



# Characterization of E-glass/Epoxy Composite Modified with Recycled Carbon-Based Material and Multi-walled Carbon Nanotubes

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## Abstract

This study was investigated the effects of additives of Multi-Walled Carbon Nanotubes(MWCNTs) and recycled carbon-based material (rCBM) obtained by chemical degradation of waste tires (ELTs) on the quality of glass-fibre reinforced epoxy composite materials. The chemical degradation method was the novel method. This novel method that has been discovered was more advantageous than other methods (pyrolysis and devulcanization). The mechanical and characteristic properties of the composite materials were determined using rCBM and MWCNT additives of 0.1 to 2.0%. The study consisted of two phases, being the production of the composite materials and the determination of mechanical, morphological and functional properties of the materials produced. As a result, both the additives increased the strength of the composite material. It was demonstrated via SEM and FT-IR analyses that carbon-based additives obtained by degradation of waste tires failed to bond to epoxy resin in the production of the composite material.

**Keywords:** Composites, Epoxy, Recycled carbon-based material, Mechanical properties.

## Gerı Dönüştürülmüş Karbon Bazlı Malzeme ve Çoğul Duvarlı Karbon Nanotüpler ile Modifiye Edilmiş E-Cam/Epoksi Kompozitin Karakterizasyonu

### Öz

Bu çalışmada, atık lastiklerin kimyasal bozundurulması sonucu elde edilen geri dönüştürülmüş karbon bazlı malzeme (gKBM) ve Karbon nanotüp (CNT) katkı maddelerinin cam elyaf takviyeli epoksi kompozit malzemelerin kalitesine etkileri araştırılmıştır. Kimyasal bozunma yöntemi yeni bir yöntemdir ve bu yeni yöntem, diğer yöntemlerden (piroliz ve devulkanizasyon) daha avantajlıdır. Kompozit malzemelerin mekanik ve karakteristik özellikleri, % 0.1 - 2.0 arasında gKBM ve MWCNT katkıları kullanılarak belirlenmiştir. Çalışma, kompozit malzemelerin üretimi ve üretilen malzemelerin mekanik, morfolojik ve fonksiyonel özelliklerinin belirlenmesi olarak iki aşamada gerçekleştirilmiştir. Sonuç olarak her iki katkı maddesi kompozit malzemenin mukavemetini arttırmıştır. Kompozit malzeme üretiminde atık lastiklerin bozunması ile elde edilen karbon bazlı malzemenin epoksi reçineye bağlanmadığı SEM ve FT-IR analizleri ile gösterilmiştir.

**Anahtar Kelimeler:** Kompozitler, Epoksi, Geri dönüştürülmüş karbon bazlı malzeme, Mekanik özellikler.

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## 1. Introduction

Polymer-based composite materials thanks to the advantages provided by stiffness, hardness and strength-weight ratio today their use is widespread in many sectors such as aviation, automotive and medicine (Zhao et al. 2018). Epoxy resins are used as the matrix in composite materials formed by a mixture of two or more components. Epoxy resins are widely preferred in the production of composite materials due to their ease of processing, low cost and environmental advantages, as well as increasing engineering applications in recent years. However, since matrix-related properties often limit the application areas of composites, these materials must be enhanced with reinforcing elements (Soğancıoğlu et al. 2017). The superior mechanical properties of composites are provided by the transfer of loads from polymers to reinforcement materials with higher strength.

The geometric organization of the polymer matrix due to the homogeneous distribution of Carbon NanoTube (CNT) throughout the polymer is a key factor controlling the transfer of stress from the CNT to the polymer matrix (Zhao et al. 2018). The homogeneous distribution of CNTs throughout the polymer plays an important role in the charge transfer between the polymer matrix and the reinforcement element (Zhao et al. 2018). The reactions formed by CNTs with polymers at the molecular level are promising for charge transfer and improvement of mechanical properties (Pramanik et al. 2018). The use of CNTs in polymers is of great interest due to their excellent thermal, mechanical, structural and electrical properties. In particular, CNTs are described to have an excellent modulus of elasticity, thermal and electrical conductivity (Tarfaoui et al. 2016). The fracture toughness of CNTs reinforced composite plates increased by 8 to 11% (Seyhan vd. 2008). The addition of small volume CNT to the matrix of glass fiber reinforced composites significantly reduced the propagation rates of delamination cracks (Grimmer and Dharan 2010). Adding 0.5% CNT to the matrix of e-glass/epoxy composites increased the glass transition temperature (Warrier et al. 2010).

One of the types of CNTs is MWCNTs. The effect of MWCNTs on mechanical properties in layered composite materials was investigated. It was observed that MWCNTs positively affect the tensile strength of the composite material (Dindar and Bektas 2018). Sandwich structures were formed with MWCNTs modified composites and some metal sheets. Researchers investigated the effect of fiber orientation angles of MWCNTs modified composites of buckling. The amount of axially oriented fibers directly affected the buckling strength of the composite (Dindar and Bektas 2019). The low interlayer mechanical properties encountered in out-of-plane loading situations are known to be one of the major defects of glass fiber reinforced composites (GFRP) (Yildiz et al. 2019).

Material recovery methods of waste tires are pyrolysis, chemical degradation and devulcanization methods. Pyrolysis is the evaporation of waste tires (or other polymeric wastes) by heating at temperatures above 400 °C in an oxygen-free, inert or atmospheric environment. Pyrolysis char is obtained by applying activation and carbonization processes to the solid product obtained at the end of the pyrolysis method (Martínez et al. 2013). The chemical degradation method is a new waste tire recovery method that is more advantageous than the pyrolysis method since the chemical degradation method is carried out using chemical materials (H<sub>2</sub>SO<sub>4</sub>, NaOH, CH<sub>3</sub>OH, HCl, NaCl),  
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which are inexpensive and easy to find in the market, at a gelling time of 20 minutes in 140 °C ambient temperature under atmospheric pressure and a degradation time of 15 minutes under room conditions (Balbay, 2017; Balbay and Acikgoz, 2019).

Soğancıoğlu et al. (2017) produced epoxy composites by pyrolysis method using the pyrolysed carbon-based material (pCBM) they obtained from high density polyethylene (HDPE) and low-density polyethylene (LDPE) wastes. pCBMs obtained at different pyrolysis temperatures were mixed with 10-50% epoxy resin and determined the mechanical properties, tensile strength, surface hardness and electrical properties of the composite material. Epoxy composite materials produced with the addition of HDPE pCBM obtained at 300 °C (ER300) demonstrated the most ideal behaviour in terms of elongation in breaking and tensile strength. The breaking elongation and the tensile strength of the composites generally reduced at increased pyrolysis temperature and pCBM amounts. The hardness of the additive-free epoxy composite materials was determined as 79.8 Shore D, and the hardness of the composites produced with the addition of LDPE and HDPE chars were 86.3 Shore D and 87 Shore D, respectively. The study offers a new improvement approach for plastic wastes of HDPE and LDPE type. Composite materials produced as having some useful properties, such as electrical conductivity, hardness, tensile strength and flame retardation, will be made available in various fields and types (Soğancıoğlu et al. 2017a). In their other studies, the authors produced epoxy composite materials using pCBM they had obtained from PET plastic wastes washed by pyrolysis method at 300-700 °C. The electrical conductivity, tensile strength and surface hardness values increased as the amount of composite materials produced with the char sample obtained for all temperatures of the washed PET increased. It was observed that the epoxy composite material produced by PET and pCBM obtained at 700 °C, had the highest tensile strength and surface hardness. Therefore, it has been concluded that waste and washed PET plastics can be used as an additive in epoxy composites to obtain semi-conductor, high-strength and hard materials (Soğancıoğlu et al. 2017b).

Information in the literature on the use of pCBM obtained from polymeric waste by the pyrolysis method for production of composite materials is limited (Soğancıoğlu et al. 2017a). Moreover, information on the production of composite materials using rCBM obtained by chemical degradation (the novel method) that has been discovered is not available in the literature. This study was investigated the effects of MWCNTs and recycled carbon-based materials (rCBM) obtained by chemical degradation of waste tires on the quality of glass-fibre reinforced epoxy composite materials. Mechanical, functional and morphological properties of reinforced glass-fibre reinforced composite materials, containing MWCNTs at 0.1, 0.2, 0.3, 0.5, 1.0, 2.0% and reinforced with the addition of rCBM additive obtained by degradation of waste tires at 0.1, 0.2, 0.3, 0.5, 1.0, 2.0%, were determined in the study.

## 2. Material and Method

### 2.1 Material

The reinforcement elements used in this study were unidirectional (UD) glass fabrics (E-glass) weighing 300 g/m<sup>2</sup>. The epoxy resin formed matrix of the composite and curing agent was epikote 828 and epikure 825 purchased from Hexion Company, respectively. Recycled carbon-based material (rCBM=RP) were

produced by chemical degradation method (Balbay, 2017; Balbay and Acikgoz, 2019). Multi-walled carbon nanotubes (MWCNT) with 99% purity and outer diameter 10-20 nm, inner diameter 5-10 nm and length 0.5-2  $\mu\text{m}$ , were supplied from Switzerland.

## 2.2. Fabrication Process

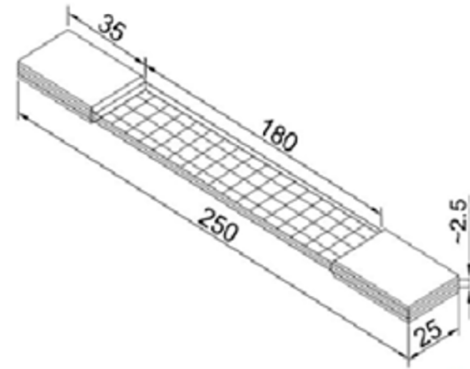
60% E-glass and 40% matrix were used in the production of the plates by weight. The production of the composite plates was carried out by İzoreel Kompozit İzole Malz. San. ve Tic. Ltd. Sti., which produces composite materials in İzmir-Turkey. Preparation phases of the plates included the preparation of E-glass, preparation of epoxy, preparation of composite layers, hot pressing of composite plates and cutting by water jet.

Composite plates were produced by hand lay-up method. This method was two stages. In the first stage, rCBM or MWCNT were weighed in the ratio of 0.1-0.2-0.3-0.5-1-2 %. Then, a mixture of resin/hardener (80-20%) recommended ratio by the company was prepared. The mixture was homogenized for 25 minutes by applying sound waves with a Hielscher UP400S ultrasonic mixing device. In the second stage, this mixture was applied on 30x30cm fabrics by lay-up technique. The fabrics were stacked in nine layers at [+90,-90] orientation angles and cured under 8 Bar pressure at 180 °C for 2 hours as shown in Fig. 1.



Fig. 1. Hot molding of composite plates.

All samples used in the experiments were cut from composite plates with a water jet of 25x250mm. Composite parts (tab) of 25x35mm were attached to the samples (Fig. 2). By making the cutting method with water jet, the cutting error that may occur in the samples and the amount of heat released were eliminated and an ideal cutting was achieved. At least three samples were tested and averaged in each test parameter. Devices used for preparing the composite plates were UP400S ultrasonic mixer of Hielscher brand, refrigerator of Laba brand, hot press designed and manufactured in İzoreel Company, and CNC water jet of ÜSJ 1220 model.



All dimensions are in mm.

Fig. 2. Technical drawing of test sample.

## 2.3 Mechanical Tests

The mechanical properties of the plates, prepared with rCBM and MWCNT additives, and of the additive-free plates were determined by tensile and buckling tests. The hardness measurements of composites, prepared with no additives and with optimum rCBM and MWCNT additives according to the results of the tensile and buckling tests, were determined by Tronic Hildebrand Shore D hardness measuring instrument.

The tests were performed in an Instron 8081 tension/compression testing machine of 50 kN capacity at Pamukkale University, Department of Engineering, Faculty of Mechanical Engineering, Department of Mechanics, and the elongation of the samples was measured by a video extensometer. The tensile speed was taken as 1 mm/sec. As a result of these measurements, the longitudinal elastic modulus (E1) and longitudinal tensile strength (Xt) parameters were automatically recorded and plotted by computer.

The samples with tensile tests completed were subjected to buckling tests in an Instron 8081 tension/compression testing machine of 50 kN capacity at Pamukkale University, Department of Engineering, Faculty of Mechanical Engineering, Department of Mechanics. The test samples were placed in the testing machine in sizes of 180 mm.

## 2.4 Characterization studies

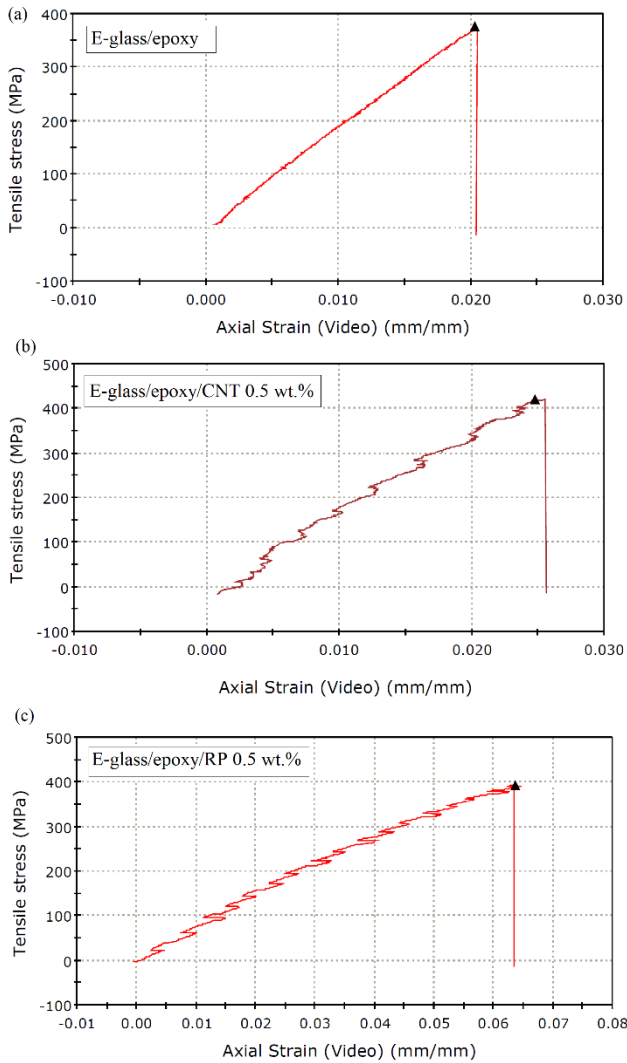
The functional groups and micro-structure of the plates, prepared with no additives, with optimum rCBM and MWCNT additives according to the results of the tensile and buckling tests, were determined in the characterization studies. Functional group analyses of the samples were performed by a Perkin Elmer Spectrum 100 Fourier Transform Infrared Spectrometer (FT-IR) at Bilecik Seyh Edebali University, Central Research and Application Laboratory. The analyses were performed at a wavelength scan interval of 400-4000  $\text{cm}^{-1}$ . The morphological structure of the samples was determined by SEM-ZEISS Supra 40VP scanning electron microscope at Bilecik Seyh Edebali University, Central Research and Application Laboratory.

## 3. Results and Discussion

### 3.1. Mechanical Properties

First of all, the mechanical properties of the samples were determined by performing tensile tests. The stress-strain graph obtained from the tensile device for the 0.5% MWCNT added

sample was given in Fig. 3. Fig 3 was given as an example for the data used in the preparation of Fig.4.



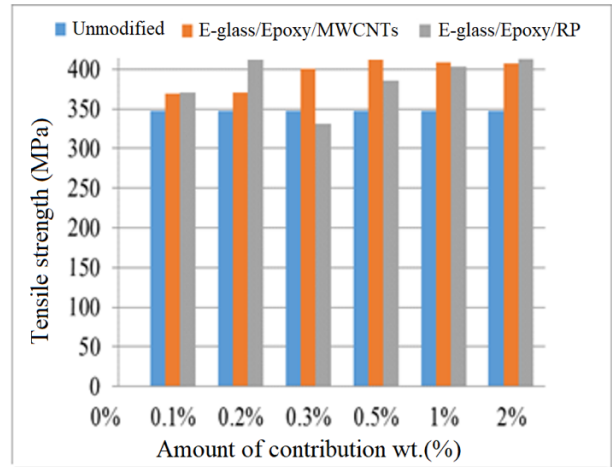
**Fig. 3.** Tensiles tress-strain curves of some samples; E-glass/epoxy (a), E-glass/epoxy/CNT 0.5 % wt. (b), E-glass/epoxy/rCBM 0.5 %wt. (c).

Tensile strength of composite materials according to their additive rates was given in Fig. 4. The tensile stress average of the additive-free sample was 347.28 MPa. The highest tensile-stress averages of the rCBM additive were found in samples with the values of 417.27 MPa and 412.16 MPa with the ratios of 0.2% and 2% by weight, respectively. The highest tensile-stress mean value of the CNT additive was 413 MPa for an additive ratio of 0.5% by weight.

It was considered based on the tensile test results that the CNT in the epoxy was created a negative effect because it reached saturation at 0.5% by weight and failed to bond after 0.5%. Moreover, it was been reported in the literature that CNT-additive epoxy composite materials can bond with epoxy resin to the saturation point, but cannot bond after the saturation point (Allaoui et al., 2002). These results supported the reports given in the literature.

The tensile test results of the waste tire-additive samples were not similar to the tensile test results of the CNT-additive samples, because the statically charged rCBM particles were not homogeneously distributed in the epoxy resin since they pushed

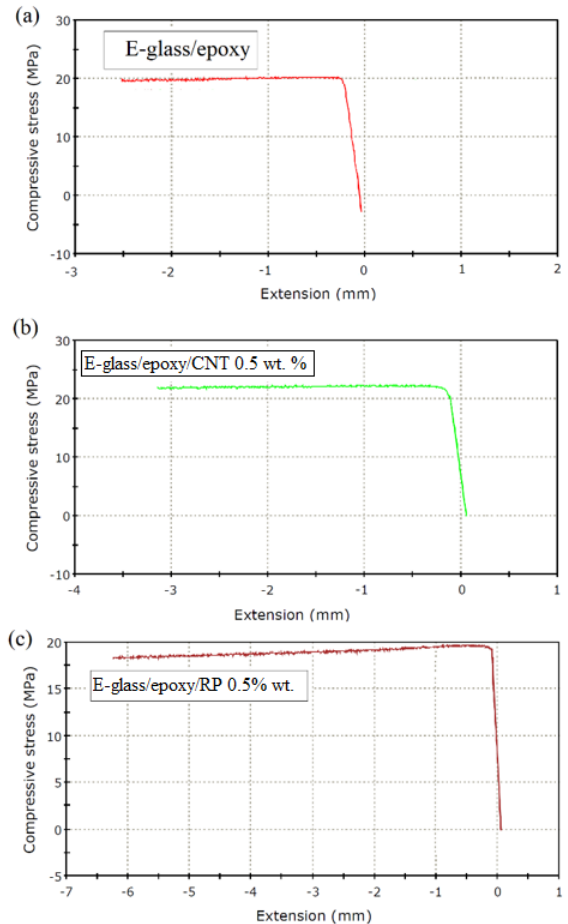
each other. Furthermore, the CNT particles in the epoxy were homogeneously distributed in the matrix. It was also believed that carbon develops in different physical properties during the chemical degradation of the waste tire and as a result, it was predicted that it was demonstrated a notch effect during the tensile test.



**Fig. 4.** Tensile strength of composites.

### 3.2. Buckling Behaviours

Fig. 5 showed the force-displacement graph of the E-glass/epoxy/0.5 MWCNTs additive sample. Fig 5 was given as an example for the data used in the preparation of Fig.6.



**Fig. 5.** Compressive sress-extension of some samples; E-glass/epoxy (a), E-glass/epoxy/CNT 0.5% wt. (b), E-glass/epoxy/rCBM 0.5 % wt. (c).

The critical buckling force ( $P_{cr}$ ) graph of the additive-free, CNT-additive and rCBM-additive plates were given in Figure 6. The breaking load for the additive-free samples was 1213.59 N. The highest critical buckling force value of the rCBM-additive was 1330.5 N for additive ratio of 2%. Furthermore, the values of 1269.1 N and 1251.71N, respectively, were found for rCBM-additive ratios of 1% and 0.2% and approximate critical buckling force values for these ratios. The highest critical buckling force value for CNT additive was 1828.7 N for additive ratio of 0.3%.

The critical buckling tensile value for the additive-free sample was 19.01 MPa. The highest critical buckling force value for waste tire additive was 22.91 MPa for additive ratio of 2%. Furthermore, the values of 20.71 MPa and 20.33 MPa, respectively, were found for CBM-additive ratios of 1% and 0.2% and approximate critical buckling stress values for these ratios. The highest critical buckling stress value for CNT additive was 27.79 MPa for additive ratio of 0.3%.

Cho and Daniel (2008) determined in their study that additives should be equally and evenly distributed into the resin serving as a matrix in the production of composite plates in order to strengthen the composite plates(Cho and Daniel, 2008). Appropriate surface adhesion between polymer and matrix directly affected the strength of the composite material. In the light of the results of the buckling test supporting this data, it was considered that the rCBM was not homogeneously distributed in the epoxy resin as in the results of the tensile tests and therefore, no bonding was achieved between the epoxy resin and the rCBM.

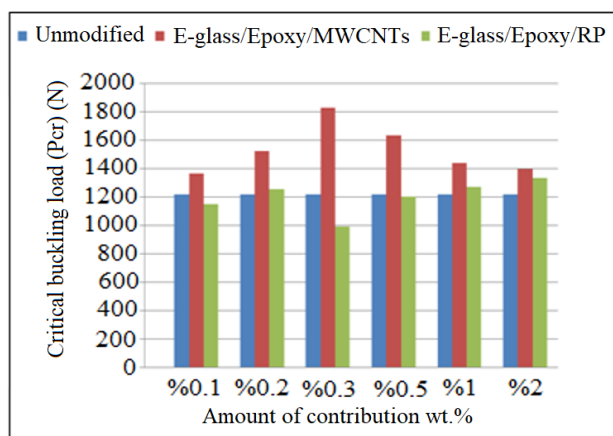


Fig. 6. Critical buckling strength of composites.

The additive ratio of 0.2% was determined as the optimum ratio for rCBM based on the tensile and buckling test results, and

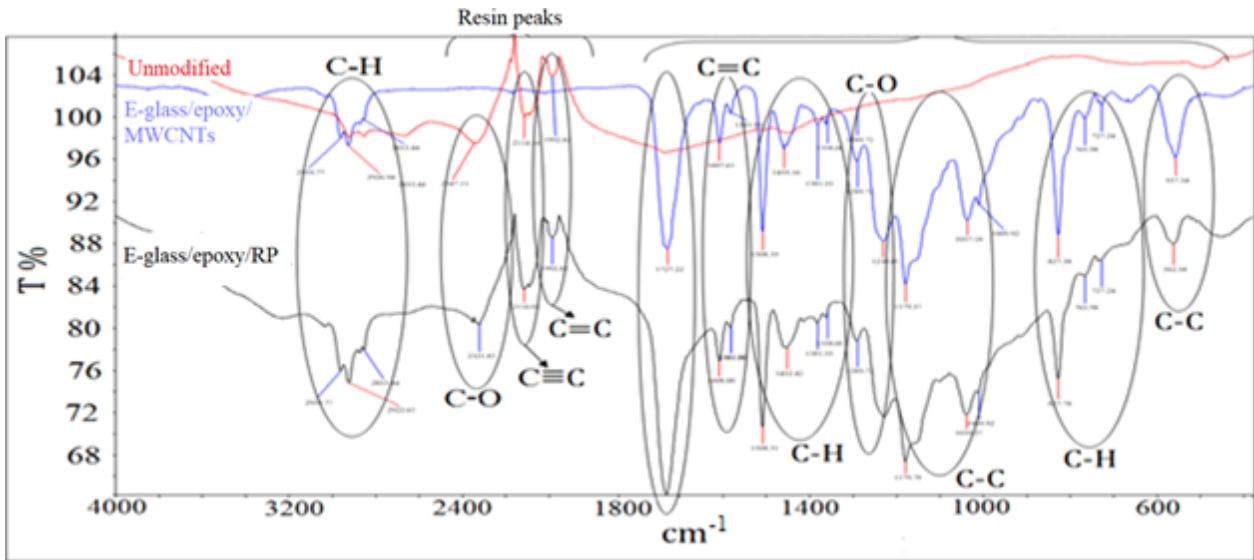
the additive ratio of 0.3% for CNT based on the buckling test results. Samples with the optimum additive ratios determine were subjected to hardness, FT-IR and SEM analyses.

The hardness of the additive-free epoxy composite material was 82 Shore D, and the hardness of the CNT-additive composite material were 86 Shore D and 85 Shore D, respectively. The hardness value of the char-additive composite material was the same as the hardness values (HDPE-87 Shore D and LDPE-86,3 Shore D) of the epoxy composite material produced by Sogancioğlu et al. (2017) using the pCBM obtained from HDPE and LDPE by pyrolysis method(Sogancioğlu et al. 2017a; Sogancioğlu et al. 2017b).

### 3.3. Fourier-transform Infrared Spectroscopy

The FT-IR graph of the functional groups of unmodified, 0.3% MWCNTs and 0.2% rCBM modified composite plates was given in Fig 7. Peaks of  $\sim 2300\text{ cm}^{-1}$  C-O (Li et al. 2002),  $\sim 2100\text{ cm}^{-1}$  C=C (McMurry, 2011) were determined in unmodified samples. E-glass/epoxy/RP modified samples peak points were  $3000\text{-}2800\text{ cm}^{-1}$  C-H (Hornback, 2005),  $\sim 1700\text{ cm}^{-1}$  C=O vibration peak (McMurry, 2011),  $\sim 1600\text{ cm}^{-1}$  C=C (McMurry, 2011),  $1500\text{-}1300\text{ cm}^{-1}$  C-H (Ning Y-C, 2011),  $1300\text{-}1000\text{ cm}^{-1}$  C-O (McMurry, 2011)  $1200\text{-}1000\text{ cm}^{-1}$  C-C (Sharma 1981),  $900\text{-}700\text{ cm}^{-1}$  C-H (McMurry, 2011)  $\sim 500\text{ cm}^{-1}$  C-C (Bergeron, 2012).

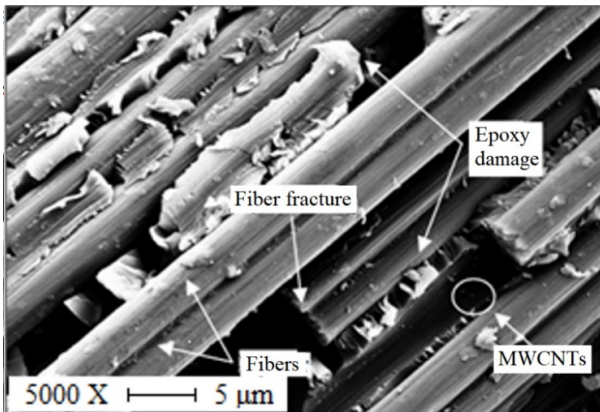
Peaks of E-glass/epoxy/MWCNTs in samples were  $3000\text{-}2800\text{ cm}^{-1}$  C-H (Derrick 2000),  $\sim 1700\text{ cm}^{-1}$  C-O (McMurry 2011),  $\sim 1600\text{ cm}^{-1}$  C=C (McMurry 2011),  $1500\text{-}1300\text{ cm}^{-1}$  C-H (Ning Y-C 2011),  $1300\text{-}1000\text{ cm}^{-1}$  C-O (Ning Y-C 2011),  $1200\text{-}1000\text{ cm}^{-1}$  C-C (Sharma 1981),  $900\text{-}700\text{ cm}^{-1}$  C-H (McMurry 2011),  $\sim 500\text{ cm}^{-1}$  C-C (Bergeron 2012). These results indicated that the MWCNT managed to bond to the epoxy resin in MWCNT-additive plate, but no bonding occurred between the rCBM and the epoxy resin.



**Fig.7.** The FT-IR spectrum of the functional groups of unmodified, 0.3% MWCNTs and 0.2% rCBM modified composite plates

### 3.4. SEM analysis

The morphological structure of the E-glass/epoxy/MWCNT 0.5% plate was given Fig. 8. SEM analysis showed that the MWCTs were distributed homogeneously. MWCNTs acted as a bridge between fiber/epoxy strengthening the interface bonds.



**Fig. 8.** SEM image of E-glass/epoxy/MWCNT 0.5%.

## 4. Conclusions and Recommendations

In recent years, studies on composite materials have increased due to the increased usage areas of composite materials. However, due to the high cost of materials used in composites, studies have accelerated to find new materials that have similar to these materials or better mechanical properties. This study examined the effects of the rCBM, obtained by chemical degradation of waste tires, which are of lower cost, on the mechanical, functional and morphological properties of the composite materials in order to serve as an alternative to MWCNT composites which are of high cost. Additive-free, MWCNT-additive and rCBM-additive in composite materials were compared in the tests performed. It was observed that the strength of the plates with additive was generally higher than the plates without additive. Furthermore, although they were both carbon-based, MWCNT managed to

bond to the epoxy resin, but no bonding occurred between the rCBM and the epoxy resin.

Accordingly, it has been determined that rCBM can be mixed with MWCNT at a maximum ratio of 0.2% in the production of epoxy composite materials. Moreover, it is thought that rCBM may be an alternative to MWCNT composites through changes in its internal structure by subjecting it to a series of chemical and physical processes.

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