



RAMAN SPECTROSCOPY AND THERMAL STUDIES OF RUBBER-NANOCOMPOSITES SYNTHESIZED FROM STYRENE-BUTADIENE RUBBER AND NANO ALUMINUM OXIDE

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Abstract

Styrene-butadiene elastic with and without nano aluminum oxide, were set up by coagulating the blend of elastic latex and distinctive measures of all around scattered nano aluminum suspension. An endeavor to anticipate mix creations was made utilizing Raman spectroscopy. It was discovered that the force of each Raman trademark crest was firmly subject to the mix organization, yet there was no critical development with the nearness of nano alumina. TGA results uncovered an enhancement in warm strength of SBR elastic with expanding nano aluminum oxide substance because of the weakening impact. This demonstrates a physical mix development, which concurred well without any movements in Raman pinnacles of the mixes in comparison with those of the individual elastic.

Keywords: Rubber, Raman Spectroscopy, SBR, TGA, Latex.

1 Introduction

Styrene-butadiene elastic (SBR) is mixed with characteristic elastic (NR) so as to accomplish parity of their properties, for example, warm form-up, scraped area and maturing obstruction [1–3]. Likewise, expansion of filler into the lattices pursued by co-coagulating the blend prompts more noteworthy scattering of filler than by ordinary dry mixing. Be that as it may, loss of either elastic or filler may happen if the co-coagulating system isn't proper. Along these lines, it is important to affirm the measures of elastic and filler in the premix or masterbatch before further intensifying by regular blending

[4]. Thermogravimetric examination (TGA) is one of the helpful systems to quantify the warm security and to separate the segments of the mixes [5–7]. Sircar and Lamond [5] could distinguish the synthesis of NR/butadiene elastic (BR) mixes by utilizing the proportion of pinnacle stature of subordinate TGA results for perfect NR and butadiene elastic at 3650C and 4650C, separately. Among different procedures, vibrational spectroscopy, for example, Fourier change infrared (FTIR) and Raman spectroscopy, have been broadly used to describe concoction structure and mix pieces of elastic mixes [8–10]. Ghebremeskel and Shield [8] utilized FTIR to decide the creation of SBR/nitrile elastic (NBR) mixes by plotting the absorbance at 1602 cm⁻¹ for SBR and 2237 cm⁻¹ for NBR as an element of SBR content in the mixes. Appel and collaborators [9] likewise connected Raman spectroscopy to screen the mix sythesis of BR/brominated isobutylene-co-pa-methylstyrene (BIMS) mixes. They found that the relative force proportion (714 cm⁻¹ and 1648 cm⁻¹ for BIMS and BR, individually) is corresponding to the focus proportion of these two parts. Moreover, this relationship can be utilized to analyze the stage isolation by Raman mapping method.

The motivation behind this work was to apply Raman spectroscopy and warm methods, including TGA for investigating mix creation of SBR mix, with and without nano aluminum oxide, arranged from latex. The reasonableness for developing alignment bends from every portrayal technique was examined. Thermal stability of the mixes was likewise assessed by utilizing TGA. What's more, the real sum and

level of nano aluminum oxide scattering in the mixes were inspected by utilizing TGA respectively.

2 Experimental

2.1 Materials

Nanocomposites were purchased from Central Scientific Company Nagpur, India. 2, 2'-dithiobis, tetramethyl thiuram disulphide, stearic acid and N, N'- Diphenyl P- phenylene diamine were purchased from Central Scientific Company Nagpur, India. Sulphur and zinc oxide (Dept of chemistry Kamla Nehru Mahavidyalaya, Nagpur, India). Styrene-butadiene rubber latex purchased from Shree Radha Polymer Company, Nagpur, India.

2.2 Preparation and Compounding of SBR-nanocomposite

Nano aluminum oxide was dispersed in toluene with vigorous stirring and nano aluminum oxide suspension was obtained at room temperature. Then SBR latex was added into the nano aluminum oxide suspension and stirred up to uniform mixing of SBR into the nano aluminum oxide. The mixture was coagulated at room temperature. Then washed with water many times and then dried at 70°C for 12h. Then SBR-nanocompound was formed. SBR-nanocompound was mixed with varied ingredients shown within the (Table 1). The SBR-nanocompounds then were vulcanized at 150°C. The mixture of SBR-nanocompound was directly used on a two-roll mill and intermixing for 15 min., then adding all necessary ingredients that are listed in (Table 1) and intermixing for 10 min.

Table 1 Formulation of rubber compound

Ingredients	Phr			
	1	2	3	4
SBR	100	100	100	100
Aluminum oxide	0	2	10	12
Stearic acid	2	2	2	2
Zinc oxide	5	5	5	5
2, 2'- dithiobis	0.5	0.5	0.5	0.5
Tetramethyl thiuram disulfide (TMTD)	0.2	0.2	0.2	0.2
N, N'- Diphenyl P- phenylene diamine	1	1	1	1
Sulphur	2	2	2	2

3 Characterizations

The Raman dispersing tests were performed by utilizing a Jobin-Yvon spectrometer. So as to limit the fluorescence impact, SBR, SBR with filler were lit up with an argon-krypton particle laser (Coherent model Innova 70C) working at the 647.1 nm (red line). The laser bar was engaged by a $\times 100$ amplification targets of a confocal magnifying

lens (Olympus BX40) (from National Chemical Laboratory Pune, India). Every range was gathered in the frequency 400-3500 cm^{-1} more than 60 s and with 10 collections to keep away from electronic pinnacles and normal foundation. Thermogravimetric analysis of the vulcanizates was carried out by a Perkin Elmer Thermal Analyser. The sample was placed on pan and heated in temperature range of RT to

700°C under air and heating rate was 10°C/min (from sophisticated Analytical Instruments Facility, SAIF, Cochin).

4 Results and discussion

4.1 Raman spectroscopy

The spectra of SBR and SBR-nano aluminum oxide composites are analyzed in (Fig.1) and band assignments are done dependent on correlation with writing spectra. This work gives a full attribution for which Raman investigation of polystyrene and butadiene rubber is a great direction. Both symmetric and asymmetric $-\text{CH}_2$ and $-\text{CH}_3$ extending vibrations normally explain up in the 2800–3000 cm^{-1} region. Obviously, $\text{C}=\text{C}$ extending vibrations of SBR are seen at

frequency 1666 and 1668, respectively. Also, there are a few covered signs of SBR and SBR-nano aluminum oxide composites which can't be utilized to recognize their commitments, in this way just the trademark crests permitting separation will be centered around. Raman spectra of unfilled SBR tests at different mix proportions are revealed in (Fig.1). The outcomes demonstrate that power of Raman crests relies upon the blend composition. As can be seen, the intensities of trademark signals at frequency 1002, 1302, 1602 and 3058 will in general increment with expanding nano aluminum oxide content in the SBR rubber, though the intensities of the detached trademark groups of SBR at frequency 1371, 1128 and 496 diminish.

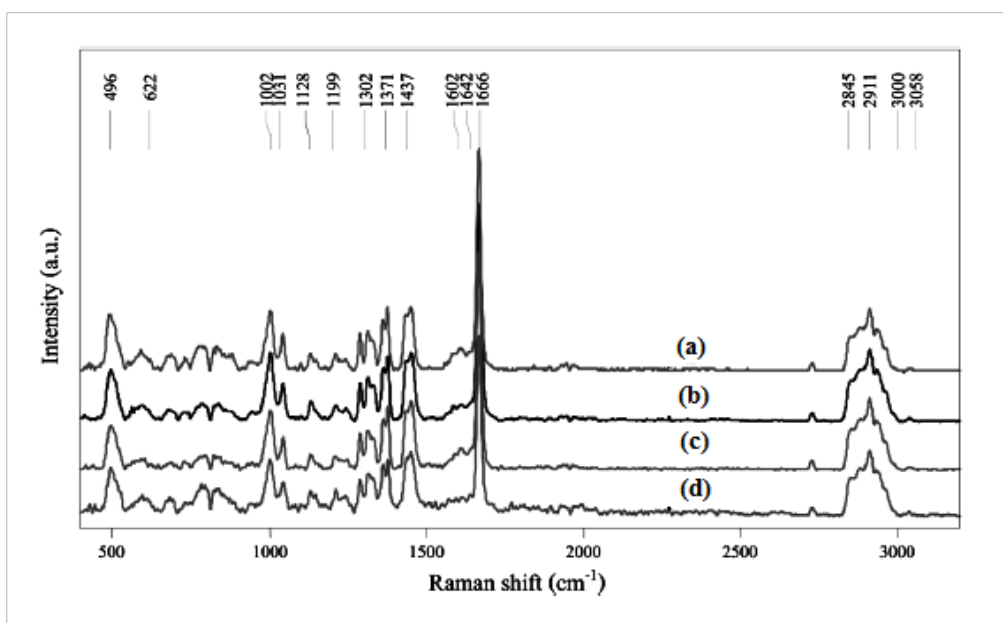


Fig. 1 Raman Spectra of SBR-Nano Composite (a) Unfilled Composites (b) 2 phr Aluminum Oxide in SBR (c) 10 phr Aluminum Oxide in SBR (d) 12 phr Aluminum Oxide in SBR

The crest at frequency 1666 isn't subject to SBR proportion and is utilized as an inside standard. From this relationship, it is conceivable to affirm composition in the blends by plotting the intensity ratio of characteristic signals of each rubber versus blend ratio. It could be noted here that the trademark signals at frequency 1371 and 1302 of $-\text{CH}_3$ asymmetric deformation and $=\text{C}-\text{H}$ in plane deformation for SBR, respectively, were decided for building an adjustment bend. This is on the grounds that these picked pinnacles have the best goals and the trademark crest at frequency 1371 isn't overlapped. Raman trademark signals of SBR

are not altogether changed after expansion of nano aluminum oxide. This perception is most likely because of the combination of low quantum proficiency in this frequency range (647.1 nm) and the weak Raman force of nano aluminum oxide particles which have an amorphous character. The typical bands of nano aluminum oxide are somewhat poor when the example is energized at 647.1 nm. In addition, these signs are not enhanced by expanding the number of scans and accumulation time. When utilizing excitation wavelength at 514.5 nm, the nano aluminum oxide sample demonstrates a Raman range having noticeable signals at

frequency 456, 796 and 994 relating to Al–O–Al and Al–OH extending vibration. Unfortunately, pinnacles of SBR allocated to $=CC_2$ rocking and $-CH_2$ stretching is situated in the similar frequency region at 496 and 1000, respectively [10]. In this way, it is hard to recognize the Raman signs of nano aluminum oxide from those of SBR.

4.2 Thermogravimetric Analysis

Thermogravimetric examination was performed to assess the thermal stability of the composites in (Fig. 2). The measure of nano aluminum oxide content in the elastic composites is resolved from lingering weight

level of a specific composite. Thermogravimetric bends are comparative for all the elastic composites (1-4). First weight reduction saw at the temperature extend $350^{\circ}C$ to $450^{\circ}C$ is because of debasement of elastic part. The following weight reduction show up at the temperature extend $500^{\circ}C$ to $560^{\circ}C$ is due to carbonaceous residue [11]. Temperature at most extreme weight reduction (T_{max}) is given from the peak of DTG curve. Every one of the composites demonstrates comparable thermal stability and T_{max} is found to be $400^{\circ}C$ and $460^{\circ}C$, respectively, for all composites.

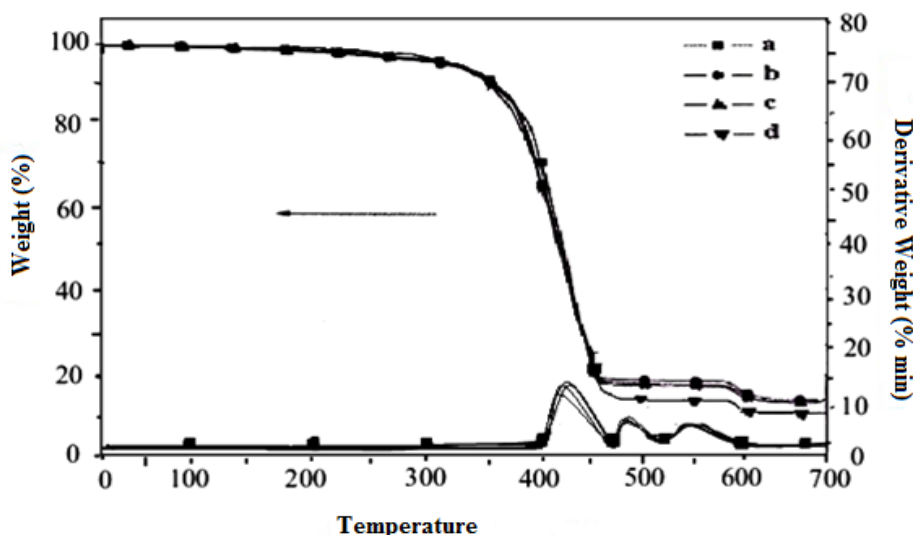


Fig. 2 Thermogravimetric Curves of SBR-Nanocomposite (a) Unfilled Composites (1) (b) 2 phr Aluminum Oxide in SBR (2) (c) 10 phr Aluminum Oxide in SBR (3) (d) 12 phr Aluminum Oxide in SBR (4)

5 Conclusions

Blend of SBR and nano aluminum oxide, with and without nano aluminum oxide filler, were set up by emulsion polymerization strategy. Raman results demonstrate the no new pinnacles nor were huge moves in the spectra, affirming no substance moieties produced in the mixing procedure. Likewise, the fuse of nano aluminum oxide into the mixes does not influence the trademark Raman pinnacle of SBR-nanocomposites blend. Thermogravimetric examination results uncovered an enhancement in thermal stability

of SBR rubber with increasing nano aluminum oxide contents due to the dilution effect.

Acknowledgments

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