

# Study of Environmental Properties of Rubber-nanocomposites Derived From Styrene-butadiene Rubber and Nano Carbon Black

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## Abstract

Elastic nanocomposite (SBR-Nano carbon black) was combined by the emulsion polymerization strategy. Elastic nanocomposite syntheses have been resolved based on their micro investigation. FTIR and Raman spectra were concentrated to illustrate the structure. The surface morphology of the copolymer gum was inspected by checking scanning electron microscopy and it sets up the change state among crystalline and formless nature. Morphology is also inspected by using transmission electron microscopy. Ozone obstruction was concentrated to explain occasional perceptions of the surface of the examples were made for break commencement. Tests were uncovered for a longer time. Flame obstruction was concentrated on measures the simplicity of termination of a flame and four appraisals are conceivable, contingent on the consuming time and the nearness of flaming drips. SBR-carbon black nanocomposites at 2 phr, 6 phr, 10 phr, and 12 phr wt% nano carbon black stacking indicates fairly enhanced combustibility property than its gum. The present investigation including the emulsion polymerization technique where the environmental performance of elastic nanocomposites is observed to be great.

**Keywords:** Styrene-Butadiene Rubber, Nanocomposite, Nano Carbon Black, FTIR, Raman, Flame, Copolymer.

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## INTRODUCTION

As of late, extraordinary considerations have been paid to nanoparticles because of its uncommon highlights, for example, surface impacts, little size impacts, limit reactions and the naturally visible quantum impacts [1-3]. Polymer corruption includes the difference in at least one physical property, bringing about the loss of the reasonableness of the material for the planned application [4]. Be that as it may, the collection of nanoparticles inferable from the high surface vitality and surface extremity debilitates their extraordinary nature [5]. Trans-polyoctylene elastic (TOR) has been presented as a compatibilizer for contrary elastic mixes containing polar rubbers, for example, acrylonitrile butadiene-elastic (NBR) and non-polar elastic styrene butadiene elastic (SBR), and as a handling helps for a to a great degree firm elastic compound, for example, profoundly filled elastic mixes for skim-covering of steel wires for tires [6]. Trans-polyoctylene elastic (TOR) is a low atomic weight polymer, produced using cyclo-octene by metathesis polymerization, it is an elite polymer that introduces a double character: amid handling, it has the capacity of a plasticizer, and after vulcanization, it acts as an elastic and has been known as a compatibilizer for contrary mixes. Styrene elastic (SBR) is a universally useful

manufactured elastic having high filler stacking limit; great flex obstruction, break inception opposition and scraped area safe, which make it helpful for a few designing and modern application. By the by, as other unsaturated rubbers, it is very defenseless against debasement because of quality of twofold securities in the primary chain [7]. When alluding to weathering presentation there is a kind of debasement that ought to be viewed as, for example, bright light corruption which is the elastic influenced by UV light and photodegradation and furthermore the ozone debasement. Despite the fact that ozone is available in the air at fixations ordinarily in the scope of 0–7 sections for every hundred million (pphm), it can extremely assault on non-safe rubbers. The association of elastic with ozone is best noted when the elastic is focused or extended being used. Progressions of splits create, after some time, which are opposite to the connected pressure. Further introduction of these split surfaces to ozone cause the break to wind up more extensive and more profound until the point that the elastic falls flat [8, 9].

In this work, we arranged SBR-nanocomposites copolymer with the utilization of nano carbon black as filler by emulsion polymerization technique. The impact of nano carbon dark on the

micro, spectral, morphology and environmental properties has been contemplated. The micro investigation, Fourier transform infrared (FTIR),

Raman spectroscopy, morphology, and environmental properties are completed for each of the five composites.

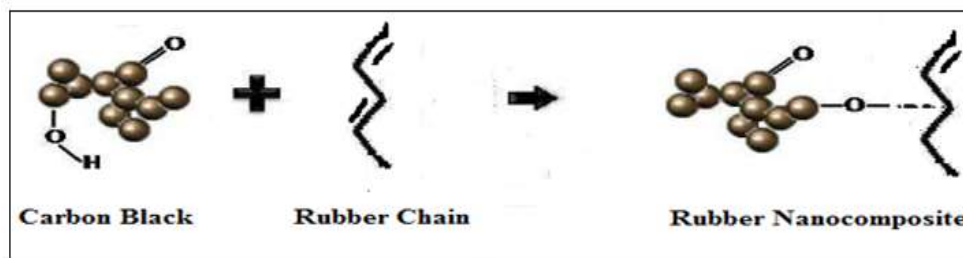


Fig-1: Schematic Diagram of SBR-Nano Carbon Black Nanocomposites

## EXPERIMENTAL SECTION

### Materials

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

### Preparation of SBR-Nanocomposite and Compounding of Rubber

First, nano carbon black was dispersed in toluene with vigorous stirring and nano carbon black

suspension was obtained at room temperature. Then SBR latex was added into the nano carbon black suspension and stirred up to uniform mixing of SBR into the nano carbon black. The mixture was coagulated at room temperature. Then washed with water several times and then dried at 70°C for 12h. Then SBR-nanocompound was formed.

SBR-nanocompound was mixed with various ingredients shown in Table-1. The SBR-nanocompound then was vulcanized at 150°C. The mixture of SBR-nanocompound was directly used on a two-roll mill and mixing for 15 min., then adding all necessary ingredients which are listed in Table 1 and mixing for 10 min. After that, the resultant compound was vulcanized at 150°C.

Table-1: Formulation of Rubber Compound

Ingredients	Phr				
	1	2	3	4	5
SBR	100	100	100	100	100
Carbon black	0	2	6	10	12
Stearic acid	2	2	2	2	2
Zinc oxide	5	5	5	5	5
2, 2'- dithiobis	0.5	0.5	0.5	0.5	0.5
Tetramethyl thiuram disulfide (TMTD)	0.2	0.2	0.2	0.2	0.2
N, N'- Diphenyl P- phenylene diamine	1	1	1	1	1
Sulphur	2	2	2	2	2

### Characterizations

#### Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR spectra of SBR-nanocomposites were determined by using Infra Red-Affinity spectrophotometer (from Dept of Chemistry Kamla Nehru Mahavidyalaya, Nagpur, India).

#### Raman Spectroscopy

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  $\text{cm}^{-1}$  more than 60 s and with 10 collections to keep away from electronic pinnacles and normal foundation.

#### Scanning Electron Microscopy (SEM)

Surface morphology of the nanocomposites was explored by utilizing Scanning electron magnifying instrument JEOL (JSM-6390, SAIF, Cochin) at speeding up the voltage of 3 kV. Because of the specific thin electron pillar, SEM micrographs have an expansive profundity of field yielding a trademark three-dimensional appearance helpful for understanding the surface structure of tests samples.

### Transmission Electron microscopy (TEM)

Morphology of SBR nanocomposites was straightforwardly seen by Transmission electron magnifying lens (Model JEM 2100) at a quickening voltage of 200 kV. The ultra-thin film of nanocomposite (under 100nm) gotten by ultra microtome cutting under cryogenic conditions was utilized for the morphological investigations (from IIT Bombay).

### Ozone Resistance

The chamber gave an air a controlled absorption of ozone and temperature. Ozone focus chose was 50 ppm, which is produced by UV quartz light. The test was completed according to ASTM D 1149-99 determinations at 38.50 C (from Shree Radha Polymer Rubber Industry Nagpur, India).

### Flame Resistance

Flame opposition of vulcanisates of SBR elastic nanocomposites was assessed according to UL 94 outline test for combustibility of plastic materials for parts in gadgets and machines (from Shree Radha Polymer Rubber Industry Nagpur, India). Strategy 94V, utilized for vertical consume test was connected. The test samples were (segments of 12 mm x 100 mm and thickness 2.0 mm cut from formed vulcanizates) held toward one side in the vertical position.

## RESULTS AND DISCUSSION

### Microanalysis Measurements

On the basis of microanalysis data given in Table-2. The composition of SBR-carbon black nanocomposites was suggested and it is found agreement with the calculated values.

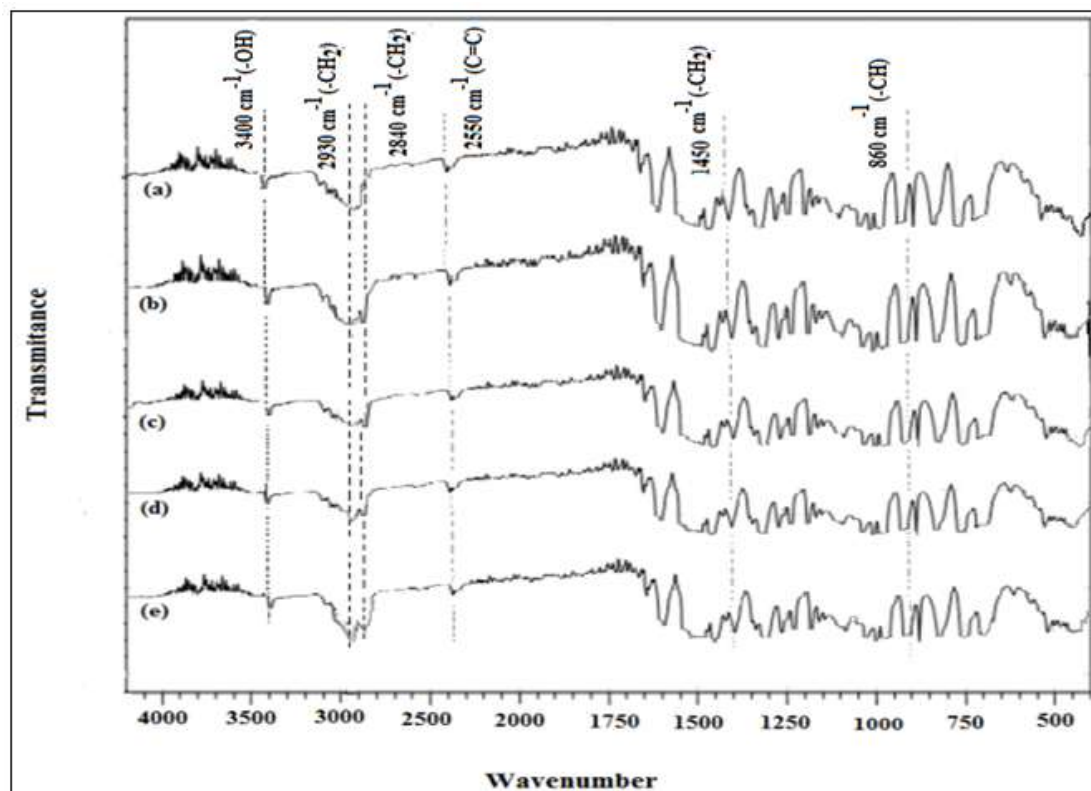
**Table-2: Microanalysis Data of Styrene-Butadiene Rubber-Carbon Black Composites**

Rubber samples	% of C observed (Cal.)	% of H observed (Cal.)	% of N observed (Cal.)	% of O observed (Cal.)	% of S observed (Cal.)	Empirical Formula	Empirical Weight
SBR	83.44 (81.44)	8.11 (8.05)	2.56 (2.31)	5.85 (5.08)	2.34 (1.39)	$C_{38}H_{44}N_1O_2S$	546.993
SBR-CB (2 phr)	85.12 (84.87)	8.16 (8.10)	2.10 (2.21)	5.40 (5.04)	1.07 (1.00)	$C_{42}H_{48}NO_2S$	592.657
SBR-CB (6 phr)	84.26 (83.20)	9.09 (8.99)	2.24 (2.20)	5.34 (5.01)	1.07 (1.02)	$C_{42}H_{54}N_1O_2S$	598.705
SBR-CB (10 phr)	80.40 (81.22)	9.65 (8.89)	2.23 (2.10)	5.66 (4.60)	2.99 (2.05)	$C_{42}H_{60}N_1O_1S_1$	627.008
SBR-CB (12 phr)	82.89 (80.67)	10.27 (9.12)	2.29 (2.12)	5.26 (4.20)	1.58 (1.02)	$C_{42}H_{62}N_1O_2S$	612.963

### FTIR Spectroscopy

FTIR (Fourier-transform infrared spectroscopy) spectra of various loadings dimensions of carbon black shows in Figure-2. In all these spectra, the expansive peak at frequency 3,400 is allocated to extending vibration of -OH group stretching vibrations alongside adsorbed water. The peaks at frequency 950 and 860 are because of C-H bending of the aromatic ring in carbon black. The crest at frequency 1100 and 470 are because of uneven and symmetric extending. The nearness of two sharp peaks at frequency 2,930 and 2,840 and one crest around 1,450 for all the spectra of SBR-filler furthermore of the retention groups relating to the vibration of the explicit functional group. These little pinnacles of around frequency range 2,930

and 2,840 are trademark groups of symmetric and antisymmetric extending vibrations of -CH<sub>2</sub> group respectively [10, 11]. At the frequency range 1,450 relating to bowing vibrations of -CH<sub>2</sub> gathering. Every one of these pinnacles is seen in spectra of SBR nanocomposites in Figure-2. This is additionally bolstered by the nearness of band at 1,080 cm<sup>-1</sup> which is trademark band of alkene gathering. The crest at 1,750 cm<sup>-1</sup> of carbon black when CO enactment which demonstrates the nearness of C=O practical gatherings described by aldehydes, ketones, carboxylic acids, and esters. The nearness of band at 2,550 cm<sup>-1</sup>, which is the characteristic band of C=C aromatic group. On account of carbon black, the filler-polymer association is for the most part of physical nature [12].



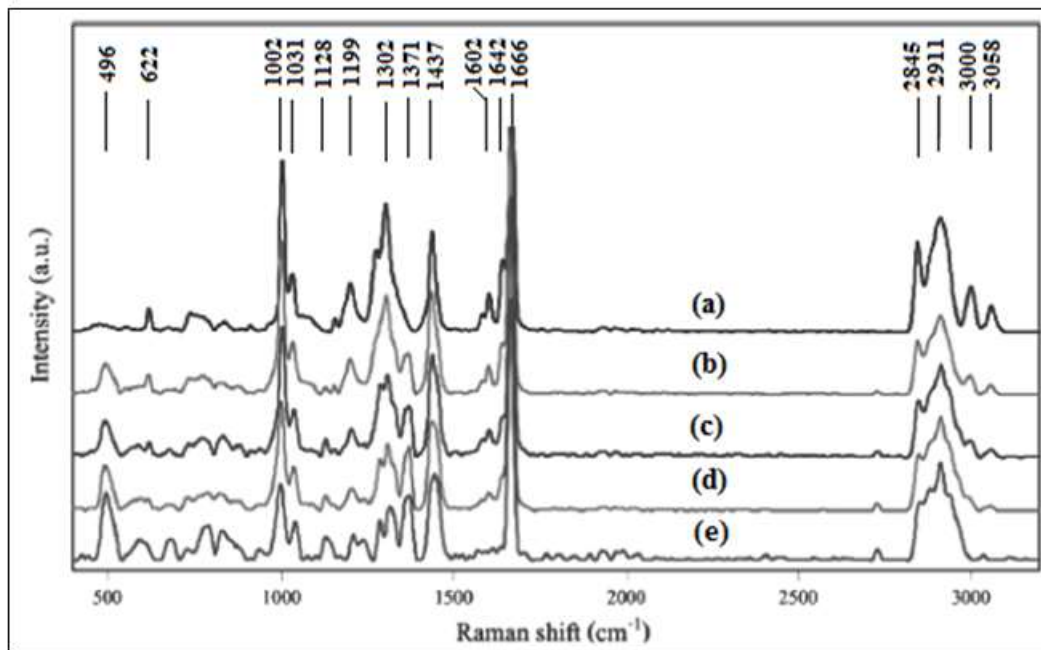
**Fig-2: FTIR (Fourier- transform infrared spectroscopy) Spectra of SBR- Nanocomposite (a) Unfilled Composites (b) 2 phr Carbon Black in SBR (c) 6 phr Carbon Black in SBR (d) 10 phr Carbon Black in SBR (e) 12 phr Carbon Black in SBR**

### Raman Spectroscopy

As indicated by the writing, the nearness of debasement and expansion of crosslinking operators, fillers and other elastic fixings into elastic are the fundamental reasons of fluorescence parasite motions in Raman dispersing range, particularly with the laser excitation lines in the obvious district [13]. The quantum yield of the fluorescence procedure is a lot higher than that of the Raman procedure, and along these lines, the principle spectroscopic data is covered. So as to obviously distinguish the trademark groups of SBR and SBR-nanocomposites, the coagulated elastic without carbon black was right off the bat chosen to perform Raman spectroscopy. The spectra of SBR and SBR-carbon black composites are thought about in Fig. 3 and band assignments are done dependent on correlation with writing spectra. The Raman pinnacles of SBR are just incompletely relegated in the writing [14]. This work gives a full attribution for which Raman investigation of polystyrene and butadiene elastic is a great direction. Both symmetric and asymmetric  $-\text{CH}_2$  and  $-\text{CH}_3$  extending vibrations ordinarily show up in the frequency region 2800–3000. Obviously,  $\text{C}=\text{C}$  extending vibrations of SBR are seen at frequency 1666 and 1668, correspondingly. Raman spectra of unfilled SBR samples at different mix proportions are delineated in Figure 3. As can be seen, the intensities of trademark signals at frequency 1002, 1302, 1602 and 3058 will in general increment with expanding carbon black substance in the SBR rubber, while the forces of the

separated trademark groups of SBR at 1371, 1128 and  $496\text{ cm}^{-1}$  diminish. The crest at frequency 1666 isn't subject to SBR proportion and is utilized as an inside standard. From this connection, it is conceivable to affirm organization in the mixes by plotting the intensity proportion of trademark signals of each rubber versus mix proportion. 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 twisting for SBR, respectively, were decided for developing an adjustment bend. This is on the grounds that these picked pinnacles have the best resolution and the trademark crest at frequency 1371 isn't covered. Raman characteristic signals of SBR are not essentially changed after expansion of carbon black. This perception is likely because of the combination of low quantum productivity in this frequency range ( $647.1\text{ nm}$ ) and the weak Raman intensity of carbon black particles which have an amorphous character. The trademark groups of carbon black are somewhat poor when the sample is energized at  $647.1\text{ nm}$ . Besides, these signals are not enhanced by expanding the number of sweeps and collection time. When utilizing excitation wavelength at  $514.5\text{ nm}$ , the carbon black sample demonstrates a Raman range having noticeable signals at  $456$ ,  $796$  and  $994\text{ cm}^{-1}$  corresponding to  $\text{C}-\text{OH}$  stretching vibration. Tragically, pinnacles of SBR allotted to  $=\text{CC}_2$  rocking and  $\text{C}-\text{CH}_2$  stretching are situated in a similar frequency region at  $496$  and  $1000\text{ cm}^{-1}$ , respectively [15]. Along these lines, it is hard to

recognize the Raman signals of carbon black from those of SBR.



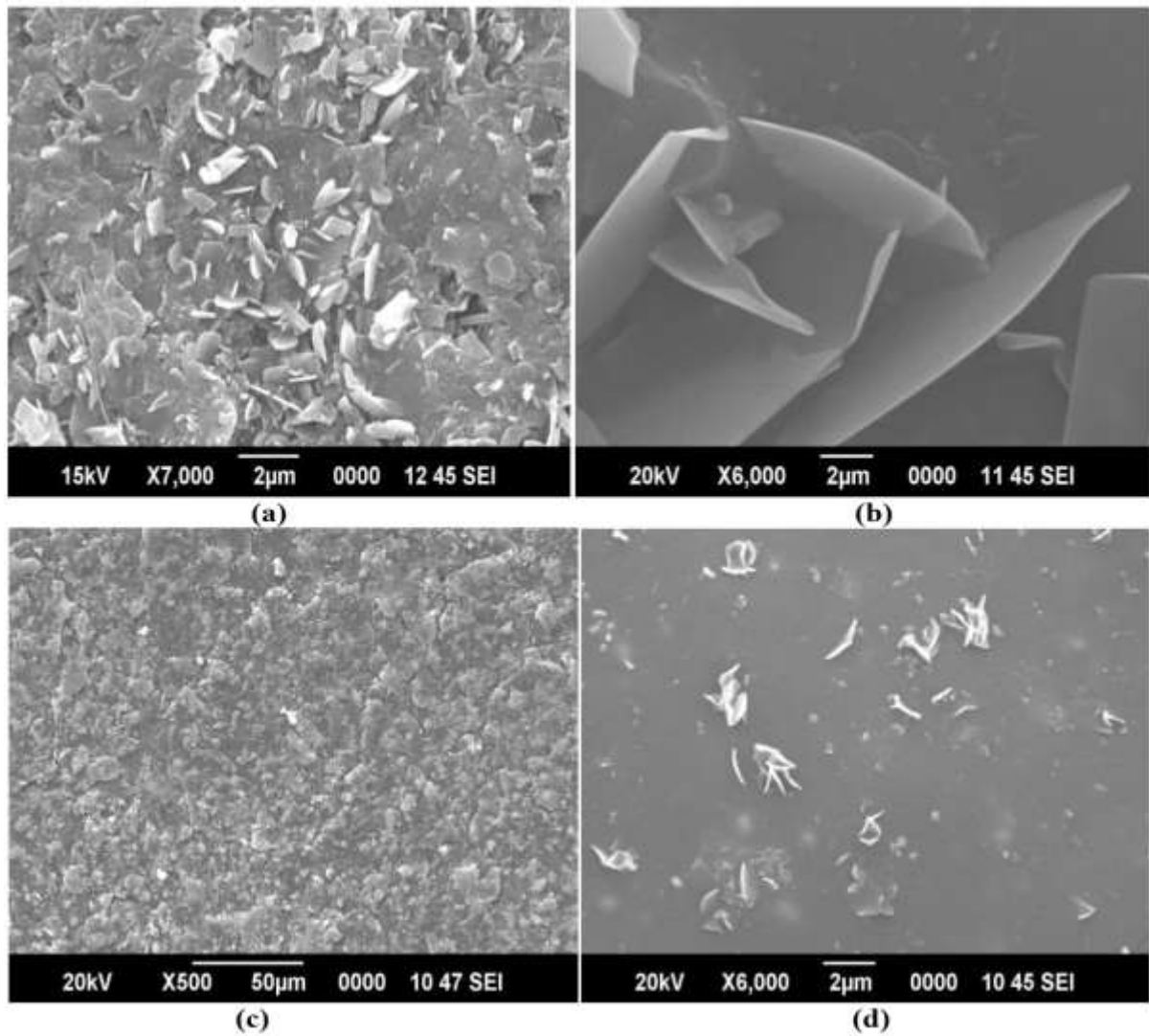
**Fig-3: Raman Spectra of SBR-Nanocomposite (a) Unfilled Composites (b) 2 phr Carbon Black in SBR (c) 6 phr Carbon Black in SBR (d) 10 phr Carbon Black in SBR (e) 12 phr Carbon Black in SBR**

### Morphology

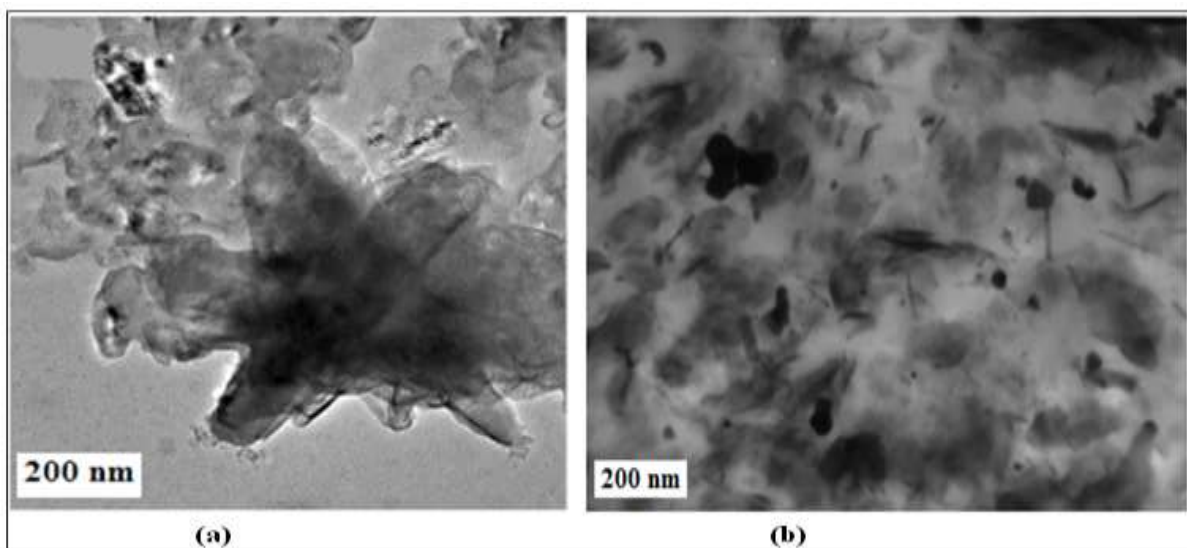
Figure-4 indicates SEM pictures for SBR-carbon black nanocomposites. The estimations were performed specifically on the brake surface (cross area) of the samples utilizing amplification level of multiple times [16, 17]. Morphology of the rubber composites at various filler stacking viz. at 2 phr, 6 phr, 10 phr, and 12 phr was explored by scanning electron microscopy (SEM) and micrographs appear in Figure-4. Figure-4 demonstrates the filler particles are homogeneously scattered all through the rubber grid with few totals at 2 phr filler content in rubber composites Figure 4(a) with increment in filler stacking (10 and 12 phr), the collection propensity of filler particles increments and it is much prevailing for elastic network loaded up with 12 phr Figure 4(d). In Figure 4(d), demonstrates the molecule estimate conveyance is in the scope of 100 to 150 nm for littler particles while the size of bigger filler particles totals lies in the scope of 300 to 450 nm. Uniform scattering of filler into elastic lattice is clear in all surface changed filler composites (a) to (d) Figure-4.

Figure-5 demonstrates the TEM pictures of SBR-Carbon black nanocomposites. In the (Figure-5(b)) dark area speak to the SBR elastic stage separately. Scattering of the carbon black particles having diverse shapes and sizes are apparent from the agglomerated dark spots in the TEM picture. Dispersion of the molecule estimate is observed to be roughly in the scope of 10-200 nm. In (Figure 5(b)) demonstrates the carbon black particles have pleasantly been scattered in the interfacial region of SBR rubber. Figure 5(a) demonstrates the SBR rubber TEM micrograph. The interlayer spacing created by the solvent molecules during the emulsion mixing, makes it possible for the elastomer chains to diffuse between the carbon black layers easily. Also, the migration of carbon black seems to affect the affinity between rubber and the carbon black, as other factors like viscosity and agglomeration could be less determining factors. The well dispersed filler can interact with the polymer much more in emulsion mixing. The images of SBR-carbon black composites clearly show the better dispersion of carbon black in the SBR system.





**Fig-4: Scanning Electron Microscopy (SEM) Images of SBR-Nanocomposite: (a) 2 phr filler, (b) 6 phr, (c) 10 phr, (d) 12 phr**



**Fig-5: TEM Morphology of SBR-Nanocomposite: (a) Unfilled Composite (b) SBR-Carbon Black Composites**

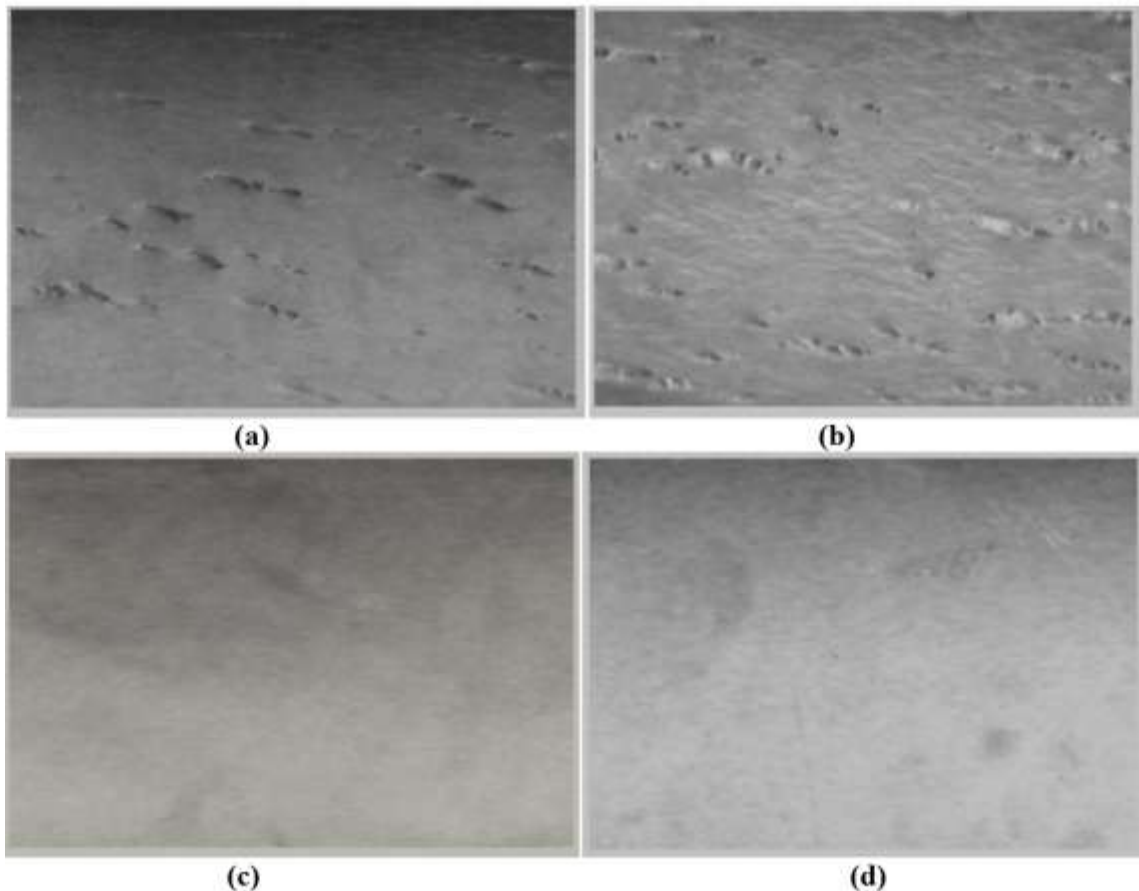
### Ozone Resistance

SBR elastic nanocomposites vulcanisates demonstrate better protection from ozone because of its polar nature. Figure 6 & 7 demonstrates the optical photos of the ozone illuminated surfaces of styrene-butadiene rubber nanocomposites in an ozone chamber at 50 pphm ozone fixations at 38°C. Inside the primary 4 hrs, itself splits were framed on the surface of SBR gum test. In any case, it is seen that as the filler stacking is expanded up to 12 wt% the time taken for the inception of break is expanded. For SBR-

nanocomposites at 2, 6, 10 and 12wt% of nano carbon black stacking; the breaks were created simply after 5hrs, 6hrs, 7hrs, and 8 hrs individually. It is comprehended that SBR-Carbon black nanocomposites (12 phr) up to specific carbon black substance are less inclined to ozone light which demonstrates that the correct scattering of carbon black in the elastic network is up to that dimension. The expansion in carbon black stacking demonstrated a slower advancement and proliferation of splits.



**Fig-6: Ozone Irradiated Surface of SBR Rubber after 4 hours of Exposure**



**Fig-7: Ozone Irradiated Surface of SBR Rubber Nanocomposites at different phr (a) 2 phr Carbon Black in SBR (b) 6 phr Carbon Black in SBR (c) 10 phr Carbon Black in SBR (d) 12 phr Carbon Black in SBR after 8 hours of Exposure**

Elastomers, particularly those containing dynamic twofold bonds in the primary chain, are seriously assaulted by ozone bringing about profound breaks toward a path opposite to the connected pressure. Security against ozone assault can be accomplished by the utilization of antiozonants. A few investigations have been accounted for on the security of elastic against ozone assault [18]. The ace bunch tests indicate preferable ozone opposition over the common means because of a more prominent impediment to the advancement of breakfront experienced because of the uniform appropriation of carbon black in the elastic lattice and the enhanced elastic filler communication.

### Flame Resistance

Flame resistance of vulcanizates of SBR elastic carbon black nanocomposites was assessed according to UL 94 examination test for combustibility vulcanizates of differing nano carbon black substance in SBR rubber were tried according to UL 94 by vertical consume test. SBR-carbon black nanocomposites kept consuming after the burner fire application and flaring

trickles touched off the cotton placed underneath the specimen yet set aside more opportunity to finish consuming than unadulterated SBR, showing enhancement in flame resistance [19, 20]. In the event that we need to use a framework as a fire retardant, it must qualify under the UL-94 convention. This is just a test that estimates the simplicity of termination of fire and four evaluations are conceivable, contingent on the consuming time and the nearness of flaming drips. If a material extinguishes quickly, without dripping, it is managed the rating V-0; a material that consumes somewhat longer degree, but does not trickle, is delegated as V-1; a material that does not self-smother in a brief timeframe but rather drips is given the rating V-2; if a material consumes longer than the convention indicates, it is said to be not characterized, NC. SBR-carbon black nanocomposites at 2 phr, 6 phr, 10 phr, and 12 phr wt% nano carbon black stacking indicates to some degree enhanced combustibility property than its gum as given in Table 3. This improvement in a property is because of the flame retardant property of nano carbon black.

**Table-3: Dripping and Burning Characteristics and UL-94 Classification for Rubber Nanocomposites**

Sample	Dripping	Burning	UL-94
SBR	Slow	Yes	NC
SBR-carbon black (2 phr)	Slow	Yes	NC
SBR-carbon black (6 phr)	Yes	Little	V-2
SBR-carbon black (10 phr)	Yes	Little	V-2
SBR-carbon black (12 phr)	Yes	Little	V-2

## CONCLUSIONS

Rubber nanocomposite was set up by the procedure of emulsion polymerization of styrene-butadiene elastic with nano carbon black and different fixings i.e. quickening agents and antioxidants. Morphology of rubber nanocomposite demonstrates the filler particles are homogeneously scattered all through the elastic grid. TEM think about demonstrates the nearness of carbon black nanoparticles of various shapes and sizes, for the most part, developed in SBR period of elastic mixes. Carbon black particles were all the more consistently scattered in SBR mixture. The ace clump tests demonstrate preferable ozone resistance over the normal means because of a more prominent obstacle to the advancement of crack front experienced because of the uniform dissemination of carbon black in the rubber matrix and the enhanced elastic filler communication. Fire opposition uses a framework as a fire retardant, it must qualify under the UL-94 convention. This is essentially a test that estimates the simplicity of annihilation of a flame and four evaluations are conceivable, contingent on the consuming time and the nearness of blazing trickles. SBR-carbon black nanocomposites at 2 phr, 6 phr, 10 phr, and 12 phr wt% nano carbon black stacking indicates fairly enhanced combustibility property than

its gum. This upgrade in property is because of the fire resistant property of nano carbon black.

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