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Article

Structural and Thermal Properties of Nitrile-Butadiene Rubber Reinforced with ISAF Carbon Black

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ABSTRACT: In this study, nitrile–butadiene rubber (NBR) composites reinforced with intermediate super abrasion furnace (ISAF) carbon black at different loadings (50, 60, 70, 80, 90, and 100 phr) were systematically investigated. Structural analysis via X-ray diffraction (XRD) revealed that the composites exhibited a predominantly amorphous character with a filler-dependent degree of crystallinity. Fourier transform infrared (FTIR) spectroscopy was employed to identify the functional groups involved in ionic crosslinking within the NBR matrix. Thermal stability was evaluated through thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) over the temperature range from ambient conditions to 400 °C, which revealed a significant increase in stability with increasing carbon black concentration. These results provide comprehensive insight into the intricate correlation between filler loading and the resulting thermal behavior of elastomeric composites. The findings reveal that variations in carbon black content significantly influence the heat transfer characteristics, thermal stability, and degradation resistance of the rubber matrix. Higher-structure grades tend to enhance thermal conductivity and improve heat dissipation, while optimal filler loading promotes balanced mechanical integrity and thermal performance. Overall, this understanding contributes to the rational design and optimization of carbon black–reinforced elastomeric materials tailored for advanced engineering applications requiring superior durability and thermal management capabilities.

1. INTRODCTION

Synthetic rubber is important in modern materials engineering because of its tunable mechanical properties, chemical resistance, and capacity to outperform natural rubber in demanding environments [1]. Historically, formulations such as styrene butadiene rubber (SBR), polybutadiene (BR), nitrile rubber (NBR), and ethylene propylene diene monomer (EPDM) rubber have enabled critical applications in tires, seals, hoses, vibration dampers, and industrial machinery owing to their resistance to abrasion, oils, temperature extremes, and weathering [2]. The incorporation of carbon black (CB) into rubber matrices has a significant effect on the mechanical, structural, and thermal properties of rubber. The main type of carbon black is furnace black, such as N220 (High Abrasion Furnace Black, HAF), N330 (Intermediate Super Abrasion Furnace Black, ISAF) and N660 (Medium Thermal/General Purpose Furnace Black, GPF) [3,4].

Recent studies have shown that the addition of carbon black not only influences the macroscopic properties of rubbers but also affects their molecular structure, which in turn governs their thermal behavior and overall performance. Structurally, carbon black particles interact with rubber polymers at the molecular level, forming a network that alters rubber morphology and rigidity. This interaction can lead to changes in the crosslink density, the arrangement of polymer chains, and the formation of interfacial zones that influence the material's stiffness and elasticity [5]. The dispersion of carbon black within the rubber matrix and the formation of aggregates or agglomerates play crucial roles in determining the final properties of the composite material [6]. Thermally, the inclusion of carbon black modifies the heat resistance of rubber compounds, affecting their glass transition temperature (T_g), thermal conductivity, and thermal degradation behavior. Recent work has focused on understanding how the structure of carbon black, such as its particle size, surface area, and level of dispersion in the matrix, impacts the thermal stability and heat dissipation properties of rubber composites [7].

Nitrile-butadiene rubber (NBR) is a synthetic copolymer composed of acrylonitrile (CH_2=CHCN) and butadiene (CH_2=CH-CH=CH_2) monomers. The balance of these two monomer units in the polymer chain gives NBR a unique combination of properties: good oil and hydrocarbon resistance, moderate tensile strength, and flexibility. The acrylonitrile

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content strongly influences properties such as swelling in oils, the glass transition temperature, gas permeability, and overall thermal stability. Owing to these excellent solvent and oil resistance characteristics, NBR has been widely used in demanding applications, including oil seals, hoses, belts, O-rings, gaskets, and brake linings, among others [8]. However, while NBR's intrinsic properties are very useful, many industrial applications require enhancements in mechanical strength, abrasion resistance, thermal stability, and durability, especially under severe operating conditions (high temperature, oil exposure, dynamic loading, etc.). One common solution to obtain these enhancements is to add reinforcing fillers. Carbon black, for example, has long been used to reinforce NBR by increasing the tensile strength, hardness, modulus, and resistance to wear and tearing [9]. In the NBR rubber industry, furnace blacks (e.g., N220, N330, N550, and N660) are the most common because they balance reinforcement, processability, and cost [10-12].

The present work focuses on studying the structural and thermal behavior of nitrile butadiene rubber (NBR) reinforced with intermediate super abrasion furnace (ISAF, or N330) carbon black. Particular attention is given to the underlying mechanisms responsible for the enhancement of material performance. Structural characterization of the composites was carried out via X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy, whereas their thermal stability was assessed via thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The novelty of this study lies in correlating the filler–matrix interactions of ISAF carbon black with both the crystallinity and the thermal degradation pathways of NBR, thereby providing new insights into optimizing filler selection for advanced elastomeric applications.

2. EXPERIMENTAL

Polymeric NBR sheets loaded with ISAF carbon black, prepared previously by our group authors [13], were used in this study. Samples with dimensions of 1x1 cm^2 were cut for characterization measurements and coded as N50, N60, N70, N80, N90 and N100 according to carbon black contents of 50, 60, 70, 80, 90 and 100 phr (part per hundred parts of rubber by weight), respectively. The structure of the NBR/ISAF composites was studied via X-ray diffraction (XRD) at room temperature with a "PANalytical X'Pert ProMPD-diffractometer" (the Netherlands) running at a voltage of 45 kV and a current of 40 mA. The equipment was fitted with a Ni filter that operates with an angle 2θ in the range of 10° to 100° using Cu Kα (λ = 1.540 Å) radiation. The chemical structure of the samples was characterized via Fourier transform infrared (FTIR) measurements using Burker VERTEX 80 (Germany) combined with a Platinum Diamond ATR, which comprises a diamond disk as an internal reflector in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and a refractive index of 2.4.

The thermal properties of the present samples were analyzed via thermogravimetric analysis (TGA), which was performed with a SHIMADZU model under a nitrogen atmosphere up to 800 °C at a heating rate of 10 °C min^{-1}. Additionally, differential scanning calorimetry (DSC), performed with a SHIMADZU differential scanning calorimeter, was carried out at a heating rate of 10°/min in a nitrogen atmosphere. The analysis was conducted in a temperature range between 50 and 400 °C.

3. RESULTS AND DISCUSSION

3.1. Structural analysis

3.1.1. *X-ray diffraction*

Figure 1 shows the XRD patterns of NBR/ISAF composites containing varying amounts of carbon black (50, 60, 70, 80, 90, and 100 phr). The broad diffraction bands observed across the XRD patterns suggest that the composites possess an almost amorphous structure with some degree of crystallinity. Notably, characteristic diffraction peaks of carbon black appear at approximately 2θ = 24° and 43°, which are easily identifiable [14]. These broad peaks correspond to Bragg diffraction from the (002) and (100) planes of carbon black, respectively [15]. Additionally, the sharp peaks observed at 2θ = 47°, 56.3°, and 68.7° are likely residuals of unvulcanized zinc (Zn) remaining from the curing process. The peaks at 2θ = 31.4° and 35.6° may be attributed to unvulcanized zinc oxide (ZnO) [16]. Furthermore, two prominent peaks at 2θ = 34° and 62.5° are probably due to residual unvulcanized sulfur (S) and oxygen (O) elements, corresponding to Bragg reflections from the S (044) and O (204) planes, respectively [17].

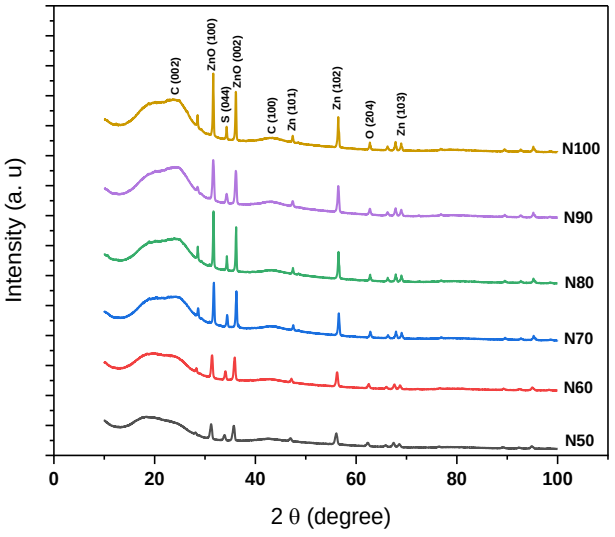

Figure 1. XRD patterns of NBR/ISAF composites with ISAF carbon black contents of 50, 60, 70, 80, 90 and 100 phr.

Analysis of the XRD curves revealed that the peak intensities increased with increasing carbon black content in the samples. Additionally, as the concentration of ISAF carbon black increased, the characteristic carbon black peaks shifted toward higher 2θ angles. Hence, the amount of carbon black filler could increase the degree of crystallinity of the resulting composites [18]. These trends suggest that adding more carbon black fillers could alter the crystallinity of the composites, as supported by previous studies [19]. The crystallite size (*L*) of the composites was determined from the XRD data via the Scherrer equation [20]:

$$L = \frac{K\,\lambda}{b\cos\theta} \quad (1)$$

In this equation, K is the Scherrer constant set at 0.9, θ represents the Bragg angle in radians, b is the full width at half maximum (FWHM) of the diffraction peak in radians, and λ = 1.54 Å is the wavelength of the X-ray photons. The calculated crystallite sizes, which are based on the primary carbon black peak, are presented in Table 1.

Table 1: Crystallite size of the NBR/ISAF composites extracted from the XRD data

Parameter	Samples					
	N50	N60	N70	N80	N90	N100
L(nm)	1.6321	1.6669	1.7779	1.855	1.9553	2.0849

Understanding these values as a function of filler content provides insight into the structural characteristics of the NBR/ISAF composites. Notably, the data clearly show that the crystallite size (L) increases significantly with increasing carbon black concentration, indicating a trend toward greater crystallinity as the filler content increases.

3.1.2. *FTIR Analysis*

Figure 2 presents the FTIR spectra of both raw NBR and synthesized NBR/ISAF composites containing different CB contents (50, 60, 70, 80, 90, and 100 phr). The spectra indicate the involvement of multiple functional groups in the ionic crosslinking process of NBR.

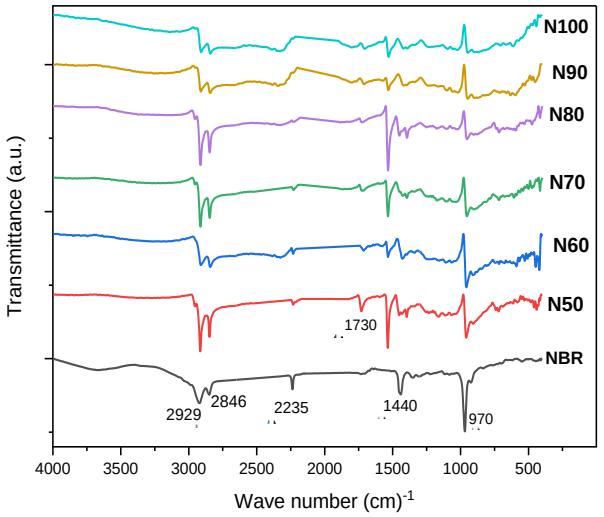

Figure 2. FTIR patterns of raw NBR and NBR/ISAF composites with ISAF carbon black loading contents of 50, 60, 60, 70, 80, 90, and 100 phr.

The typical absorption peak of the nitrile group (C≡N) stretching vibration is shown by NBR at 2235 cm^{-1} [21]. The presence of benzene rings is shown by the absorption band at 1440 cm^{-1} caused by aromatic C=C stretching vibrations. Methylene groups can be detected via absorption bands at wavenumbers 2929 and 2846 cm^{-1}, which are associated with the C–H stretching vibrations of CH_2 groups [22]. In addition, the C=C bonding of the alkane group is indicated by the peak at 970 cm^{-1} [23]. The FTIR spectrum clearly shows that the characteristic absorption peaks appear in the NBR/ISAF composites as well. The intensity of the peak at 2235 cm^{-1} decreased after vulcanization with increasing CB content. This is probably due to the conversion of the C-N group into a carboxyl group (COOH), which appears at the absorption band at 1730 cm^{-1} upon hydrolysis [24]. Some additional peaks appeared for samples N60 to N100, which had ISAF carbon black concentrations above 50 phr. This is probably due to the interaction of the CB filler with the NBR chains to form the composites.

3.2. Thermal analysis

3.2.1. *TGA characteristics*

The thermal stability of the vulcanized composites was examined via TGA measurements. **Figures 3&4** show the TGA curves of the NBR samples loaded with various concentrations of ISAF carbon black. **Figure 3** represents the weight of the samples (in *mg*) as the temperature increased from room temperature to almost 800 °C for all samples, whereas **Figure 4** depicts the derived weight (in *mg/min*), i.e., the rate of weight loss with temperature.

Generally, the TGA thermographs showed similar trends for all NBR/ISAF composites, with slight variations depending on the carbon black content. Three distinct stages of the thermal behavior of the material can be recorded. As an example, for sample N50, the first stage extends from approximately 32 °C to 312 °C, where 0.794 mg of sample N50 is lost with a ratio of 4.9% of its initial weight. The loss of moisture and gas outlets such as carbon monoxide and dopant could cause such weight loss in this stage. No decomposition of the sample could be considered in this temperature interval. The real thermal degradation is recorded in the second and third stages. The second one starts from approximately 312 °C up to 503 °C (for sample N50), with a weight loss of ~ 51%. This is followed by a full breakdown and decomposition of the polymer composite in the third stage, which occurs between nearly 503 °C and 606 °C, with a weight loss of ~ 39% [25]. At temperatures above 606 °C, the remainder of the sample is completely burned and converted to ash, where it loses more than 96% of its weight during the whole process [26, 27].

From **Figure 4** one can obtain the temperature corresponding to the maximum degradation (T_{max}) in each stage. For example, in sample N50, these values are 244, 456 and 558 °C, respectively.

Additionally, the temperature at 50% weight loss was determined to be 470 °C. The same findings are obtained for all the other samples, and these findings are shown in Table 2 for all the NBR/ISAF composite systems. Both the 50% weight loss temperature and the temperature at maximum decomposition are clearly influenced by the presence of ISAF carbon black in NBR.

The decomposition of NBR polymeric rubbers is normally accompanied by a degradation process caused by both the random-chain scission of butadiene and/or the nature of the additives in NBR. Moreover, the strong electronegative nitrile groups present in NBR lead to sufficiently high interactions and

high heat resistance in such elastomers [26]. Additionally, the data in Table 2 demonstrate that the onset temperature of the decomposition stages increased with increasing CB content of the NBR vulcanized composites. This could be utilized in considering NBR-based composites as heat-resistant materials that are useful in the manufacturing of automobile tires compared with other rubbers. This finding matches the work of Alneamah and Almaamori [28], who reported that the NBR degradation temperature is improved from 360 to 368 °C with the incorporation of polyimide.

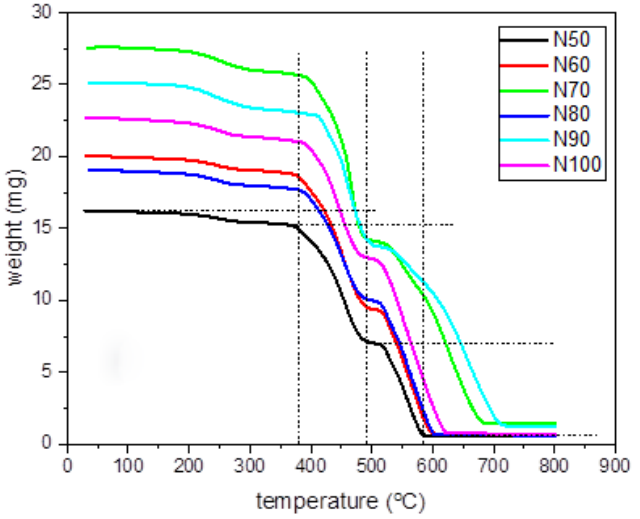

Figure 3. Weight versus temperature of NBR samples loaded with various contents of ISAF carbon black.

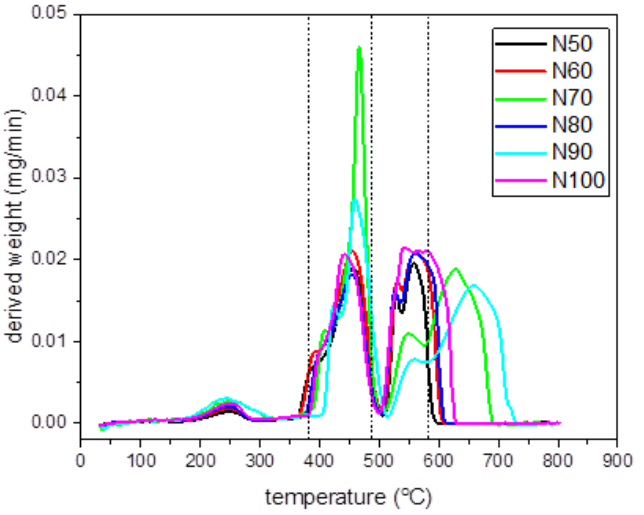

Figure 4. Derived weight versus temperature of NBR samples loaded with various contents of ISAF carbon black.

3.2.2. *DSC characteristics*

Another thermal characteristic, such as the crystalline temperature and/or melting behavior, can be obtained from DSC measurements. **Figure 5** shows the DSC curves of the synthesized NBR/ISAF samples with different carbon black contents: 50, 60, 70, 80, 90, and 100 phr. The charts represent the amount of heat flux due to the increase in the sample temperature from room temperature to 400 °C.

The Figure reveals that, below approximately 320 °C, the heat flux of the systems continuously decreases with increasing temperature, which indicates endothermic features of the samples. The decrease in the heat flux is followed by a noticeable increase at almost 330 °C, in which minimum in the curves occur. The minimum in the DSC curve is an indication of the presence of an exothermic transition of the NBR/ISAF composites that probably determines the crystallization temperature of such semicrystalline polymers [29]. The semicrystalline feature of the synthesized NBR/ISAF composites, rather than the normal amorphous phase of NBR, may be attributed to the presence of ISAF carbon black with various contents in the NBR matrix. This conclusion matches well with the XRD results, which revealed that the crystallinity of the composites tended to increase with increasing carbon black content. A similar finding was obtained by Farida et al. [30] as an effect of loading carbon black in natural rubber. Inspection of the DSC thermographs revealed noticeable maxima in the curves at approximately 370 °C for all the samples, which may represent the melting point of the NBR/carbon black system [31].

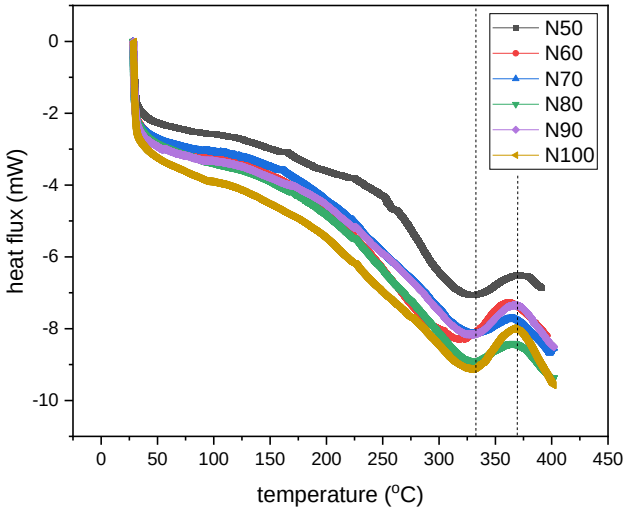

Figure 5. DSC curves of NBR/ ISAF carbon black composites with various carbon black contents.

Table 2: Degradation temperature (T_d **°C**), weight loss percentage (wt %), temperature at peak derived weight loss (T_{max} **°C**) and degradation temperature at 50% weight loss in each thermal stage of NBR/ISAF samples extracted from TGA curves.

Sample	1st stage			2nd stage			3rd stage			50% wt loss
	T_d	wt%	T_{max}	T_d	wt%	T_{max}	T_d	wt%	T_{max}	T_d
N50	32-312	4%	244	312-503	51%	456	503-606	39%	558	470
N60	31-313	4%	247	313-501	48%	455	501-614	43%	560	478
N70	41-325	6%	249	325-573	53%	466	573-701	34%	628	530
N80	35-322	5%	247	322-498	41%	454	498-661	49%	561	520
N90	37-338	7%	243	338-515	37%	459	515-726	49%	658	558
N100	31-319	5%	251	319-497	37%	441	497-635	53%	541	530

4. CONCLUSIONS

Composites of NBR loaded with various amounts of ISAF carbon black were synthesized via a mixing technique and vulcanized at 150 °C and 15 MPa. Structural characterization, as obtained from the XRD patterns, revealed an increase in the crystallite size with increasing carbon black content. The characteristic FTIR absorption peaks clearly appeared in the NBR/ISAF composites. The intensity of the peak at 2235 cm^{-1} decreased after vulcanization with increasing CB content, which is probably due to the conversion of the C-N group into a carboxyl group (COOH), which appears at the absorption band at 1730 cm^{-1} upon hydrolysis. The thermal analysis of the composites revealed real thermal degradation of the samples from approximately 312 °C, suggesting the safe use of such polymeric elastomers in practical and technological applications before reaching this temperature.

CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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