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CHARACTERIZATION OF NANOCELLULOSE/PYROLYSIS OIL NANOCOMPOSITE FILMS

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ABSTRACT

In this study, the sustainable recycling of tire waste, which is frequently formed in the automotive industry, and the transformation of this recycling into valuable materials are in question. Waste tire pyrolysis oil obtained as a result of the pyrolysis of tire wastes was evaluated for the first time as a reinforcement element in nanocellulose-based nanocomposite films. Nanocellulose was produced using the TEMPO method (2,2,6,6-tetramethylpiperidine-1-oxyl radical). 5 %, 10 % and 20 % pyrolysis oil were added to the nanocomposite films. Thermal (thermal gravimetric analysis, differential scanning calorimetry, thermomechanical (dynamic mechanical thermal analysis and morphological (scanning electron microscopy) characterization of the produced nanocomposite films were performed. The highest thermal stability was observed in the nanocellulose/pyrolysis oil-20 sample with 20% pyrolysis oil additive. The pyrolysis oil-reinforced nanocomposites resulted in an excellent increase in storage and loss modulus. The storage modulus of the 20 % pyrolysis oil added sample at 100 °C was exactly 18 times that of pure nanocellulose. Nanocellulose-based nanocomposite films with superior thermal properties and structural compatibility demonstrated by characterized results have been shown to be pioneers in future industrial applications such as pharmacy, coating, green packaging.

Keywords: Nanocellulose, pyrolysis oil, recycling, sustainable materials, thermal analysis.

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INTRODUCTION

The use of lignocellulosic biomass for sustainable production is one of the promising raw materials. Lignocellulosic biomass can be used in the production of high performance, environmentally friendly and value-added industrial products (Zor *et al.* 2023). The abundant and renewable properties of lignocellulosic materials make lignocellulosic materials a critical material for the world economy (Iqbal *et al.* 2013). Cellulose nanomaterials have diameters less than 100 nm. They are only a few microns in length. Their structures contain both crystalline and amorphous regions (Pennells *et al.* 2020). Nanocellulose (NC) is a derivative of cellulose. Morphologically, it usually has rod, needle or spherical shapes. It can be obtained from many different sources (such as waste biomass, agricultural waste, wood). Due to its outstanding properties (biodegradability/compatibility, environmental friendliness, excellent surface charge, large surface area, non-toxicity, excellent mechanical properties), typically produced by acid hydrolysis, NC is used in many different fields such as defense, medicine, automotive, pharmaceutical, food packaging, energy finds a place (Miao and Hamad 2019, Kim *et al.* 2019, Phanthong *et al.* 2018).

The process of heating a raw material to temperatures higher than approximately 400 °C in an oxygen-free environment to bring a raw material to its volatile form and ultimately to produce oil, gas or coal is called pyrolysis. The oil obtained because of the pyrolysis process is known as pyrolysis oil (PO). PO has the potential to be used directly in fuels or in industry to produce chemicals. The PO obtained because of the pyrolysis of tire wastes contains many compounds. Limonene, polyaromatic hydrocarbons, benzene, toluene are just a few of these chemical compounds (Williams 2013, Kaminsky *et al.* 2009, Hu *et al.* 2014, Martínez *et al.* 2013, Dai *et al.* 2001, Alsaleh and Sattler 2014, Kebritchi *et al.* 2013, Roy *et al.* 1990, Williams and Bottrill 1995). The properties of PO vary depending on several factors (reactor type, rubber source, processing parameters and pyrolytic condition-mechanism) (Yousefi *et al.* 2000). When the studies are examined, PO finds a place for itself with the potential to be used as a fuel in the field of energy in general. Using PO directly as fuel is inefficient. Because PO has a low calorific value and is chemically unstable (Ighalo *et al.* 2021). Instead, PO can be used as an additive in composite materials. In the literature, very limited use of PO as reinforcement material has been reported. Prabhahar *et al.* (2022) applied bio-oil obtained from the pyrolysis of the baboon tree to natural fiber reinforced composite laminates. Composite laminates mixed with bio-oil and conventional epoxy resin exhibited a higher strength compared to pure epoxy resin-based composites. In another study, the reuse of resin pyrolytic oil was evaluated by producing pyrolytic oil-based composites. The obtained data proved that it is possible to manufacture high performance composites using pyrolytic oil (Guo *et al.* 2022). Verma *et al.* (2019) produced and characterized composites using epoxy resin with pyrolysis oil obtained from the pyrolysis of tire wastes. The results showed that composites with lower density and high tensile strength can be produced compared to pure epoxy and that the produced composites can be used in the automotive industry.

In this study, it was aimed to produce nanocellulose/pyrolysis oil (NCPO) nanocomposite films formed by combining the pyrolysis oil obtained as a result of the pyrolysis of waste tire wastes for recycling and nanocellulose, an environmentally friendly nanomaterial. Thermal gravimetric analysis (TGA), differential scanning calorimetry (DSC), dynamic mechanical thermal analysis (DMTA) and scanning electron microscope (SEM) characterization processes have been performed for the first time in the literature to determine the thermal and morphological properties of the produced nanocomposite films. Eliminating the deficiencies in the literature on the production and characterization of nanocellulose-based nanocomposites, recycling tire wastes into valuable products, and using the pyrolysis oil obtained from the pyrolysis of waste tires as a reinforcement in a composite material shows the originality of our study. In addition, we expand the possibilities of new generation nanocomposite material designs with the work done.

MATERIALS AND METHODS

Material

Nanocellulose was synthesized according to the method of Candan *et al.* (2016). The nanocellulose used in this study is bleached softwood kraft pulp. Nanocellulose was string-like particles with a diameter of approximately 20 nm and a length of 1 µm. The nanocellulose used in this study was obtained from bleached wood pulp using the oxidation mediator 2,2,6,6-tetramethyl-1-piperidin-1-oxyl (TEMPO method), which oxidizes some alcohol groups in the cellulose chain to carboxylic acids, and uses ionic repulsion to help separate the fibrils used.

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Preparation of NCPO nanocomposite films

Nanocellulose, pyrolysis oil, which is 5 %, 10 % and 20 % of the mass of nanocellulose, and distilled water were mixed. The resulting mixture was kept in an ultrasonic bath for 30 min. The obtained homogeneous mixture was grown by pouring into petri dishes and removing the water at 40 °C. It also prepared a control sample consisting of only nanocellulose. The contents of the nanocomposites are shown in Table 1 and the visual representing their preparation is shown in Figure 1.

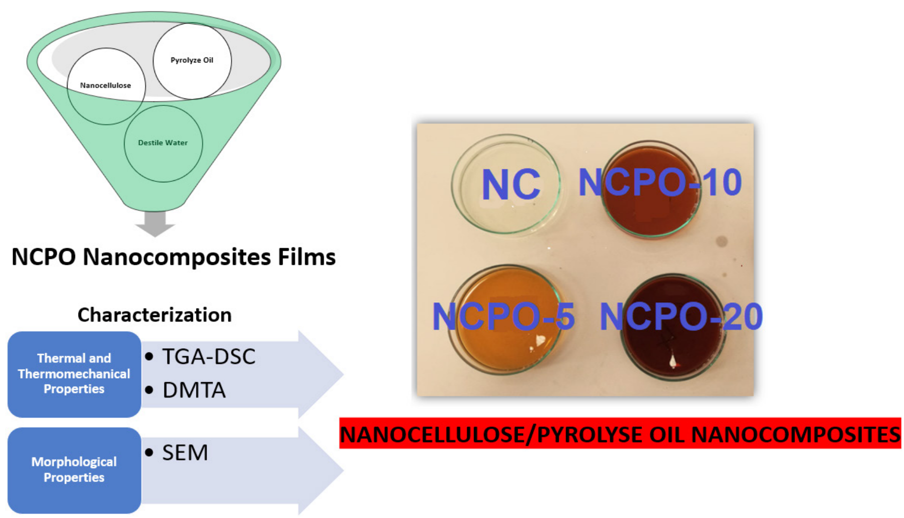

Figure 1: Design and manufacture of NCPO nanocomposite films.

Table 1: Formulation of NCPO nanocomposites.

Formulation	Nanocellulose (NC) (g)	Pyrolysis Oil (PO) (%)
NC	5	-
NCPO-5	5	5
NCPO-10	5	10
NCPO-20	5	20

Characterization

TGA, DSC and DMTA were used to determine the thermal properties of the nanocomposite films. TGA analysis, Şen *et al.* (2022) was performed under the conditions previously stated in the literature. DSC analysis was performed between 30 °C-350 °C and additionally in nitrogen atmosphere and with a heating rate of 10 °C/min. Determination of viscoelastic properties was carried out in the HITACHI device between 30-250 °C temperatures, 1 Hz frequency and 5 °C/min heating rate. The morphological structure of NCPO nanocomposite films was determined using SEM (TESCAN brand). Before analysis, the surface of the samples was coated with platinum.

RESULTS AND DISCUSSION

Thermal and viscolastic properties of the NCPO nanocomposites films

The thermal stability of NCPO nanocomposite films was investigated using TGA. The thermal degradation curve obtained because of the characterization is similar to the curve also called the cellulose pyrolysis curve in the literature. It is known that the cellulose pyrolysis curve is affected by chemical and physical effects, impurities, cellulose crystallinity, type of cellulose, the atmosphere measured, temperature and heating time (Radakisnin *et al.* 2020). When the thermogram for Figure 2 is examined, it is seen that all nanocomposite samples start to lose weight between temperatures between about 49 °C and 118 °C. In addition, the thermal degradation of the nanocellulose started at lower temperatures than obtained the nanocomposites. From this, it appears that pyrolysis oil needs higher temperatures for its thermal cracking. The losses at these temperatures are related to the moisture content of the nanocellulose (Priyadharshini *et al.* 2019). In the next region, while the volatile substances in the nanocellulose structure were removed at the weight loss, the pyrolysis oil began to decompose. When the literature is examined, the main degradation temperature of nanocellulose is around 200-300 °C, which shows that the results of our study are consistent (Gan *et al.* 2020). Table 2 shows the summary of data from TGA analyzes of nanocellulose-based nanocomposites. The main decomposition temperature of all samples is generally observed between 319 °C and 350 °C. The degradation at these temperatures is attributed to the thermal degradation of the long chains in the cellulose structure. The amount of ash remaining as a result of nanocellulose degradation is less than the amount of ash left in the nanocomposite samples. The reason for this situation is that nanocellulose decomposes more than pyrolysis oil and turns into a gaseous product. The addition of pyrolysis oil to the nanocellulose caused a partial increase in the degradation temperature. When all the results were examined, the highest thermal stability was observed in the NCPO-20 sample with 20 % pyrolysis oil.

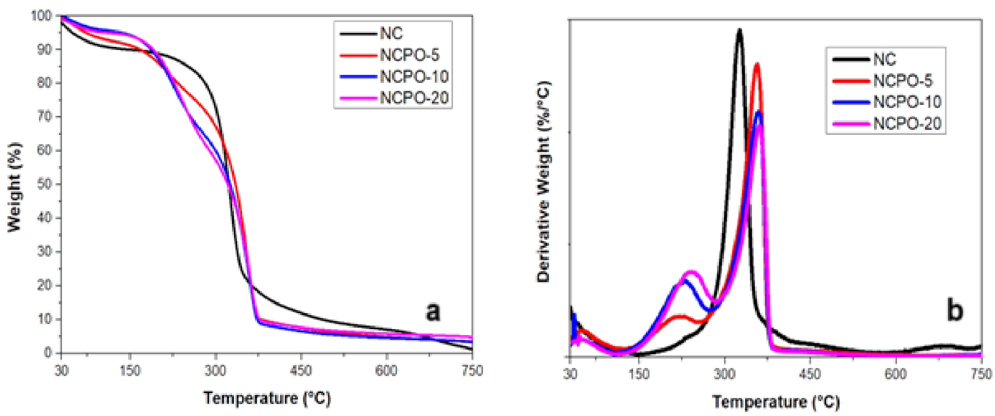

Figure 2: TGA curves (a) and DTG curves (b) of NCPO

The DSC curve of nanocomposite films reinforced with pyrolysis oil is shown in Figure 3. Two important endothermic peaks are observed around 80 °C and 350 °C. For the peaks observed around 80 °C, the increase in the amount of pyrolysis oil doped in the nanocellulose-based nanocomposite caused a partial shift in the endothermic peaks and an increase in temperature. Since pyrolysis oil is obtained using waste products, it is difficult to detect thermal transitions in produced PO-doped nanocomposites compared to other materials (Rajasekaran *et al.* 2021). Subsequent endothermic peaks observed around 350 °C indicate the degree of cellulose decomposition. Increasing the decomposition temperature has been associated with higher thermal stability, and this behavior can be attributed to a high degree of material crystallinity (Draman *et al.* 2014).

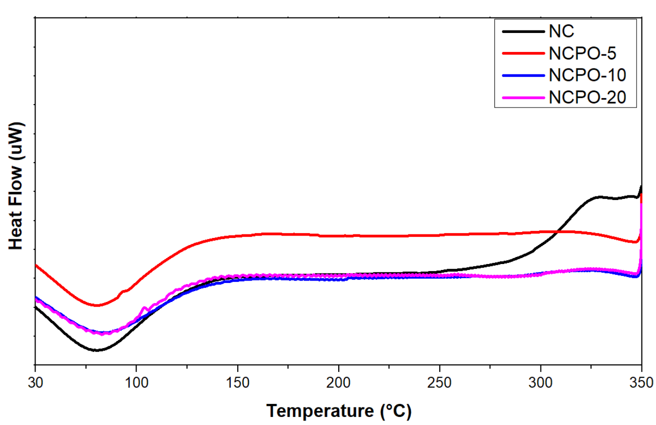

Figure 3: DSC chart of NCPOs.

Table 2: Thermal properties of NCPO nanocomposite films.

Sample	T_{50} % (°C)	Max. weight loss (°C)	Char yield (%)
NC	321	326	1,21
NCPO-5	334	357	2,22
NCPO-10	325	359	2,14
NCPO-20	319	361	3,87

DMTA is frequently used to determine the damping properties of polymer nanocomposites depending on temperature (Kumar *et al.* 2019, Soudmand *et al.* 2020). In this study, DMTA analysis was used to determine the rheological properties of NCPO nanocomposites. The variation of the storage module E' as a function of temperature is shown in Figure 4. For pure NC, the initial storage modulus E' was determined to be 0,05 GPa. The effect of pyrolysis oil added to the NC matrix on the storage modulus of nanocomposites is clearly seen in Figure 4. Increasing the amount of pyrolysis oil doped into the nanocomposite resulted in extraordinary increases in the storage modulus values. According to the initial storage modulus values, the highest storage modulus value was observed in the NCPO-20 sample with 0,79 GPa. Moreover, the initial storage modulus of the fabricated nanocomposites was determined as 2,8-fold for NCPO-5, 8,4-fold for NCPO-10, and approximately 20-fold for NCPO-20, in parallel with the increasing amount of pyrolysis oil compared to pure NC. Increasing the temperature from the initial temperature up to 100 °C did not decrease the storage modulus values of the nanocomposite samples. On the contrary, increasing PO amount and temperature resulted in excellent increases in storage modulus values. When all nanocomposite samples were examined, the highest storage modulus was in the NCPO-20 sample at approximately 100 °C 1,48 GPa. Additionally, at around 100 °C, the storage modulus of NCPO-20 was exactly 18 times that of pure NC. A similar situation was observed in the loss modulus (E'') values of nanocomposites. As shown in Figure 5, as with the Storage modulus, temperature and increasing PO amount caused an increase in the loss modulus values. Karabork and Tipirdamaz (2016) investigated its properties by adding pyrolysis oil obtained from waste tires to the mixture of natural and synthetic rubber. DMTA analysis results showed an increase in elastic behavior with increasing PO content. They attributed this increase to increased crosslinking density. Tan δ the loss coefficient is known as a measure of the damping of the material (Cho and Park 2011). Figure 6 shows the tan δ of NCPO nanocomposites as a function of temperature. The tan δ values of all NCPO varied between approximately 0,04 and 0,12. When the loss coefficient in the prepared nanocomposites is compared with pure NC, a general decrease in Tan δ peaks is observed. The tan δ peak height value for NCPO-10 nanocomposite has a minimum value compared to other prepared nanocomposites. The decrease in tan δ peak height indicates that the molecular chain structure of the reinforced PO is hindered by the additives (Thomas *et al.* 2021, Boonbumrung *et al.* 2016). The storage modulus and loss modulus values of nanocellulose-based nanocomposites are summarized in Table 3 and Table 4.

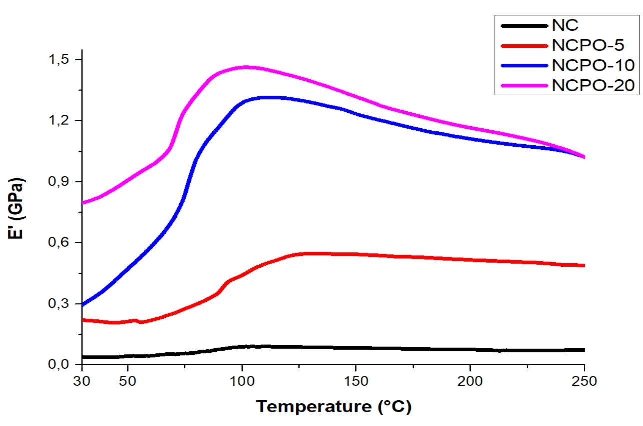

Figure 4: Storage modulus (E') plot of nanocellulose-based nanocomposites.

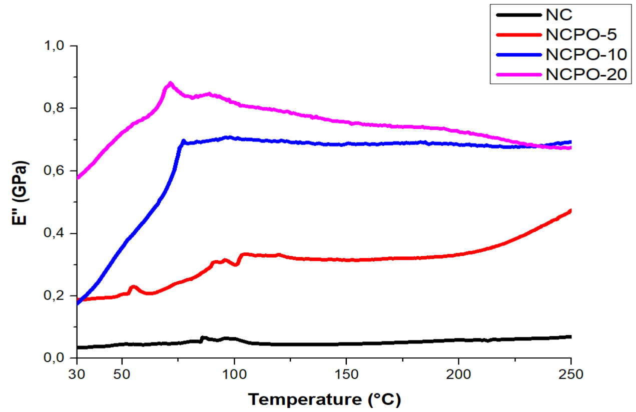

Figure 5: Loss modulus (E'') plot of NCPO nanocomposites.

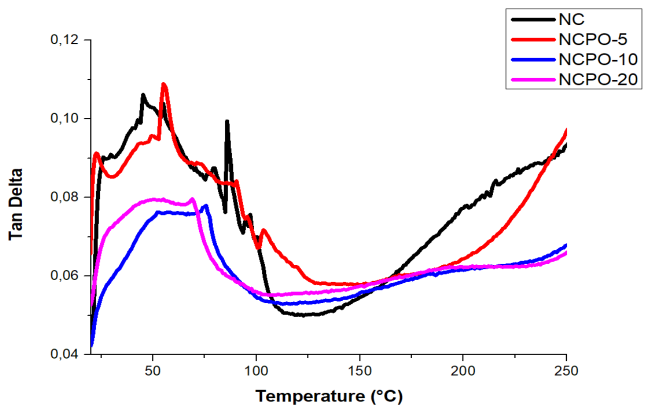

Figure 6: Tan δ of NCPO nanocomposites.

Table 3: Storage modulus and viscous-rheological properties of NCPO nanocomposite films

Sample	Initial Storage Modulus 30 °C	E'(GPa) 50 °C	E'(GPa) 100 °C	E'(GPa) 200 °C	E'(GPa) Final Temperature
NC	0,05	0,04	0,08	0,06	0,07
NCPO-5	0,22	0,21	0,44	0,52	0,48
NCPO-10	0,30	0,47	1,28	1,10	1,02
NCPO-20	0,79	0,88	1,48	1,18	1,07

Table 4: Loss modulus and viscous-rheological properties of NCPO nanocomposite films.

Sample	**Initial Storage Modulus 30 °C**	**E''(GPa) 50 °C**	**E''(GPa) 100°C**	**E''(GPa) 200°C**	**E''(GPa) Final Temperature**
NC	0,03	0,04	0,06	0,05	0,07
NCPO-5	0,18	0,21	0,31	0,33	0,45
NCPO-10	0,16	0,35	0,70	0,69	0,69
NCPO-20	0,57	0,72	0,82	0,74	0,67

The results acquired from the DMTA analysis revealed that the storage modulus values of the reinforced nanocomposites were higher than that of the control group. All the storage modulus values of the nanocomposite films or control group increases while the temperature increases from 30 °C to around 125 °C. It might be attributed to water removal from the nanocellulose-based materials (Candan et al. 2016, Poyraz et al. 2023). Previous studies done by Candan, Poyraz indicate that lignocellulosic materials have both cell wall water (bound water) and capillary water (free water) (Thybring et al. 2022). When it moves from the structure, the materials are going to be mor stiff. When the temperature increased from around 100 °C to 250 °C, the storage modulus values of all the composites decreased. The reduction rate increases while the PO loading ratio increases.

Morphology of the NCPO nanocomposites films

This analysis was performed to determine the effect of nanocellulose on the morphological surface of the nanocomposite films, the presence of PO and its appearance in the structure. SEM analysis is important in determining the morphological properties of nanocomposite films (Syafiq *et al.* 2020). Figure 7, different magnifications for (a) NCPO-5 with 5 % PO, (b) NCPO-10 with 10 % PO, and (c) NCPO-20 with 20 % PO at different magnifications (x 5000, x 10000, x 20000, and x 50000) shows SEM micrographs of NCPO nanocomposite films. All SEM images clearly show the effect of TEMPO-oxidized cellulose on NCPO nanocomposite films. Strong repulsive forces affect the nanofibrils within the cellulose cell wall. Increasing the ionic strength in its environment weakens the effects of the repulsive forces. This results in a reduction in fiber width. On the contrary, the repulsive forces become stronger as the ionic strength decreases. The strengthening of the repulsive forces causes the cell walls to separate from each other. This separation results in an increase in lateral dimension (Levanic *et al.* 2020, Isogai *et al.* 2011, Fall *et al.* 2011). The driving force effect mentioned here may explain the images observed in the morphological structure of the NCPO films produced. SEM photographs consist of dense pyrolysis oil dispersed in a nanocellulose matrix. The PO is mixed homogeneously throughout the matrix. The homogeneous mixture is the reason for the strong interfacial bond between the PO and the nanocellulose matrix. Increasing the percentage of pyrolysis oil resulted in the bleached area in the images. In their study, Syafiq *et al.* (2020) showed the scattering of light by the effect of cavities as the cause of this whitening. Moreover, they reported that the mechanism of the formation of cavities may be due to the mixing method by cavitation of oil. In general, it can be said that increasing the amount of pyrolysis oil reinforced into the nanocomposite leads to a decrease in the pore concentration and size.

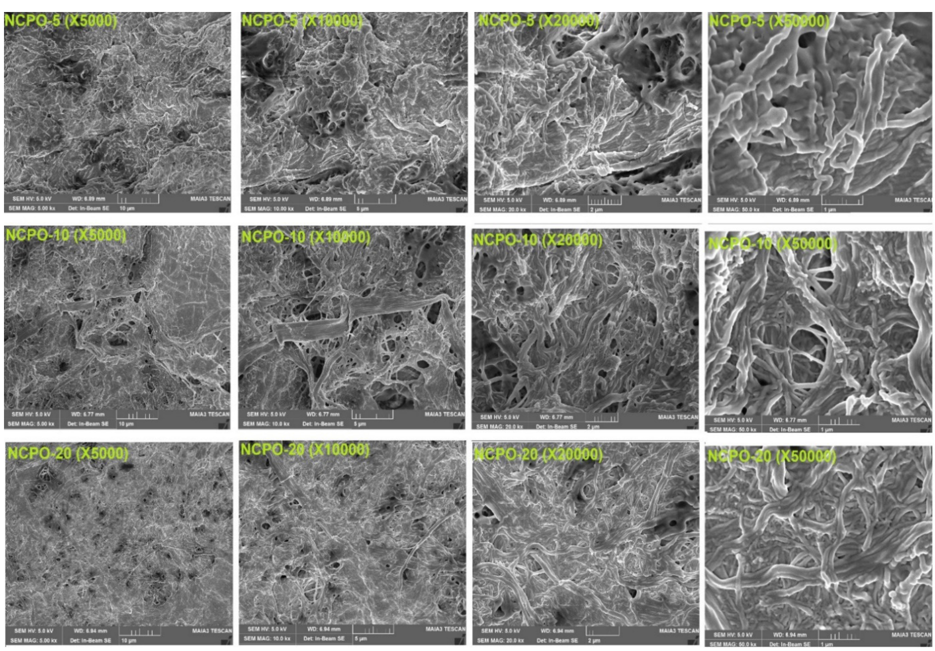

Figure 7: Morphological structure of NCPO nanocomposites.

CONCLUSIONS

Waste management is among the most important issues of the modern age. The study focuses on the sustainable recycling of tire wastes, which are frequently encountered in the automotive industry, and the transformation of this recycling into valuable materials. Waste tire pyrolysis oil obtained because of the pyrolysis of tire wastes was evaluated as a reinforcement element in the composite material. For this, nanocellulose was used and new nanocomposite films based on nanocellulose were produced. The thermal and morphological characterization processes of the produced pyrolysis oil reinforced nanocellulose based nanocomposite films confirmed the existence of the obtained composites. The addition of pyrolysis oil to the nanocellulose matrix resulted in a significant increase in the thermal stability and viscoelastic properties of the nanocomposite films. The data obtained because of the thermal analysis were supported by the SEM analysis data.

This study also sheds light on the characterization processes of nanocellulose-based nanocomposites. Considering all the above results, the superior thermal and structural characterization of the produced NCPO nanocomposite films reveals the importance of a recyclable green world. In order to make the pyrolysis process of waste tires more economical, it can be recommended to develop refining processes and process them to produce high value-added products. Especially in the pyrolysis of wastes, different factors can be focused on the conditions of the production process.

Authorship contributions

M. Z.: Investigation, methodology, writing – original draft. E. K.: Investigation, visualization, writing – original draft. F. Ş.: Investigation, data curation, writing – original draft. B. O.: Resources, investigation, writing – original draft. Z. C.: Funding acquisition, project administration, writing – review & editing.

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