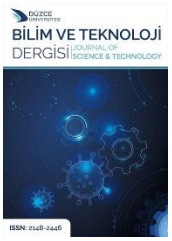

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Research paper

Biodegradable CNFC/EPDM Green Composites: A Sustainable Alternative for Automotive Sealing Profiles

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ABSTRACT

Sustainable material development is one of the most researched topics in the automotive industry. Sealing profiles, which are indispensable components of automotive vehicles, contain many petroleum-based products which automotive manufacturers are working to replace with biodegradable materials. The aim of this study is to investigate the effect of carboxylated nanofibrillated cellulose (CNFC) on the ethylene propylene diene monomer (EPDM) rubber in terms of evaluation some properties such as mechanical, chemical, thermal, morphological, rheological, and deformation. The CNFC was added at levels of 1, 3, 5, and 10 parts per hundred parts of rubber (pphr) to the EPDM rubber compound instead of synthetic and petroleum-based EPDM to develop CNFC/EPDM green composites. The rheological results showed that the CNFC/EPDM green composites were still acceptable according to the strength specifications of automotive manufacturers. Despite some lower mechanical properties, CNFC addition up to 10 pphr was still a suitable replacement for EPDM because the values were within specifications of automotive manufacturers. However, when the CNFC was added at more than 3 pphr, the lifespan of the CNFC/EPDM green composites was limited to three years according to the weathering results.

Keywords: *EPDM, CNFC, Nanofibrillated Cellulose, Sealing Profiles, Biodegradable Fillers*

I. INTRODUCTION

Ethylene propylene diene monomer (EPDM) is the most common synthetic rubber. Ethylene propylene diene monomer has become a widely used material in almost every industry due to its many advantages, including very good elasticity at low temperatures, high dielectric properties, high filling and oil absorption features for creating economical mixtures, excellent resistance to chemicals, weather conditions, ozone, heat, and oxidation, a low density that facilitates the production of low weight products, and high resistance to aqueous solutions, concentrated acid, and alkalis (Ginic-Markovic et al., 2000; Kucuk et al., 2018).

The automotive industry uses EPDM extensively. From commercial vehicles to luxury vehicles, all automotive vehicles have sheet metal, plastic, or fiberglass exteriors with multiple body panels, and nearly all automobiles have windows in the side doors. Consequently, various designs of air-retention strips, called sealing profiles, are needed to seal the gaps between body panel structures (Keeney & Mayfield, 2002). Considering the end-product requirements set by automotive manufacturers, EPDM rubber is reinforced

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with various fillers, such as carbon black, white fillers, process oils, activators, and vulcanizers (Delor-Jestin et al., 2000; Feldman, 2002; Arroyo et al., 2003; Choi et al., 2003; Snijders et al., 2005; Morlat-Therias et al., 2006; Kumar et al., 2007). All of these reinforcement materials are inorganic and, due to their nature, hazardous to humans and the environment (Dikmen Kucuk et al., 2022).

In recent years, the automotive industry has incorporated organic and natural bio-degradable materials in the manufacturing process. Many automotive manufacturers have started to research and study the use of biodegradable contents instead of petroleum-based materials in the interior and exterior parts of vehicles (Freise, 2002). This has created a demand for biodegradable materials instead of petroleum-based EPDM rubber for the sealing profiles that are used in most of the vehicle.

Cellulose is a good alternative among organic biodegradable materials due to its abundant nature, renewability, and superior mechanical and physical properties. Recently, the use of cellulose in nanofibrils such as nanofibrillated cellulose (NFC) and nanocrystalline cellulose (NCC) with nanotechnological developments has made it a sought-after natural material (Clarkson & Youngblood, 2018). There are many studies on the use of NFC in polymers. The results from these studies commonly show that incorporating NFC into the polymer matrix improves the mechanical properties of the polymer (Sarkhel & Choudhurry, 2008; Menezes et al., 2009; Ansari et al., 2015; Xu et al., 2015; Lavoratti et al., 2016; Nair et al., 2017). In addition to previously studied materials, research has also explored the use of TEMPO (2,2,6,6-tetramethylpiperidine 1-oxyl) oxidized nanofibrillated cellulose (TEMPO–CNF) in EPDM composites (Dikmen Kucuk et al., 2020). In a subsequent study, carboxylated nanofibrillated cellulose (CNFC) was developed via a carboxylation process applied to wood-derived cellulose fibers as an alternative to TEMPO oxidation, yielding enhanced results (Li et al., 2021). Studies investigating the influence of carboxylation degree on CNFs and their nanocomposites have shown that although carboxylation may reduce the crystallinity and thermal stability of CNFs (Kim et al., 2000; Hua et al., 2016), the resulting composites exhibit significantly improved mechanical performance, as well as enhanced thermal and thermomechanical stability, compared to their unreinforced counterparts (Cheng et al., 2016). For instance, in composites reinforced with carboxylated cellulose fibers, a 377% increase in tensile strength and an 86% increase in elongation at break have been reported (Larraza et al., 2020).

Despite the widespread use of carboxylated cellulose nanofibers (CCNF) in various polymeric composites (Hubbe et al., 2008; Aulin et al., 2009; Lin et al., 2012), there remains a lack of studies focusing specifically on their effect when incorporated into an EPDM rubber matrix. Therefore, the aim of the present study is to investigate the effect of CNFC on EPDM rubber by evaluating its mechanical, chemical, thermal, morphological, rheological, and deformation properties. Ultimately, this study aims to assess the potential of CNFC as a sustainable, biodegradable alternative to conventional petroleum-based EPDM rubber.

II. MATERIAL AND METHOD

A. Material

The EPDM rubber mixture used in this study was prepared by the company Standard Profile (Düzce, Turkey). The formula for the EPDM rubber mixture is given in Table 1. The biodegradable and non-toxic CNFC material was used in a certain amount instead of the EPDM. The CNFC was supplied commercially from the company Cellulose Lab (Fredericton, Canada).

Table 1. CNFC/EPDM composite formulas.

Sample	EPDM	Carbon Black + White Filler	Process Oil	Activators	Sulphur (S)	CNFC
EPDM	100	165	63	11	6.5	0
EPDM-C1	99	165	63	11	6.5	1
EPDM-C3	97	165	63	11	6.5	3
EPDM-C5	95	165	63	11	6.5	5
EPDM-C10	90	165	63	11	6.5	10

*All units are parts per hundred parts of rubber (pphr)

B. Method

B.1. Preparation of CNFC/EPDM Composites

As presented in Table 1, CNFC was incorporated into EPDM rubber at concentrations of 0, 1, 3, 5, and 10 parts per hundred parts of rubber (pphr), replacing an equivalent amount of petroleum-based EPDM. For each formulation, three replicates were prepared, resulting in a total of 15 distinct EPDM rubber plates. The materials were mixed for 5 min at a speed of 47 rpm in a lab-scale mixer (Carter Bros, Manchester, England). The volume of the mixer was 1.5 L with internal temperature 23 °C to 100 °C. The compound rubber samples were prepared by passing them through a lab-scale cylinder and pressing 7.5 min at 180 °C under 10 MPa pressure with a lab-scale injector. The devices used in the sample preparation stage and the samples obtained are shown in Figure 1 and Figure 2, respectively.

Figure 1. CNFC/EPDM composites preparation.

Figure 2. CNFC/EPDM composites samples.

B.2. Fourier Transform Infrared Spectroscopy Analysis (FTIR)

The chemical analysis of the composites was made with Fourier transform infrared (FTIR) spectroscopy, using a Shimadzu IR Prestige-21. The IR spectra were taken to see the chemical interaction from the vibration frequencies of the bonds between the EPDM and the CNFC. The molecular vibration signals were in the range of 4000 cm^{-1} to 600 cm^{-1}. The resolution was 4 cm^{-1} and the number of scans to record IR spectra was 32.

B.3. Thermogravimetric Analysis (TGA)

The prepared plates were analyzed thermally via a Shimadzu DTG 60 test machine. After the thermogravimetric analysis (TGA), the mass changes of the samples were measured. The TGA analysis were conducted at a speed of 75 mL/min and the temperatures changed between 0 °C to 550 °C with a heating rate of 20 °C/min.

B.4. Mechanical Properties

The mechanical properties of the plates were determined in terms of the tensile strength, tear strength, permanent set values, and elongation at break. The tensile strength and the elongation at break tests were carried regarding to the DIN standard 53504 (2017), while the tear strength was performed according to the ISO standard 34-1 (2015) standard. The mechanical tests were performed using a universal testing machine at room temperature. The speed of the test machine was 200 mm/min.

The permanent set values were measured by aging the plates 22 h + 2 h at 100 °C regarding to the DBL standard 5571 (2011). The heights of the samples were measured before and after aging with a CR2032/192-613 Mitutoyo height gage (Kawasaki, Japan). The values were calculated according to Eq. 1,

$$\text{Permanent Set} = \frac{\text{hi} - \text{hf}}{\text{hi} - \text{h0}} \text{x } 100 \quad (1)$$

where h_i is the height of the sample before thermal aging, h_f is the height of the sample after aging, and h_0 is the compression distance.

B.5. Rheological Properties

EPDM composites were controlled rheologically in terms of mooney viscosity, mooney scorch, and the moving die rheometer (MDR) according to the ASTM standard D1646 (2018) with an MDR 2000 test machine. Mooney viscosity test, which shows the flow motion, and shape-taking properties of the plates was carried out for (1 min preheating +4 min testing) at 100 °C. The vulcanization properties of EPDM plates in the extruder were simulated by mooney scorch test which was performed for (1 min preheating + 20 min testing) at 121 °C. The MDR tests which show the vulcanization times of ts_2 (scorch time) and t_{90} (90% optimum vulcanization time of rubber) were conducted for 2.5 min at 180 °C.

B.6. Artificial Weathering Test

The artificial weathering results of samples were simulated with an Atlas Ci4000 Weather-Ometer (Mount Prospect, IL). The accelerated weathering was performed for 100 h and 250 h regarding to the Volkswagen standard PV 3929 (2011). The wavelength of the ultra-violet (UV) radiation was 300 nm to 400 nm and the irradiance intensity was 75 W/m^2. The temperature was 90 °C with 20% relative humidity.

B.7. Artificial Weathering Test

The surface morphology of the CNFC/EPDM green composites was analyzed using an FEI Quanta FEG 250 optical microscope (Hillsboro, OR). An electron accelerating voltage of 20 KeV was used, and SEM photographs were taken during the analysis to view the chemical characterization of the composite plates.

III. RESULTS AND DISCUSSIONS

Chemical Characterization

The FTIR spectra of the composites are presented in Figure 3. The asymmetric, symmetric, and bending vibrations of the rubber components significantly overlap, making them difficult to distinguish. The C–H stretching vibrations observed between 2925 cm^{-1} and 2853 cm^{-1}, and the bending vibrations between 1460 cm^{-1} and 1377 cm^{-1}, correspond to CH_2 and CH_3 groups, respectively, which are characteristic of the EPDM structure. These C–H vibrations exhibit the highest intensity, partly due to the contribution of C–H bending vibrations from the CNFC. Peaks observed around 2320 cm^{-1} and 2180 cm^{-1} are attributed to the CS_2 groups, indicating the presence of crosslinking. Based on the FTIR analysis, the incorporation of CNFC does not lead to degradation of the polymer matrix.

In a study investigating the effects of microcrystalline cellulose (MCC), used as a substitute for EPDM in EPDM elastomer composites, C-H asymmetric vibrations were observed at 2916 cm^{-1}, while C-H symmetric vibrations were observed at 2845 cm^{-1}. The study, which noted that C-H bending vibrations were observed at 1376 cm^{-1} and 1462 cm^{-1}, observed cross-linked C-S bonds at 2310 cm^{-1} and 2095 cm^{-1}, respectively. In that study, MCC affected the chemical structure of the EPDM rubber matrix, while the CCNF used in our study did not cause any chemical changes in the EPDM matrix (Poyraz et al., 2022).

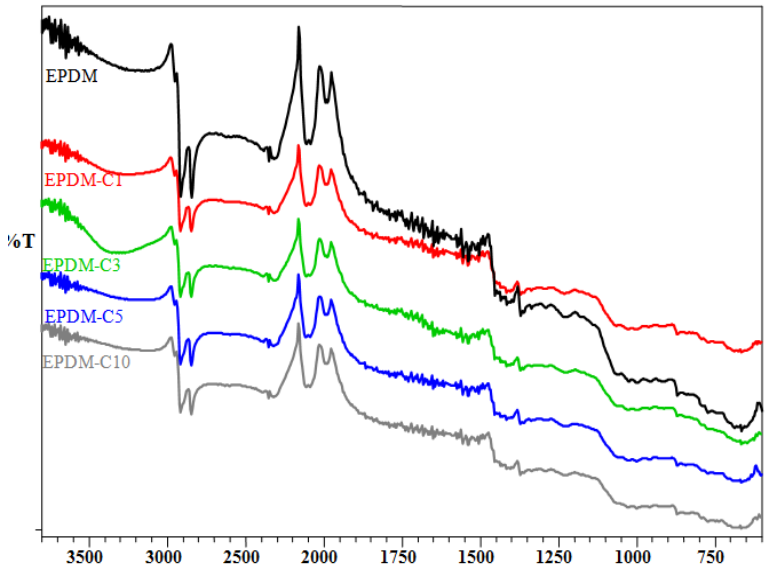


Figure 3. FTIR spectra of CNFC/EPDM composites.

Thermal Characterization

According to the TGA curves (Figure 4), the initial decomposition of CNFC begins around 320 °C, and a notable mass loss is observed in the range of 300–400 °C. Compared to the neat EPDM sample, the total weight loss of the 10 pphr CNFC composite increased from 26.4% to 34.7%, indicating a 31.4% relative increase in thermal degradation due to the presence of thermally less stable CNFC. This confirms that increasing CNFC content decreases thermal stability, consistent with the nanocellulose's tendency to degrade at lower temperatures than EPDM. In the study investigating the thermograms of EPDM elastomers containing MCC, it was reported that nanocellulose reduces the thermostability of EPDM due to the restriction of chain mobility in the matrix at long distances, which supports our finding (Poyraz et al., 2022; Shimazaki et al., 2007).

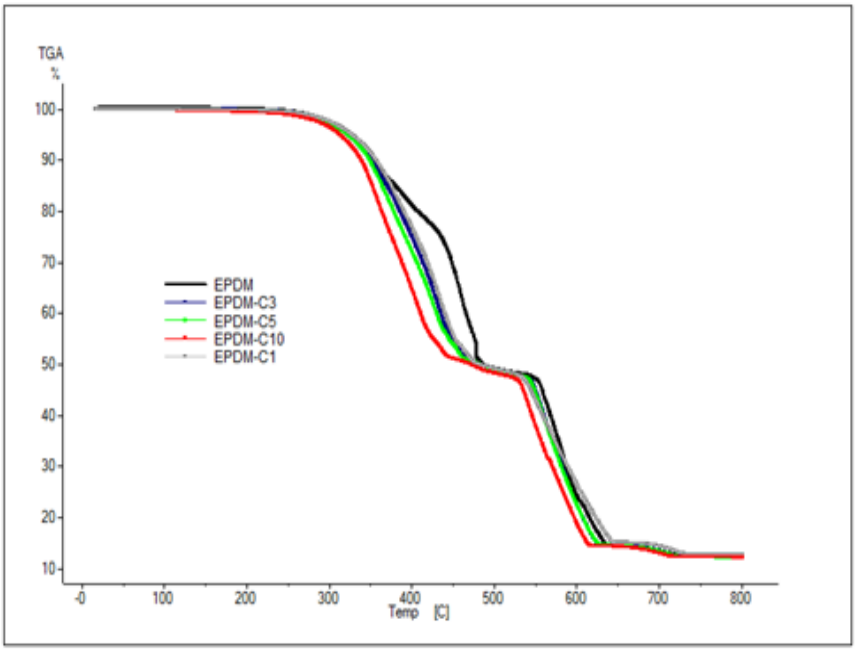


Figure 4. Thermal properties of CNFC/EPDM composites.

Mechanical Properties

When the CNFC was used instead of the EPDM in the EPDM rubber compound, the tensile strength and the elongation at break values decreased while the tear strength and the permanent deformation values increased. The decreased tensile strength and elongation at break values were caused by the chemical bond between the CNFC and the EPDM. The tensile strength and the elongation at break curves can be seen in Figure 5 and Figure 6, respectively.

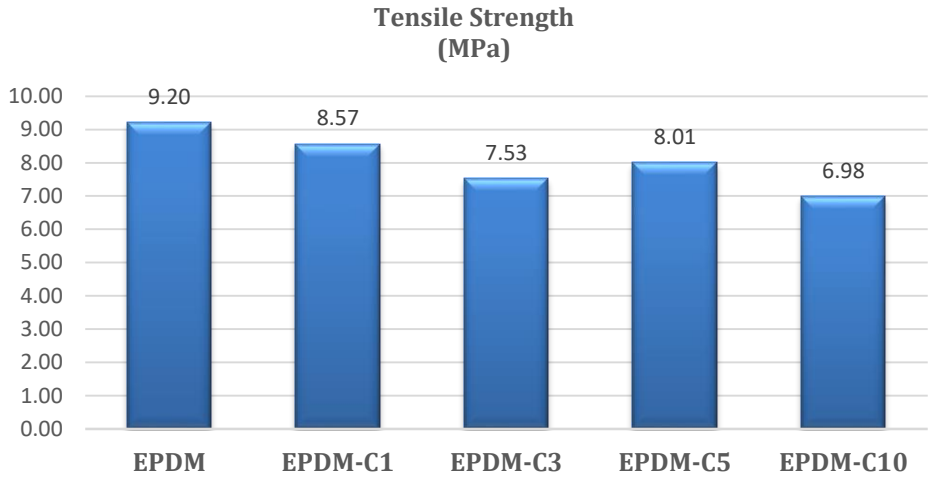


Figure 5. CNFC effect on tensile strength of EPDM rubber.

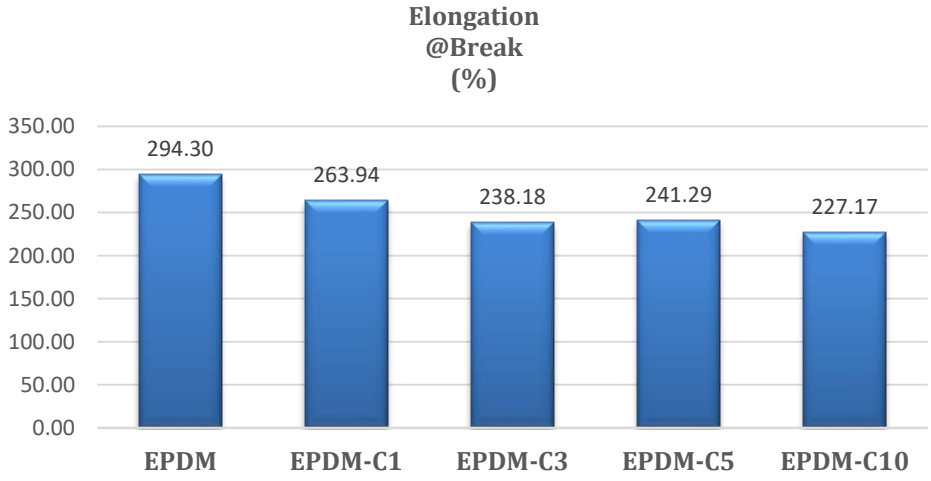


Figure 6. CNFC effect on elongation at break of EPDM rubber.

As shown in Figure 5, the tensile strength of neat EPDM was measured as 8.9 MPa, whereas it decreased to 7.5 MPa with 3 pphr CNFC and 7.1 MPa with 5 pphr CNFC, corresponding to an overall 20.2% reduction. Although a significant drop was observed, the values remained within the acceptable limits set by the Volkswagen standard (≥7 MPa). However, at 10 pphr CNFC loading, the tensile strength further declined to 6.6 MPa, falling 25.8% below the reference value and breaching the required threshold, thereby confirming the mechanical performance limit. The addition of CNFC to the EPDM compound resulted in a gradual decrease in tensile strength. This decline can be attributed to the restricted polymer chain mobility and increased rigidity introduced by the CNFC particles. Despite the reduction, all values remained above the minimum tensile strength requirement of 7 MPa defined by the Volkswagen standard TL 52345 (2013), up to a CNFC content of 10 pphr. Beyond this concentration, however, the tensile strength falls below the acceptable limit. This situation can be explained by the saturation point. In this study, the saturation point refers to the maximum CNFC content (10 pphr) at which the tensile strength remains within the acceptable specification range. Addition of CNFC beyond this level results in further weakening of the composite

structure, likely due to particle agglomeration and poor stress transfer between the matrix and filler. The elongation at break values exhibited a decreasing trend with increasing CNFC content. While neat EPDM demonstrated an elongation of 440%, this value dropped to 385% at 3 pphr CNFC and 360% at 5 pphr CNFC, indicating a 18.2% reduction. At 10 pphr, elongation further decreased to 330%, representing a total decrease of 25% relative to the control. This decline can be attributed to the restriction of polymer chain mobility caused by the rigid CNFC fibrils embedded within the elastomeric matrix, which reduces flexibility and ductility. A lot of studies in literature support these values. When EPDM was reinforced by rice husk, the tensile strength decreased, but it was within specifications with a value of 8.6 MPa (Siriwerdana et al., 2001). Similarly, EPDM and natural rubber (NR) mixing compound was filled with rice husk and the tensile strength value was found to be 13.5 MPa (Arayapranee & Rempel, 2008). In another study, EPDM and styrene-butadiene rubber (SBR) mixing compound with hemp hurd powder filler had a tensile strength value of 9.8 MPa (Ahmad & Luyt, 2012). The tensile strength and the elongation at break values decreased as the CNFC content increased. This can be explained by the fact that the CNFC made the EPDM compound more rigid, causing a limit within the polymer chains of rubber. This caused a lower ductility due to the stretching (Wang et al., 2011).

On the other hand, the tear strength values showed a non-linear trend. As seen in Figure 7, the tear strength decreased slightly from 6.2 N/mm^2 (pure EPDM) to 5.6 N/mm^2 at 3 pphr CNFC due to limited filler-matrix interface interaction. However, at 10 pphr CNFC, the value increased again to 6.3 N/mm^2, surpassing that of pure EPDM. This reversal can be explained by filler agglomeration at higher concentrations, which can form microbarriers that resist crack propagation, thus improving tear resistance. The saturation point for tear strength appears to be between 5 and 10 pphr, where the balance between dispersion and particle interaction begins to shift. If CNFC is added at levels higher than 10 pphr, it exceeds the saturation point, resulting in difficult tearing (Wang et al., 2011; John & Thomas; 2008). Because low CNFC additions (up to 5 phr) tend to reduce tear strength due to their reinforcing effect, this suggests that at low CNFC particle concentrations, sufficient adhesion can be achieved due to the increased surface area and strong interaction between the polymer matrix and the CNFC particles. However, at higher concentrations, tear strength values increase due to the tendency of the CNFC particles to agglomerate.

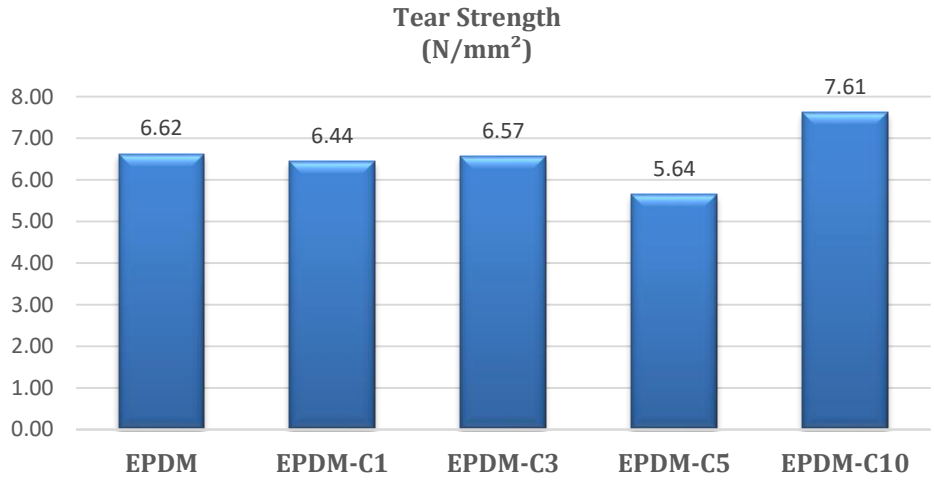


Figure 7. CNFC effect on tear strength of EPDM rubber.

The observed changes in mechanical properties can be explained by considering the molecular-level interactions between CNFC and the EPDM matrix. CNFC consists of rigid, high-aspect-ratio fibrils with abundant surface carboxyl and hydroxyl groups. When incorporated into the EPDM matrix, these polar groups form weak hydrogen bonds and van der Waals interactions with the relatively non-polar EPDM chains. This limited interfacial adhesion restricts stress transfer under load, which leads to decreased tensile strength and elongation at break. Additionally, the rigid nature of CNFC particles disrupts the mobility of the elastomeric chains, reducing the flexibility of the matrix. At lower CNFC concentrations, these particles may act as stress concentrators, while at higher loadings, particle agglomeration further impedes uniform dispersion and stress distribution. Consequently, the polymer chains experience localized stress, leading to premature failure and deterioration in mechanical performance. This mechanistic interpretation is consistent with previously reported findings for nanocellulose-reinforced rubber composites (Poyraz et al., 2022).

The deformation property increased with the addition of CNFC. Increasing of permanent set values means loosing the elasticity of EPDM rubber. However, as seen in Figure 8, the values are close to each other and are within the specifications of the Volkswagen standard TL 52345 (2013) that the permanent set values should reach a maximum value of 35%. Therefore, the CNFC did not have a noticeable impact on the permanent set values of the EPDM rubber.

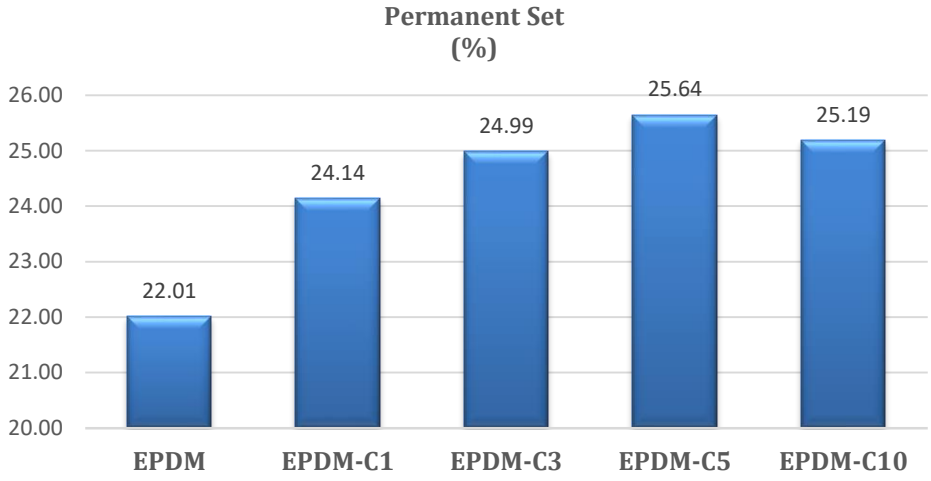


Figure 8. CNFC effect on permanent set of EPDM rubber.

Rheological Properties

The rheological properties of the CNFC/EPDM green composites were investigated to determine their process and vulcanization conditions. As seen in Figure 9, lower viscosity values were found after the addition of CNFC. This can be attributed to the presence of a weak EPDM-CNFC interaction due to increased molecular motion. Therefore, it can be predicted that the torque values required for vulcanization will decrease, and more tensile forces will be required to rotate the rotor. In a study examining the curing and tearing properties of bentonite-filled EPDM rubber composites, it was found that torque values increased with increasing viscosity values (Mat et al., 2016). Figure 10 and Figure 11 show that the scorch, ts_2, and t_{90} values, which allow estimation of production conditions and curing time, did not change. While micro- and macro-sized cellulose additives, available in the literature, accelerated vulcanization and altered the process conditions, nano-sized additives were found to cause no change in the process and curing conditions (Poyraz et al., 2022; George et al., 2021; Miao & Hamad, 2016; Nafeesa & Azura, 2018).

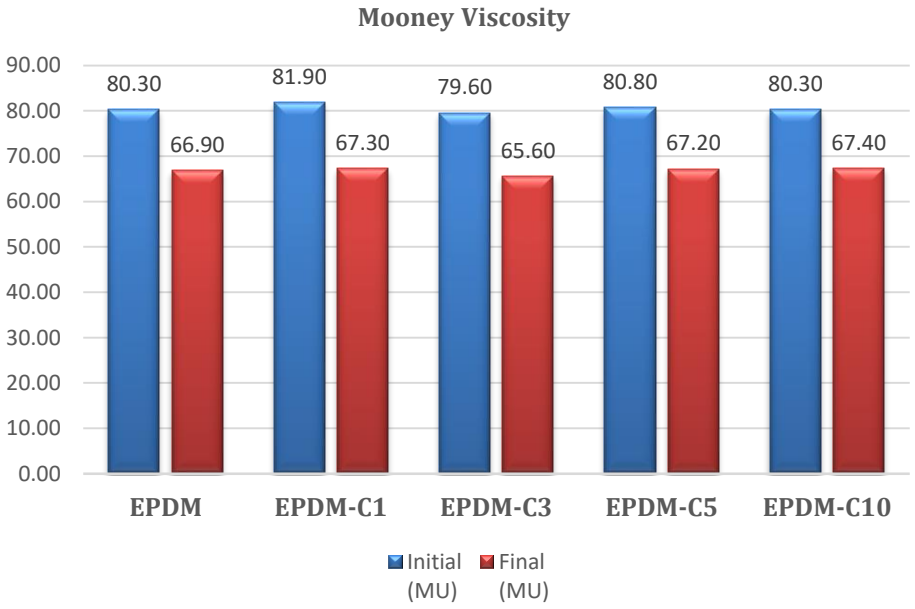


Figure 9. CNFC effect on mooney viscosity of EPDM rubber.

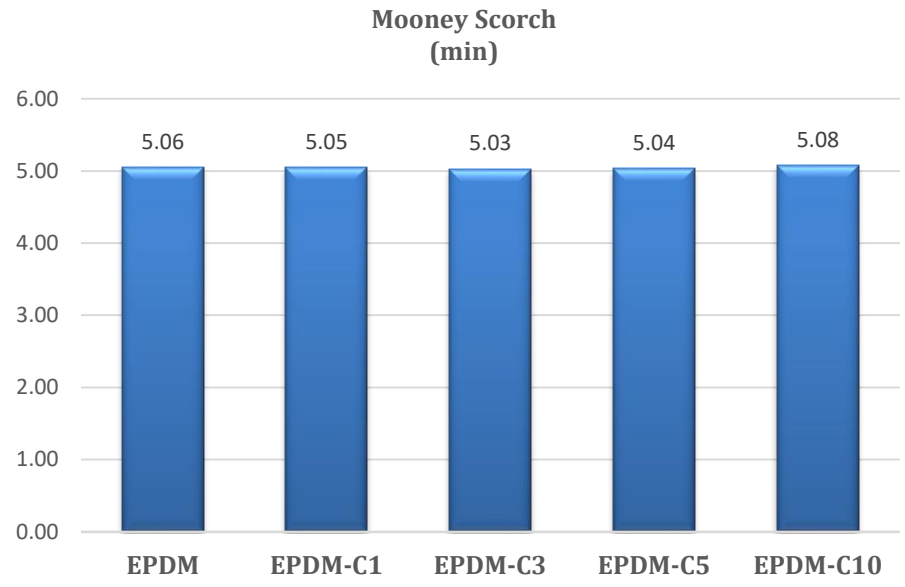


Figure 10. CNFC effect on mooney scorch of EPDM rubber.

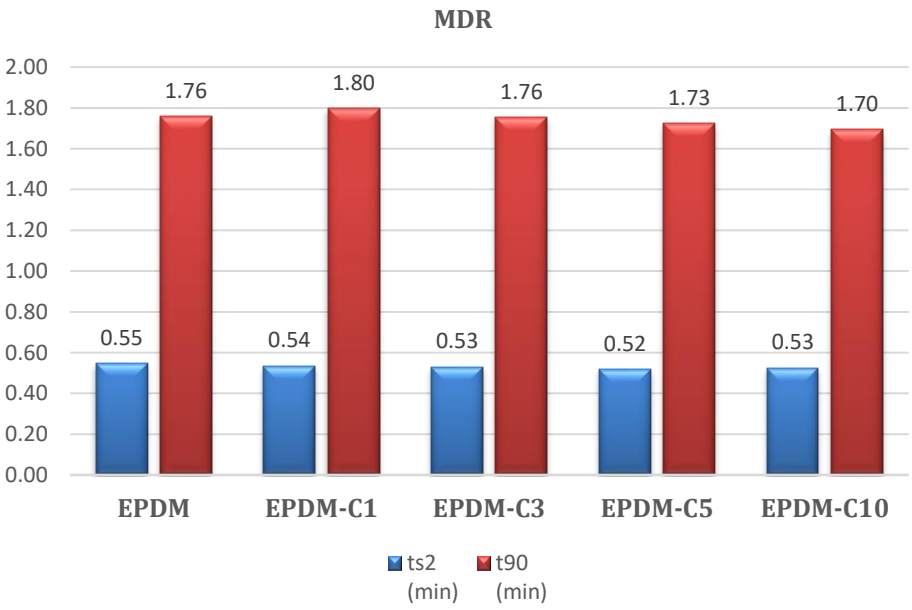


Figure 11. CNFC effect on vulcanization times of EPDM rubber.

Artificial Weathering Analysis

The visual controls of the plates after weathering 250 h are shown in Figure 12. After 100 h of artificial Kalahari weathering, there was no cracking, whitening, staining, or color change on the surface. However, after 250 h of weathering, staining, and whitening started to occur on the surface of composites if the CNFC was added at more than 3.0 pphr. 100 h of Kalahari weathering equates to a three-year lifespan (Thomas & Shaw; 1991). The weathering results showed that if CNFC was added at levels higher than 3.0 pphr, the CNFC/EPDM composite had a three-year lifespan.

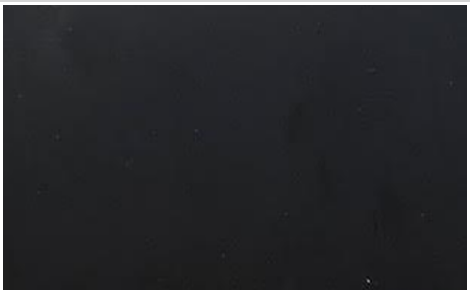
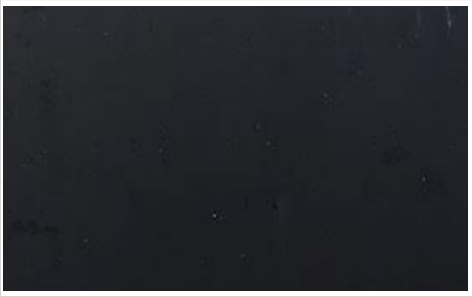
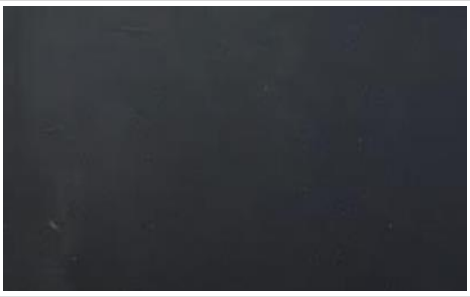
EPDM Plate	100 h Kalahari Aging	250 h Kalahari Aging
EPDM		
EPDM-C1		
EPDM-C3		
EPDM-C5		
EPDM-C10		

Figure 12. Visually control after weathering of CNFC/EPDM composites.

Scanning Electron Microscopy (SEM) Analysis

The SEM images presented in Figure 13 reveal the surface morphology of the CNFC/EPDM composites. At lower CNFC concentrations (1 and 3 pphr), the CNFC appears to be well dispersed within the EPDM matrix, with no visible signs of agglomeration. The uniform distribution suggests favorable processing conditions and good initial compatibility between the CNFC and the rubber phase. However, at higher CNFC loading levels (particularly 10 pphr), slight agglomeration of CNFC fibrils becomes apparent. This aggregation may lead to microstructural inhomogeneities, acting as stress concentrators and potentially compromising mechanical performance. Moreover, the interface between the CNFC and the EPDM matrix

appears to lack strong interfacial adhesion, as no significant signs of polymer-filler interpenetration or bonding are visible. This weak interface may hinder efficient stress transfer during mechanical loading, contributing to the observed reduction in tensile strength and elongation at break. These morphological observations are consistent with the mechanical property trends discussed earlier.

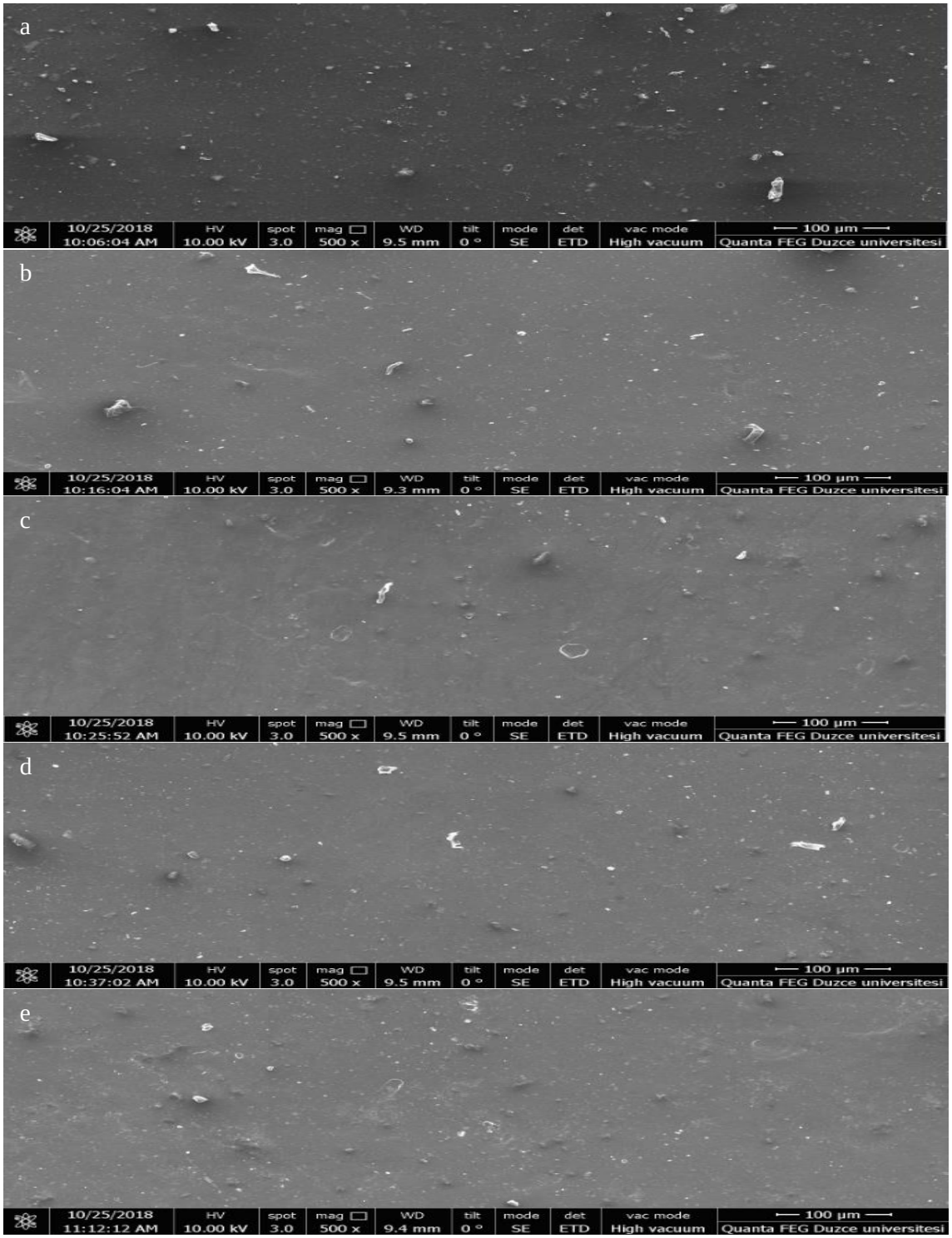


Figure 13. SEM images of the (a) EPDM plate, (b) EPDM-C1 plate, (c) EPDM-C3 plate, (d) EPDM-C5 plate and (e) EPDM-C10 plate

IV. CONCLUSION

In this study, biodegradable carboxylated nanofibrillated cellulose (CNFC) was incorporated into EPDM rubber to evaluate its potential as a sustainable alternative for automotive sealing profiles. CNFC addition influenced the mechanical, thermal, and aging behavior of the composites, while FTIR and SEM analyses confirmed no chemical degradation or phase separation within the EPDM matrix. Rheological results indicated that the composites could be processed under standard EPDM conditions. Increasing CNFC content led to reductions in tensile strength and elongation at break due to limited interfacial adhesion and restricted chain mobility. Nonetheless, mechanical properties remained within automotive specification limits up to 10 pphr. Tear strength exhibited a non-linear trend, with slight improvement at higher CNFC levels, likely due to filler agglomeration that may inhibit crack propagation. Thermal stability decreased with increasing CNFC due to the lower degradation onset of cellulose. Artificial weathering tests simulated short-term exposure (up to 250 h), equivalent to approximately three years of service life. Results showed that CNFC/EPDM composites maintained acceptable performance up to 3.0 pphr CNFC under these conditions. However, long-term durability beyond this period remains uncertain. In conclusion, CNFC offers a promising route for developing bio-based EPDM composites for short- to medium-term applications. For uses requiring limited service life, up to 10 pphr CNFC may be employed. For longer-term performance, CNFC loading should be restricted to 3 pphr or less. Future studies should focus on enhancing the aging resistance of CNFC-reinforced EPDM, such as through surface modification of nanofibers or the integration of hybrid filler systems.

DECLARATIONS

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Author Contributions: All parts of the study were conducted by the Author.

Conflict of Interest Disclosure: The author/authors declare no conflict of interest.

Funding/Supporting Organizations: This research received no external funding.

Ethical Approval and Participant Consent: This study does not involve human or animal participants. All procedures followed scientific and ethical principles, and all referenced studies are appropriately cited.

Plagiarism Statement: This article has been evaluated for plagiarism and no instances of plagiarism were detected.

Availability of Data and Materials: The availability of supporting data and materials should be clearly stated.

Use of AI Tools: The author/authors declare that no Artificial Intelligence (AI) tools were used in the creation of this article.

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