) <sup>a</sup>					IPDT	OI + 0.03
	T <sub>0</sub>	T <sub>10</sub>	T <sub>20</sub>	T <sub>50</sub>	T <sub>max</sub>		
PET nonwoven fabric	206	415	428	445	550	610	1.19
100/0	244	418	437	449	595	682	0.56
90/10	244	418	437	448	593	679	0.42
80/20	241	416	435	448	592	678	0.53
70/30	239	412	433	446	592	678	0.53
60/40	210	401	419	432	574	659	0.56
50/50	201	392	409	424	561	642	0.46

<sup>a</sup> T<sub>0</sub>, temperature of onset decomposition; T<sub>10</sub>, temperature of 10% weight loss; T<sub>20</sub>, temperature of 20% weight loss; T<sub>50</sub>, temperature of 50% weight loss; T<sub>max</sub>, temperature at which decomposition is completed.

composites with poly(styrene-co-butyl acrylate) containing different compositions of St/BA. From Table II, it can be observed that the thermal stability of the poly(styrene-co-butyl acrylate)-polyester nonwoven fabric composites have been reduced with increasing the BA content in the composition as evidenced by the decreased  $T_i$ ,  $T_d$ , and  $T_c$  values. The ash content of poly(styrene-co-butyl acrylate)-polyester nonwoven composite lie in the range 6.0%–8.0%. The obtained ash content is high in case of polyester nonwoven fabric (17%) compared with their composites with poly(styrene-co-butyl acrylate). This may be due to the fact that, during pyrolysis, the intermediate product (carbonized residue) formed by thermo-oxidative process leads to the formation of hydro peroxides, which acts as radical scavengers<sup>30</sup> for poly(styrene-co-butyl acrylate).

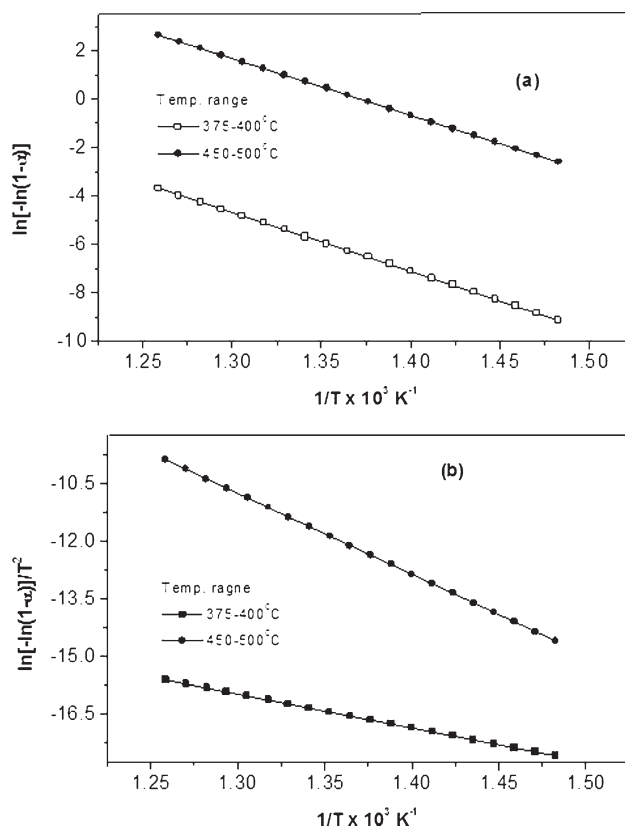
Kinetic parameters were evaluated from the TGA curves using the plots of Broido (BR) and Coats-Redfern (CR) methods. To know the activation energy of the composites for both initial and final

degradation steps, the complete thermogram was divided into two distinct sections. The BR and CR plots for polyester nonwoven fabric are given in Figure 4(a,b). The plots of BR and CR for polyester nonwoven fabric impregnated poly(styrene-co-butyl acrylate) composites are shown in Figures 5(a,b) and 6(a,b), respectively. Regression analysis was performed for all the plots and the mechanism of  $R^2$  close to unity was chosen. The regression analysis gives the results of slopes, constants, and  $R^2$  values corresponding to thermal degradation for the

**TABLE II**  
Data Obtained Form TGA of Polyester Nonwoven Fabric Composites With Different Composition of Styrene and Butyl Acrylate

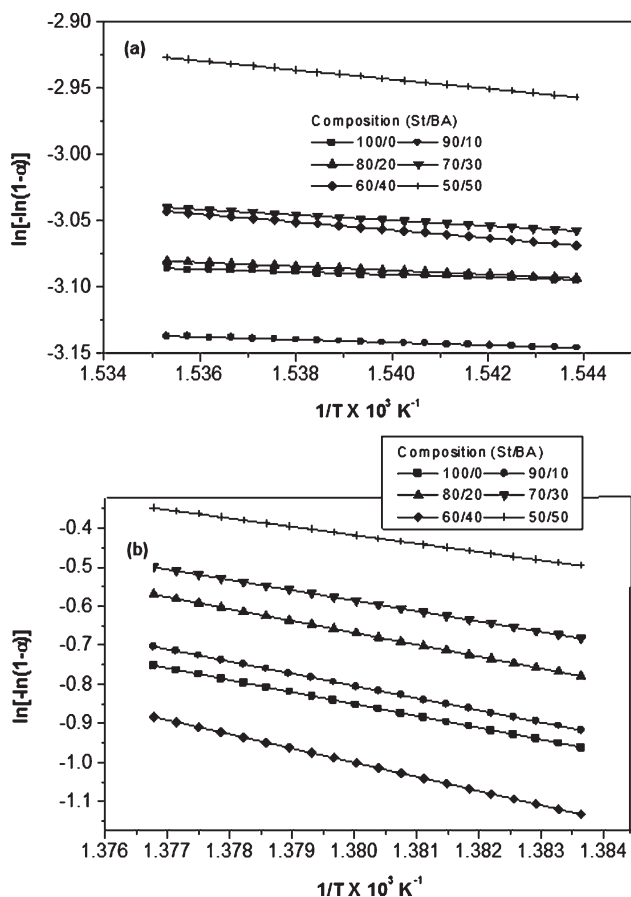
Composition (St/BA)	Transition temperature range (°C) <sup>a</sup>			Weight loss (%)	Ash (%)
	T <sub>i</sub>	T <sub>d</sub>	T <sub>c</sub>		
PET nonwoven fabric	393	439	552	83.0	17.0
100/0	389	445	594	92.0	8.0
90/10	394	445	593	94.0	6.0
80/20	395	445	592	93.0	7.0
70/30	395	448	592	93.0	7.0
60/40	396	447	592	92.0	8.0
50/50	350	433	561	93.5	6.5

<sup>a</sup> T<sub>i</sub>, temperature at which decomposition starts; T<sub>d</sub>, temperature at which decomposition rate is maximum; T<sub>c</sub>, temperature at which decomposition is completed.



**Figure 4** Plots of (a) Broido and (b) Coats-Redfern at different temperatures for the determination of activation energies for polyester nonwoven fabric.



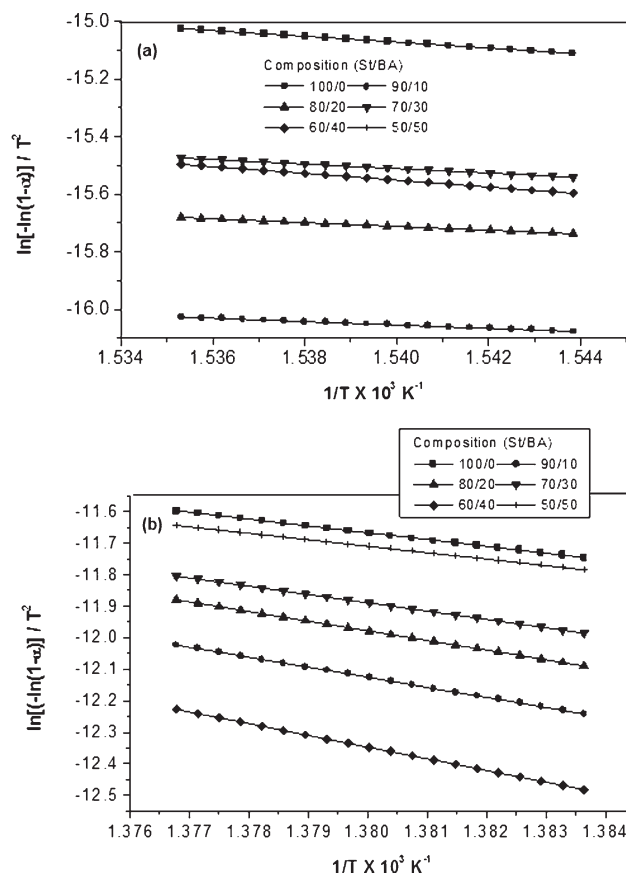


**Figure 5** Typical Broido plots for the determination of activation energies for temperature range (a) 375–400°C and (b) 450–500°C.

selected temperature range. The  $R^2$  values and the calculated activation energy ( $E_a$ ) for each selected thermal degradation process and for BR and CR methods are tabulated in Table III. The lowest  $E_a$  values were observed for the initial thermal degradation process compared with final degradation for both the methods. This is due to the lower energy required to remove loosely bound water and the other low-molecular weight impurities present in poly(styrene-co-butyl acrylate). Higher  $E_a$  values were observed for second step, because higher energy is required for bond scission and unzipping of polymer chains. The  $E_a$  values obtained by CR method are higher than those obtained by BR method. The observed difference in the  $E_a$  values for the applied two methods may be attributed to the different mathematical approaches used to calculate the kinetic parameters.<sup>41</sup> It was also observed that with increase in the BA, the composites showed a reduction in  $E_a$  values.

### Morphological behavior

The nonwoven fabrics bonded with polymers of different nature will have different types of bond struc-

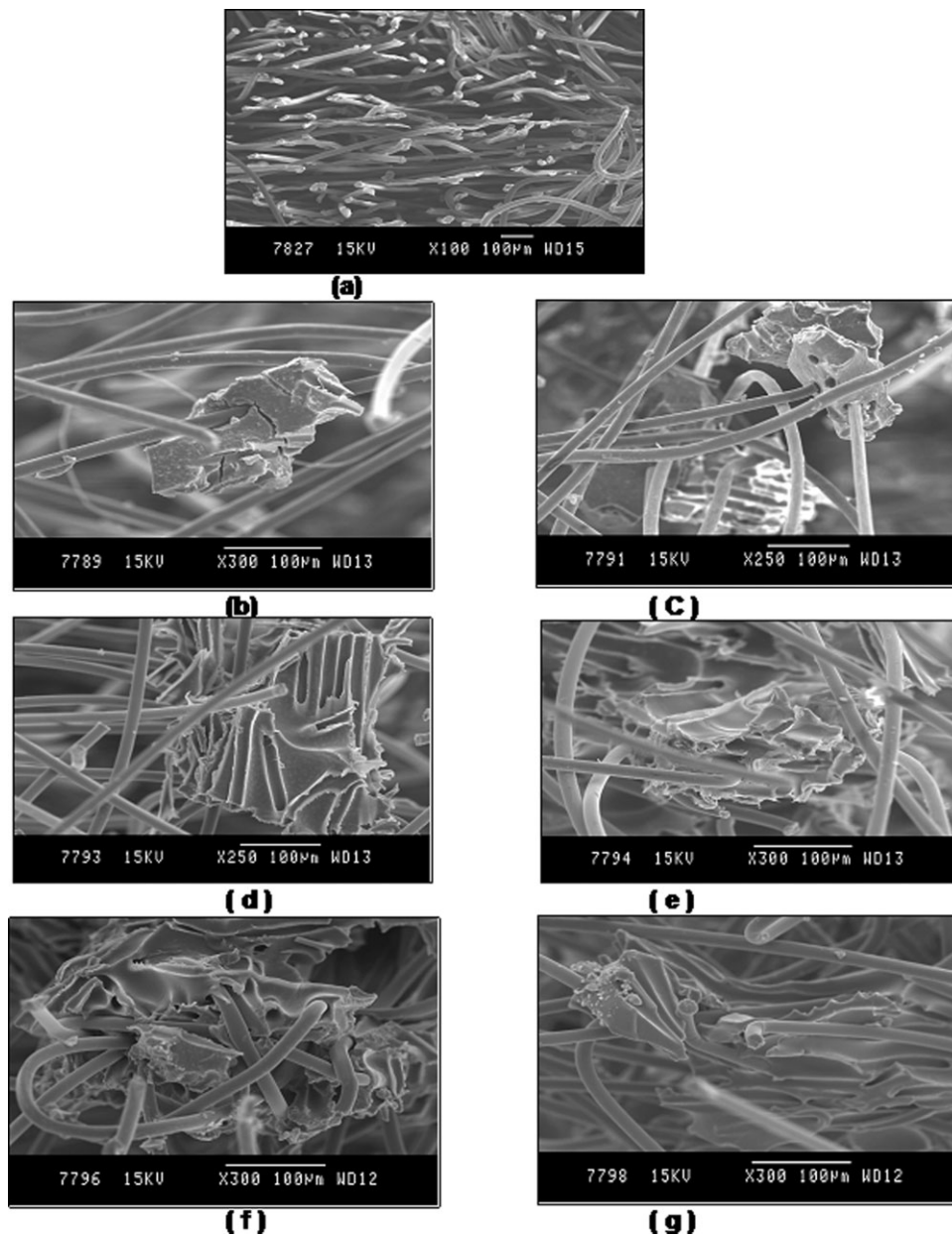


**Figure 6** Typical Coats-Redfern plots for the determination of activation energies for temperature range (a) 375–400°C and (b) 450–500°C.

tures. Formation of these structures depends on the amount of matrix incorporated. The resulting structure affects the composite performance. The

**TABLE III**  
Activation Energies Calculated by Broido (BR) and Coats-Redfern (CR) Methods for Poly(styrene-co-butyl acrylate) – Polyester Nonwoven Fabric Composites

Composition (St/BA)	Temperature range (°C)	Activation energy (kJ/mol)			
		BR	$R^2$	CR	$R^2$
Polyester nonwoven fabric	375–400	6.9	0.990	56	0.997
	450–500	192	0.993	195	0.993
100/0	375–400	8.6	0.989	84	0.987
	450–500	261	0.989	264	0.988
90/10	375–400	8.2	0.997	81	0.999
	450–500	257	0.996	260	0.995
80/20	375–400	8.2	0.995	75	0.994
	450–500	253	0.999	257	0.999
70/30	375–400	8.2	0.992	72	0.996
	450–500	222	0.999	255	0.999
60/40	375–400	7.5	0.992	66	0.995
	450–500	203	0.999	219	0.999
50/50	375–400	7.1	0.992	60	0.996
	450–500	180	0.992	171	0.997



**Figure 7** Scanning electron micrographs of (a) polyester nonwoven fabric and polyester nonwoven fabric composites with different weight ratio of St/BA in poly(styrene-*co*-butyl acrylate) viz., (b) 100/0, (c) 90/10, (d) 80/20, (e) 70/30, (f) 60/40, and (g) 50/50.

scanning electron micrograph (SEM) of tensile-fractured polyester nonwoven fabric and their composites with poly(styrene-*co*-butyl acrylate) latex having different weight ratio of St/BA are shown in Figures 7(a-g). The SEM images of polyester nonwoven fabric [Fig. 7 (a)] showed the fibrillar nature of the nonwoven fabric. As can be seen from the figure that the fibers are curled or deformed indicating the poor stress transfer characteristics. All the micrographs revealed the presence of matrix adhesion between the fibers. The brittle nature of the PS adhered on the polyester fiber can be seen in Figure

7(b). The use of St/BA copolymer latex into polyester nonwoven fibrous structures resulted smooth surface and no cracking or brittleness of the matrix was noticed. The presence of discrete lumps in the micrographs of Figure 7(b,c) revealed that these composites have agglomerated bonding structures. In agglomerated bonding structures, the entire matrix does not contribute to the bonding of fibers due to some matrix particles adhering only to a single fiber. The micrographs of Figures 7(d) to (f) showed the formation of a matrix film or sail-like enclosure of the fibers.

## CONCLUSIONS

From this study, it can be concluded that composites of polyester nonwoven fabric with poly(styrene-*co*-butyl acrylate) latex showed a little improvement in the thermal stability compared with polyester nonwoven fabric. The composites showed a reduction in thermal stability with increasing BA content. The observed reduction in thermal stability was high for the composites fabricated with 60/40 and 50/50 weight ratio systems. Lowest activation energy was observed at the initial degradation step compared with final degradation step. This is because of less energy is utilized to remove the loosely bound water and low-molecular weight materials present in the latex. The morphology of tensile-fractured specimens revealed that the incorporation of either PS or poly(styrene-*co*-butyl acrylate) latex into fibrous nonwovens structures may enhance the stress transfer characteristics. The improved stress transfer ability of the composites may be due to the better bonding of nonwoven fibrous structures by the polymer latex.

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