

NANOCOMPOSITES BASED ON WOOD FLOUR,  
ETHYL SILICATE AND VARIOUS NANOPARTICLES*Tamara Tatrishvili<sup>1,2,✉</sup>, Nikolozi Kvinikadze<sup>1,2</sup>, Tinatini Bukia<sup>2,3</sup>*<sup>1</sup> Ivane Javakhishvili Tbilisi State University, Department of Macromolecular Chemistry. 1 I. Chavchavadze Ave., Tbilisi 0179, Georgia<sup>2</sup> Institute of Macromolecular Chemistry and Polymeric Materials, Ivane Javakhishvili Tbilisi State University, 13 University St., Tbilisi 0186, Georgia<sup>3</sup> Vladimir Chavchanidze Institute of Cybernetics of the Georgian Technical University. 5 Z. Andjaparidze St., Tbilisi 0186, Georgia✉ *tamar.tatrishvili@tsu.ge*

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**Abstract.** This study provides a comprehensive review of contemporary research and characterization techniques for nanocomposites, along with a thorough analysis of the latest trends in this field. Nanocomposites represent a novel category of material, characterized by the presence of fillers with a nanoscale dimension (graphene 5-25 nm, nano silica 70-90 nm). These materials possess considerable potential for application in diverse industrial sectors, including the automotive, aerospace, construction, electrical, and food packaging domains. There is a substantial interest in the utilization of nanoparticles, such as graphene and nano-silica fillers, in the development of innovative natural composites. The possibility of obtaining environmentally friendly nanocomposite materials with improved properties based on renewable natural raw materials (wood flour), which are easy to obtain and inexpensive, as well as nanoparticles as important fillers in polymer composites, is demonstrated.

A range of composite materials has been developed, based on wood flour, with varying dispersion qualities, and with different percentage contents of binder (3-20%), ethyl silicate (40%), as well as nanofillers, including graphene and nano-silica particles. It has been demonstrated that the addition of nanoparticles enhances the mechanical properties and overall performance of the composites.

In order to identify the composition of the nanocomposites, a series of Fourier transform infrared spectroscopic studies was carried out. The physical-mechanical properties and water absorption of the compositions were studied, and surface morphology was investigated using the optical microscopic method. In addition, thermogravimetric analysis methods were used to observe the thermal properties of the materials.

**Keywords:** wood flour, nanocomposites; fillers, nano-silica, graphene, mechanical and thermal properties.

## Introduction

Polymer (nano)composites have been extensively studied over a long period of time. They are generally organic polymer composites, mostly filled with inorganic fillers. Their properties combine the advantages of the inorganic filler material (*i.e.*, rigidity, thermal stability) and of the organic polymer (*i.e.*, flexibility, ductility, processability). However, the main advantage of these composites is characterized by the volume fraction and size of the fillers. If the fillers decrease in size from the micro- to the nanoscopic scale, unique properties of polymer nanocomposites are emphasized as the small size of the fillers leads to a dramatic increase in the interfacial area as compared with ordinary composites.<sup>1,2</sup>

Polymer nanocomposites consist of polymer matrices that contain organic/inorganic fillers having a uniform nanoscale distribution. The size of the nanofiller normally ranges from 10 to 100 nm in at least one dimension.<sup>3</sup> A large number of polymeric materials possess good dielectric properties in addition to stable physical and chemical properties. These properties decide the applicability of polymers in various fields. The electrical, mechanical, and other operational properties of polymers can be altered *via* the modification of polymeric materials with various inorganic fillers. The change in specific properties depends upon the nature, morphology, composition concentration of filler, and flow process for filler introduction into the matrix.

In the fast-growing field of nanotechnology, polymer nanocomposites have become a prominent area of current research and development. Silica/polymer nanocomposites are dominating the polymer and composite literature as well as a variety of applications, many industrial products, and other significant areas of current and emerging interest.<sup>4</sup>

Among the numerous polymer composites, silica/polymer nanocomposites are the most commonly

reported in the literature and are also employed in a variety of applications, such as electronics, automotive, and aerospace industries, as well as used in many industrial products due to their good mechanical characteristics.<sup>5</sup>

Silica (SiO<sub>2</sub>) is one of the most promising inorganic nanofillers, and it has been widely used in the preparation of polymer nanocomposites due to its adjustable morphology, large specific surface area, and ease of functionalization. The surface modification, structural characteristics, and bonding mechanisms of SiO<sub>2</sub> are crucial in enhancing the overall performance of polymer nanocomposites.<sup>6</sup>

The discovery of graphene with its combination of extraordinary physical properties and ability to be dispersed in various polymer matrices has created a new class of polymer nanocomposites.<sup>7-9</sup> Recently, graphene, a two-dimensional, layered carbon nanomaterial, has gained the attention of researchers working on nanomaterials due to its outstanding mechanical, thermal, and electrical properties. When incorporated into polymer matrices, graphene serves as a versatile filler, resulting in the development of high-performance nanocomposites with a variety of applications.<sup>10</sup>

The objective of this work is to provide a comprehensive overview of the predominant and most widely employed techniques and strategies for the preparation of ecologically sustainable nanocomposites utilising diverse nanofillers. This is followed by a concise discussion of the primary characterisation methods, mechanical testing, properties, and applications. This study examines how surface modification affects the material's morphology, thermal properties, and other characteristics.

Further properties are listed below.

## 2. Experimental

### 2.1. Materials

We manufactured new nanocomposites on the basis of wood flour using tetraethyl orthosilicate (TEOS), formally named tetraethoxysilane Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub> as a binder and nanofillers (graphene, nano-silica) with different degrees of silylation (3-20%). In addition, aluminum hydroxide was used as a fire retardant. Al(OH)<sub>3</sub> was selected because it is known to be harmless to the environment and has no effect on the human body; it can react with the binder used to make composites, releasing methanol or forming hydrogen bonds. The components were procured from Aldrich and utilised in their original state or following purification, prior to utilisation.<sup>11,12</sup>

The solvents were purified using standard methods before being used: toluene was distilled from sodium.

The wood flour used in the composite materials was purchased online. Prior to use, the wood flour was air-dried at a low temperature (48 hours at 323–343 K) using a dryer.

### 2.2. Processing

The composites were prepared using a hot-pressing method of highly dispersed dry wood flour, with the utilisation of ethyl silicate as a binding agent, in conjunction with a variety of nanoparticles. This subject is currently being researched.

The focus of this study pertains to nanocomposites, which were fabricated under constant pressure of 15 MPa at temperatures ranging from 413 to 493 K.<sup>13</sup>

### 2.3. Preparation of Samples

Two different types of samples were prepared: cylindrical (d = 1.5 cm, h = 2 cm) and parallelepipedal ones (12 cm in length, 0.7 cm in height, and 1.5 cm in width). The following properties were determined: strength on bending, impact viscosity, and thermal stability.<sup>14</sup>

### 2.4. Characterization

**Fourier transform infrared spectroscopy (FTIR)** studies were conducted on a Nicolet TM iS50 FTIR Spectrometer-Thermo Fisher Scientific in the infrared region of 4000–400 cm<sup>-1</sup> (scan 32, resolution 4 cm<sup>-1</sup>); band intensities were denominated in transmittance.

**Optical microscopic** examinations of composite materials were performed on an OMAX-type polarised microscope equipped with a high-resolution digital camera A35140U3 14 MP.

**Water absorption** was determined by subjecting the specimens to immersion in a water bath maintained at the ambient temperature. The measurement process was conducted at 3-hour and 24-hour intervals.

**Water absorption** (WA, in percent) is calculated by the formula:  $WA (\%) = \frac{W_1 - W_0}{W_0} \times 100$  where W<sub>0</sub> and W<sub>1</sub> are the weights of the sample before and after immersion.

**The bending test** (the experimental procedure, also referred to as flexural testing) was conducted with the objective of quantifying the flexural stiffness and strength properties of polymer matrix composites. The study was conducted in accordance with the established protocol. The following investigation focuses on parallelepipeds with a length of 10 cm. In addition, a vertical element is required. The square cross-section is characterised by a numerical value of 1 cm<sup>2</sup>.

Each specimen was positioned on two prisms, with a distance of 8.0 cm between them. The indenter was a metal cylinder with a diameter of 10 mm, which was applied from above to the midpoint of the specimen. Bending strength, otherwise referred to as flexural strength, is defined as the tensile force required to induce a fracture (*i.e.* a crack) in the outer surface of the test specimen.<sup>15</sup>

**Impact viscosity investigation,** also the phenomenon has been termed "shock". The viscosity test is a technique that is applied to soft materials.<sup>15,16</sup> The drop height ( $h$ ) is defined as the vertical distance between the upper surface of the material that was tested ( $h_1$ ) and the bottom surface of the drop hammer at the end of the impact event ( $h_2$ ). It can be demonstrated that, in order to calculate the work performed by the falling hammer, the mass of the hammer ( $m$ ) and the acceleration due to gravity ( $g$ ) must be considered.

The work performed is then equal to  $mg(h_1 - h_2)$ , with  $h_1$  and  $h_2$  being the heights of the hammer before and after it falls, respectively. This calculation is then normalised with regard to the horizontal cross-section of the specimen.

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**Thermogravimetric investigations (TGA)** were conducted on a Netzsch Instruments analyser (model TG 209 F1 Taurus). The apparatus measures mass change as a function of temperature, with an operational temperature range of 10 to 1100°C. During the testing process, an increase in temperature was observed at a rate of approximately 10°C per minute in an open area.

### 3. Results and Discussion

Technological processes have been optimised to obtain materials with the desired chemical, thermal, and other physical properties. New composites have been obtained using dry wood sawdust (flour) and some organic-inorganic binders (separate and combined) under conditions of temperatures ranging from 413 to 493 K and pressures of 15 MPa.<sup>17,18</sup>

A comparative analysis has been undertaken to explore the potential of replacing formaldehyde products in wood-based panels with non-toxic alternatives, excluding the use of phenol, melamine, urea, or carbamide-formaldehyde pitches, to enhance the affordability and accessibility of the composite materials.

#### 3.1. Probable chemical reactions

From scientific-technical literature, it is known that wood mainly contains sufficient amounts of starch, cellulose, hemicellulose, pectic substances, lignin, and

polyphenols. All the above-mentioned compounds may react with the proposed binders. For example, ethylsilicate (TEOS-40), containing an ethoxy group, participates in the etherification reaction with wood through the macromolecular and intramolecular reactions. Processes that occur during the curing are complex and varied.<sup>19-21</sup>

In solution, the degree of polymerization of silicate anions is known to depend on two factors - the silica modulus and the solution concentration. Each solution has a distribution of anion polymerization degree. Distribution is superimposed on the polymer distribution of anions on the charges, which is also determined by these two factors.

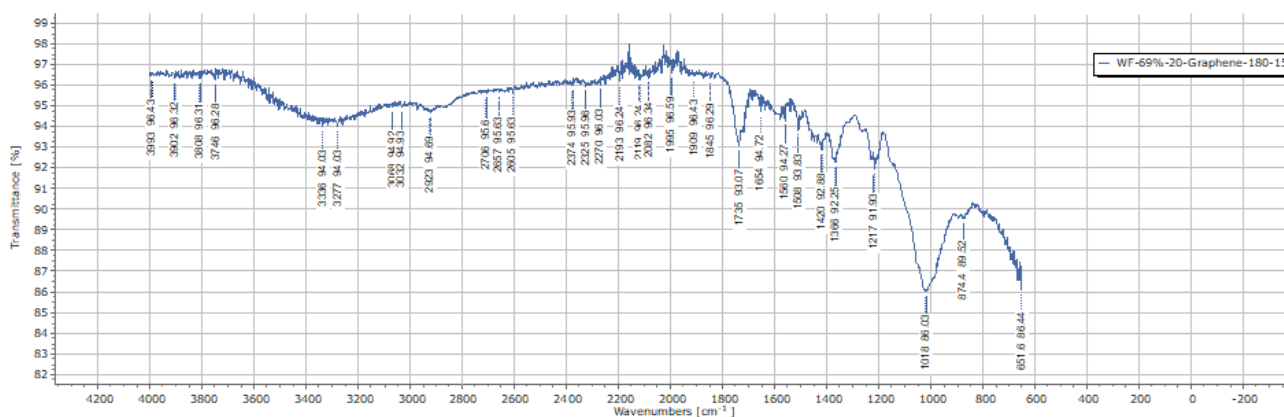
It is possible that in the intermediate stages, the reactions between of  $\equiv\text{Si-OH}$  band contained cellulose molecules, which take place with dehydration reactions. These reactions go through obtaining three-dimensional structures. Formation of such structures also takes place with the use of hardener  $\text{Na}_2\text{SiF}_6$ , which accelerates the processes.

With the use of TEOS binder, the condensation reaction of ethoxy groups with hydroxyl groups is also possible according to the scheme:  $-\text{OH} + \text{C}_2\text{H}_5\text{O-Si} \equiv \rightarrow -\text{O-Si} \equiv + \text{C}_2\text{H}_5\text{OH}$ , which may proceed further with obtaining three-dimensional systems.

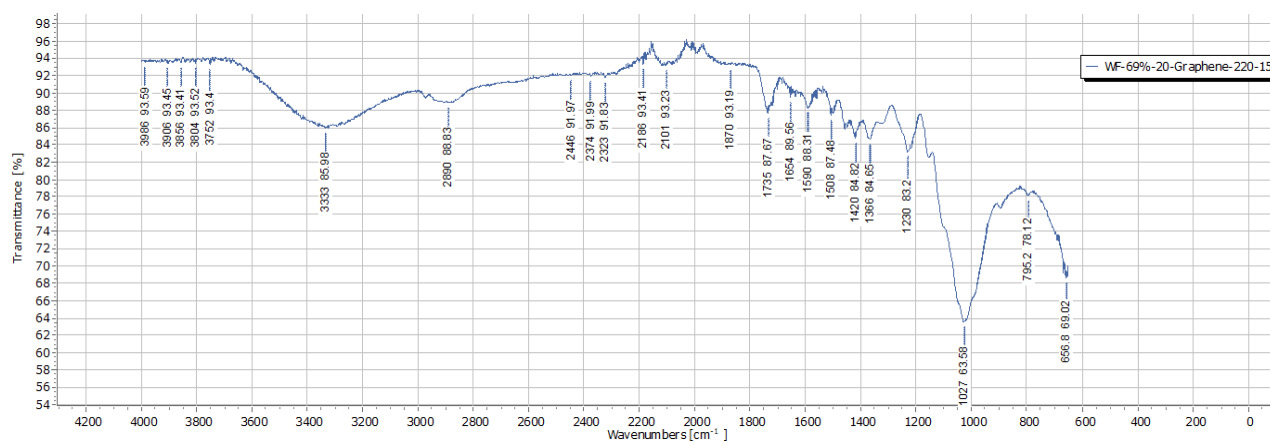
#### 3.2. Fourier transform infrared spectroscopy (FTIR)

The objective of this study is twofold: firstly, to assess the structural changes in the composite materials, and secondly, to clarify the differences in the properties mentioned above. The obtained composites were subjected to Fourier transform infrared (FTIR) spectroscopy investigations.<sup>22-25</sup> These investigations enabled the determination of the formation of new bonds in the composites. Some of the FTIR spectra are illustrated in Fig. 1 (a, b).

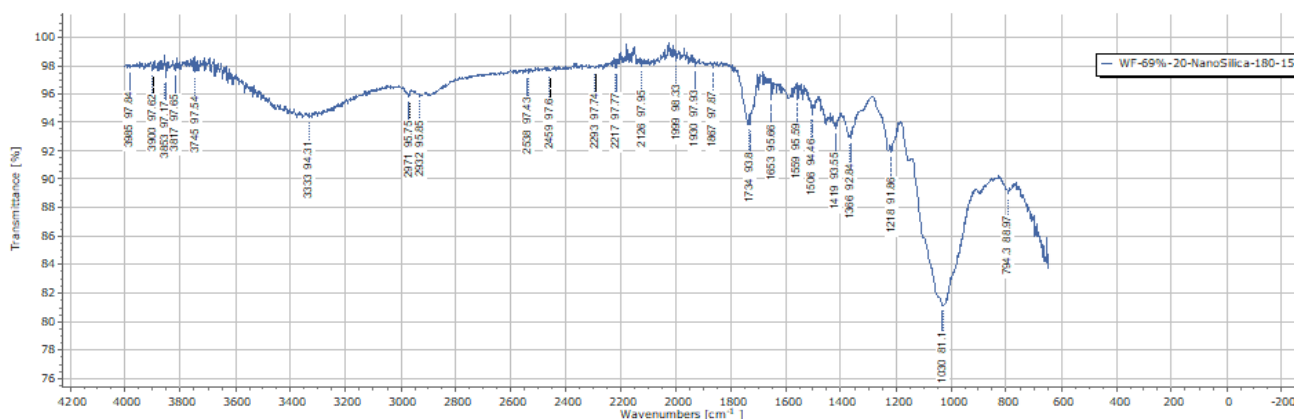
FTIR spectroscopy was performed to characterize a composite material composed of the following components (Fig. 1a,b): wood flour (69%), TEOS (tetraethyl orthosilicate) (20%), graphene (1%), and  $\text{Al}(\text{OH})_3$  (10%). Functional groups expected to be observed in this composite include OH groups from wood flour and  $\text{Al}(\text{OH})_3$ , which are anticipated to appear as broad peaks in the range of 3277 to 3336  $\text{cm}^{-1}$ . Si-O bonds from the TEOS component are expected to appear around 1018 and 1027  $\text{cm}^{-1}$ . The  $\text{CH}_2$  and  $\text{CH}_3$  bond peaks from TEOS are observed at 2923 and 2890  $\text{cm}^{-1}$ , and graphene-related peaks, such as C-H bending or stretching, are typically observed around 2900  $\text{cm}^{-1}$  or near 1508  $\text{cm}^{-1}$ , depending on the interaction between graphene and the matrix.



a) Wood Flour -69%, TEOS -20%, Graphene -1%, T- 453 K, P-15MPa

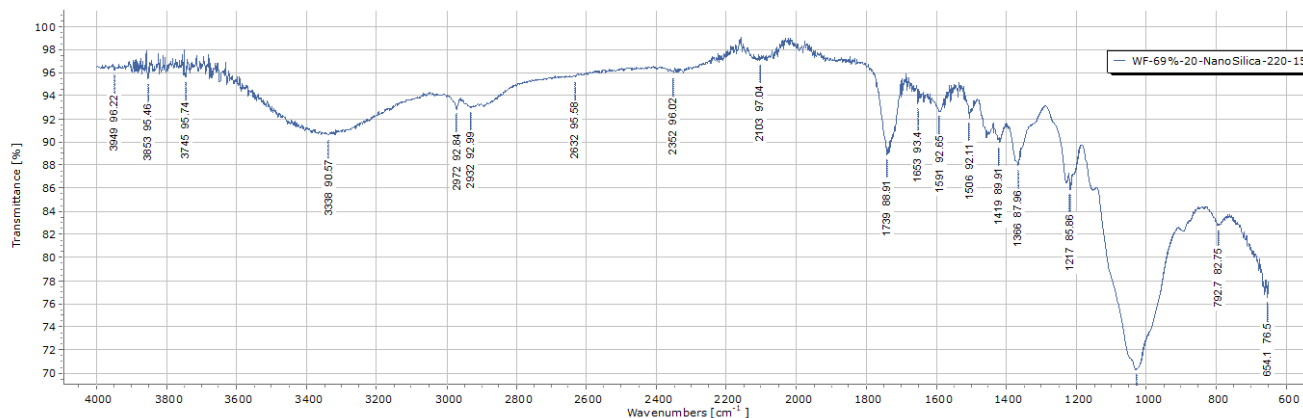


b) Wood Flour -69%, TEOS -20%, Graphene -1%, T- 493 K, P-15MPa

**Fig. 1.** FTIR spectra of composites XXIX (a) and XXX (b) based on Ethyl silicate, Wood Flour and Graphene

a) Wood Flour -69%, TEOS -20%, NanoSilica -1%, T- 453 K, P-15MPa

**Fig. 2.** FTIR spectra of composites XXIX (a) and XXX



b) Wood Flour -69%, TEOS -20%, Nano silica -1%, T- 493 K, P-15MPa

**Fig. 2.** (Continuation) FTIR spectra of composites XXIX (b) based on Ethyl silicate, Wood Flour and Nano silica

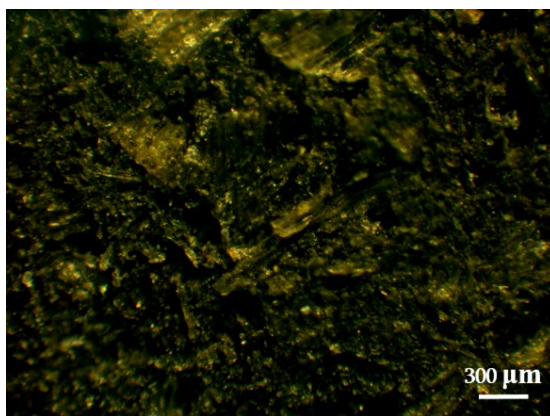
The FTIR spectra of the composite materials, as shown in Figs. 3 and 4, consist of TEOS (20%),  $\text{Al}(\text{OH})_3$  (10%), wood flour (69%), and nano-silica (1%), and reveal the following characteristic features. The O-H stretch from wood flour and the hydroxyl groups in  $\text{Al}(\text{OH})_3$  typically appear as broad peaks between  $3338 \text{ cm}^{-1}$  and  $3333 \text{ cm}^{-1}$ , attributed to hydrogen bonding in the hydroxyl groups of cellulose and hemicellulose in the wood flour. The C-H stretching (alkyl groups) peaks appear between  $2971 \text{ cm}^{-1}$  and  $2932 \text{ cm}^{-1}$  due to the ethyl groups ( $-\text{C}_2\text{H}_5$ ) in TEOS. The C-O stretch is observed at  $1218 \text{ cm}^{-1}$ , corresponding to the cellulose and hemicellulose structures. The Si-O stretching, a characteristic peak, is observed around  $1030$ – $1032 \text{ cm}^{-1}$ , corresponding to the Si-O bond in TEOS. Si-O-C bending typically appears in the  $850 \text{ cm}^{-1}$  region. The Al-O stretch may appear around  $620 \text{ cm}^{-1}$ , representing the bending vibration of  $\text{Al}(\text{OH})_3$ . Si-O-Si stretching from nano-silica is identified by a sharp peak around  $1030 \text{ cm}^{-1}$ . Additional smaller peaks may appear in the lower wavenumber regions (below  $794 \text{ cm}^{-1}$  and  $792 \text{ cm}^{-1}$ ) due

to specific interactions between the components, such as Si-O-C bending from TEOS or Si-O-Si bending from nano-silica.

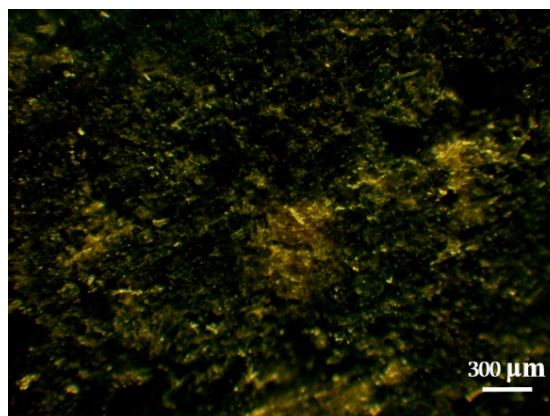
### 3.3. Microstructural Characterisation

The microstructure of the compact samples has been studied by applying optical microscopy analysis. It offers powerful quality control as well as an important analytical tool.<sup>26</sup> Optical microscopy investigation enables us to understand the relationship between the structure and properties of materials and to develop new materials with desired properties.

We have observed the transverse surfaces, the longitudinal ones, or the inner parts of the sample and the inclusions under magnifications of 50, 100, 200, and 500 times. As demonstrated by the optical microscope images, the binder exhibits an upward tendency in relation to the surfaces. The alteration in temperature does not exert an effect on the dimensions of the inclusions or cleavages.



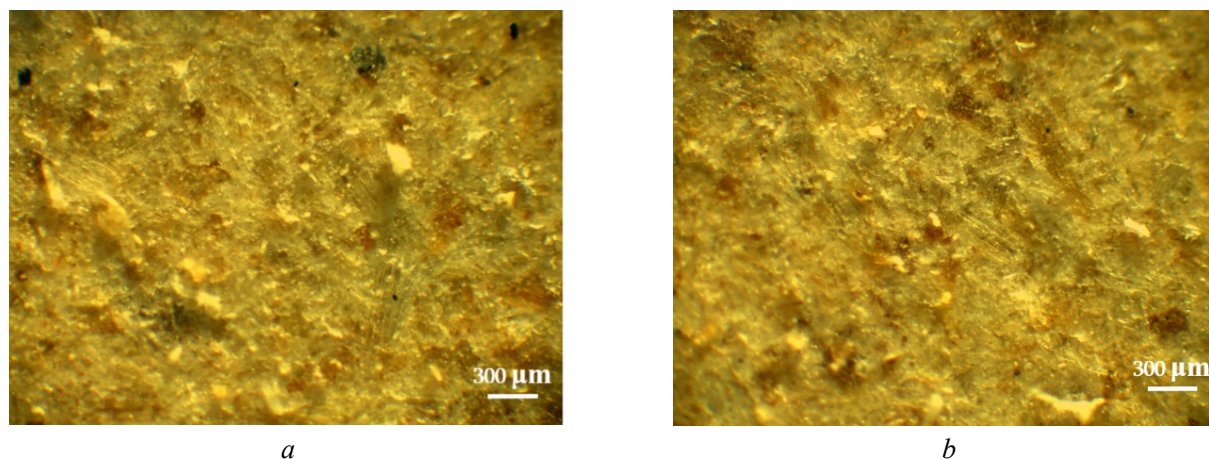
a



b

**Fig 3.** Optical micrographs of samples XIV (a) and XV (b)





**Fig 4.** Optical micrographs of samples XIX (a) and XXX (b)

In Figs. 3 (a, b) and 4 (a, b), we display some of the micrographs for WNC (Wood Nano Composites). One notices the fibrous character of supramolecular structures. Comparing Figs. 3 and 4, we see how 1 % of graphene dramatically changes color. A fairly uniform distribution of components is seen.

The optical microscopic data of the composite materials under study show two components, which have different colors for different inserts. Inserts do not have a clearly defined border. The surface is almost homogeneous. Definition of the sizes of inserts or cracks one established with the use of the scale shown in pictures.<sup>27-29</sup> When comparing Fig. 3 (a, b) and Fig. 4 (a, b), we can see a similarity in colour for composites. According to the figures, we do not observe significant differences among the samples obtained at the different temperatures.

### 3.4. Physical – Mechanical Properties

Mechanics of materials is the study of a material's response to a physical stressor. Generally, this is assumed to pertain to the study of how materials fail.<sup>30,31</sup> Mechanical testing provides important information about the physical

behavior of a specimen. In general, these tests are conducted *via* the application of force, which causes deformation (*i.e.*, a change in size or shape of the specimen), which is then measured. Alternatively, deformation can be applied, and force can be measured. The parameters of the mathematical equations relating force and deformation are obtained and can be utilized for direct statistical comparisons between groups (*e.g.*, to identify the impact of a disease or disorder) and as inputs to computational models, giving researchers the capability to simulate situations that are difficult to recreate in the laboratory. Finally, as mentioned above, these parameters can be utilized to select the appropriate material for a specific application.<sup>32</sup>

Among physical-mechanical properties, the strengthening of bending and impact viscosity was investigated. The experimental data are presented in Table 1.

Bending tests generally involve the measurement of the ductility of a sample material. Bending tests may include taking the sample material to a specific limit and determining the load measurement and its relationship to a load specification.

**Table 1.** Dependence of the values of bending strength and impact viscosity of composites containing Wood flour on the conditions (temperature, pressure, binder concentration) of sample preparation

#	Composite, %	Temperature, T, K	Pressure, MPa	Bending strength, MPa	Impact viscosity, kJ/m <sup>2</sup>
1	2	3	4	5	6
I	TEOS 3% + Wood flour 86% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	27.673	20.657
II	TEOS 3% + Wood flour 86% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	33.686	19.330
III	TEOS 3%+ Wood flour 86% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	35.468	17.512
IV	TEOS 5% + Wood flour 84% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	23.901	21.849
V	TEOS 5% + Wood flour 84% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	28.075	15.228
VI	TEOS 5% + Wood flour 84% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	36.084	15.159

**Table 1.** (Continuation). Dependence of the values of bending strength and impact viscosity of composites containing Wood flour on the conditions (temperature, pressure, binder concentration) of sample preparation

1	2	3	4	5	6
VII	TEOS 10% + Wood flour 79% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	34.296	24.169
VIII	TEOS 10% + Wood flour 79% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	38.410	23.292
IX	TEOS 10% + Wood flour 79% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	47.724	16.957
X	TEOS 15% + Wood flour 74% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	25.530	15.391
XI	TEOS 15% + Wood flour 74% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	33.851	14.667
XII	TEOS 15% + Wood flour 74% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	49.132	15.741
XIII	TEOS 20% + Wood flour 69% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	36.464	19.512
XIV	TEOS 20% + Wood flour 69% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	37.034	14.249
XV	TEOS 20% + Wood flour 69% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	58.463	20.918
XVI	TEOS 3% + Wood flour 86% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	21.988	21.567
XVII	TEOS 3% + Wood flour 86% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	26.340	19.713
XVIII	TEOS 3% + Wood flour 86% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	33.819	17.226
XIX	TEOS 5% + Wood flour 84% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	29.133	19.512
XX	TEOS 5% + Wood flour 84% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	37.227	19.817
XXI	TEOS 5% + Wood flour 84% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	41.199	20.971
XXII	TEOS 10% + Wood flour 79% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	28.344	21.435
XXIII	TEOS 10% + Wood flour 79% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	42.214	22.774
XXIV	TEOS 10% + Wood flour 79% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	58.432	18.804
XXV	TEOS 15% + Wood flour 74% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	37.861	16.904
XXVI	TEOS 15% + Wood flour 74% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	43.408	13.055
XXVII	TEOS 15% + Wood flour 74% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	50.543	12.397
XXVIII	TEOS 20% + Wood flour 69% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	39.193	19.179
XXIX	TEOS 20% + Wood flour 69% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	51.707	17.778
XXX	TEOS 20% + Wood flour 69% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	66.455	17.958

We studied nanocomposites using both nanofillers: graphene and nano-silica. According to the data presented in Table 1, it can be observed that the concentration of composite ingredients and reaction conditions have a notable impact on the parameter of strengthening at bending and impact viscosity. The results, however, are not entirely consistent. In general, it can be seen that increasing temperature and binder concentration and using nanoparticles tend to improve the mechanical properties of WNC, except for some data.

At high temperatures, heterogeneous esterification reactions and donor-acceptor interactions occur between the active groups of ethyl silicate molecules and functional groups of wood, increasing the spatial concentration of chemical bonds. The minimal value for bending strength is 21.988 at TEOS 3% + Wood flour 86% + NanoSilica 1% + Al(OH)<sub>3</sub> 10% and 23.901 at TEOS 5% + Wood flour 84% + Graphene 1% + Al(OH)<sub>3</sub> 10%, for both at 413 K, and 15 MPa. Maximum data is 66.455 at TEOS 20% + Wood flour 69% + NanoSilica 1% + Al(OH)<sub>3</sub> 10%, 58.463 for both at 493 K and 15 MPa.

The impact viscosity investigation was conducted. This parameter is of critical importance in the context of exploitation, particularly during instantaneous loading processes. The findings of this study demonstrate the clear relationship between the tested variables and the impact viscosity of the composites. As demonstrated in Table 1, an increase in binder concentration and pressing temperature has been shown to result in an increase in the impact viscosity of the composites. However, in the series of composites with the same composition, increasing temperature causes a decrease in the impact viscosity values. The primary reason for these discrepancies in dependencies observed in the two studies is attributed to the extent of inhomogeneity in the distribution of binders within the composites.

In the case of high binder content, the binders themselves form clusters, which has the potential to weaken the mechanical properties of the composite. However, the impact of pressure is found to be non-significant.<sup>33-35</sup>

Water absorption is a critical consideration for wood composites, given their frequent utilisation in high-humidity environments. An experiment was conducted in which composite samples were immersed in water. The results of this study demonstrated a substantial decrease in the tensile strength of the samples following the immersion process. This decline was further substantiated through a morphological study, which provided additional insights into the structural alterations caused by the water interaction.<sup>14</sup>

Lignocellulosic fibers have poor resistance to moisture absorption, and due to this, natural fiber-reinforced composites have undesirable effects on dimensional stability and mechanical properties when exposed to moisture in the environment. In order to understand the durability of composites based on the field of application, it is necessary to study the moisture absorption behaviour of natural fiber composites. In general, water molecules penetrate natural fiber composites

by three different mechanisms: diffusion of moisture content within the micro-gaps between the polymer chains; capillary transport into the micro-gaps; and flaws in the interfaces between the fibers and the matrix.<sup>36–40</sup> The moisture is mainly absorbed by the interface between the fiber and the matrix, as well as by the fiber itself *via* hydrogen bonding.

The composite samples for water absorption measurements were cut into 10 × 10 mm squares with a thickness of 1 mm. The samples were dried in a vacuum oven at 50 °C for 24 h, then cooled in a desiccator and weighed. A mass balance with a precision of ± 1 mg was used to measure the sample weight. The percentage of water absorption ( $W_a$ ) at the time (t) was calculated using Eq. (1), where  $W_d$  and  $W_n$  are the original dried sample weight and weight of the sample after absorbing water, respectively.<sup>14</sup>

$$W_a = \frac{W_n - W_d}{W_d} \times 100\% \quad (1)$$

**Table 2.** The investigation into the water absorption properties of nanocomposites based on wood flour, TEOS and nanoparticles has been completed

#	Composite, %	Temperature, K	Pressure, MPa	Mass, g	Volume, cm <sup>3</sup>	Density, g/cm <sup>3</sup>	Mass after 3 h-s of exposure in water (g)	Mass after 24 h-s of exposure in water (g)	Water absorption after 3h exposure in water in wt.%	Water absorption after 24h exposure in water in wt.%
1	2	3	4	5	6	7	8	9	10	11
I	TEOS 3% + Wood flour 86% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	3.550	2.399	1.480	3.681	4.675	3.69	31.69
II	TEOS 3% + Wood flour 86% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	3.205	2.155	1.487	3.287	3.693	2.56	15.23
III	TEOS 3% + Wood flour 86% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	3.317	2.289	1.449	3.406	3.597	2.68	8.44
IV	TEOS 5% + Wood flour 84% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	3.255	2.225	1.463	3.377	4.245	3.75	30.41
V	TEOS 5% + Wood flour 84% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	3.628	2.490	1.457	3.715	3.870	2.4	6.67
VI	TEOS 5% + Wood flour 84% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	3.937	2.607	1.510	4.003	4.171	1.68	5.94



**Table 2.** (Continuation). The investigation into the water absorption properties of nanocomposites based on wood flour, TEOS and nanoparticles has been completed

1	2	3	4	5	6	7	8	9	10	11
VII	TEOS 10% + Wood flour 79% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	3.742	2.575	1.453	3.865	4.784	3.29	27.85
VIII	TEOS 10% + Wood flour 79% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	3.913	2.623	1.492	3.988	4.17	1.92	6.57
IX	TEOS 10% + Wood flour 79% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	3.772	2.6	1.451	3.812	3.968	2.12	5.20
X	TEOS 15% + Wood flour 74% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	3.440	2.342	1.469	3.551	3.967	3.23	15.32
XI	TEOS 15% + Wood flour 74% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	3.694	2.528	1.461	3.773	3.919	2.14	6.09
XII	TEOS 15% + Wood flour 74% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	3.869	2.618	1.478	3.929	4.001	1.55	3.41
XIII	TEOS 20% + Wood flour 69% + Graphene 1% + Al(OH) <sub>3</sub> 10%	413	15	3.642	2.478	1.47	3.743	4.172	2.77	14.55
XIV	TEOS 20% + Wood flour 69% + Graphene 1% + Al(OH) <sub>3</sub> 10%	453	15	3.118	2.144	1.454	3.196	3.323	2.5	6.57
XV	TEOS 20% + Wood flour 69% + Graphene 1% + Al(OH) <sub>3</sub> 10%	493	15	3.875	2.586	1.498	3.925	3.973	1.29	2.53
XVI	TEOS 3% + Wood flour 86% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	3.379	2.291	1.475	3.520	4.299	4.17	27.23
XVII	TEOS 3% + Wood flour 86% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	3.257	2.254	1.445	3.407	3.826	4.61	17.47
XVIII	TEOS 3% + Wood flour 86% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	3.236	2.277	1.421	3.375	3.610	4.3	11.56
XIX	TEOS 5% + Wood flour 84% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	3.591	2.441	1.471	3.718	4.227	3.54	17.71
XX	TEOS 5% + Wood flour 84% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	3.456	2.342	1.476	3.550	3.776	2.72	9.26
XXI	TEOS 5% + Wood flour 84% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	3.395	2.363	1.437	3.410	3.620	0.44	6.33
XXII	TEOS 10% + Wood flour 79% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	3.721	2.526	1.473	3.826	4.364	2.82	17.28
XXIII	TEOS 10% + Wood flour 79% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	3.713	2.512	1.478	3.806	3.974	2.5	7.03
XXIV	TEOS 10% + Wood flour 79% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	3.554	2.459	1.445	3.642	3.728	2.48	4.9
XXV	TEOS 15% + Wood flour 74% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	3.886	2.632	1.476	3.980	4.416	2.42	13.64

**Table 2.** (Continuation). The investigation into the water absorption properties of nanocomposites based on wood flour, TEOS and nanoparticles has been completed

1	2	3	4	5	6	7	8	9	10	11
XXVI	TEOS 15% + Wood flour 74% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	3.676	2.515	1.462	3.753	3.870	2.09	5.28
XXVII	TEOS 15% + Wood flour 74% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	3.838	2.568	1.495	3.913	4.034	1.95	5.11
XXVIII	TEOS 20% + Wood flour 69% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	413	15	3.932	2.614	1.504	4.024	4.283	2.34	8.93
XXIX	TEOS 20 % + Wood flour 69% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	453	15	3.863	2.561	1.508	3.934	4.053	1.84	4.92
XXX	TEOS 20% + Wood flour 69% + NanoSilica 1% + Al(OH) <sub>3</sub> 10%	493	15	3.725	2.540	1.467	3.798	3.897	1.96	4.62

The results of the corresponding experiments are presented in Table 2. As seen from the Table, with the increase in pressing temperature at the same pressure, the density of composites slightly increases with some exceptions, and the water absorption decreases. The magnitude of water absorption is contingent on the quantity of ingredients in the composite and the conditions under which it is obtained.

Two kinds of nanoparticles were utilised in the study. The superior performance of graphene in comparison to nano silica is evident. The phenomenon under discussion can be explained by reference to the size of the nanoparticles. The best result is observed for composites XV (TEOS 20% + Wood flour 69% + Graphene 1% + Al(OH)<sub>3</sub> 10%) and XXX (TEOS 20% + Wood flour 69% + Nanosilica 1% + Al(OH)<sub>3</sub> 10%) at the temperature 493 K and pressure 15 MPa. The obtained results indicate that the properties of the composites have been enhanced.

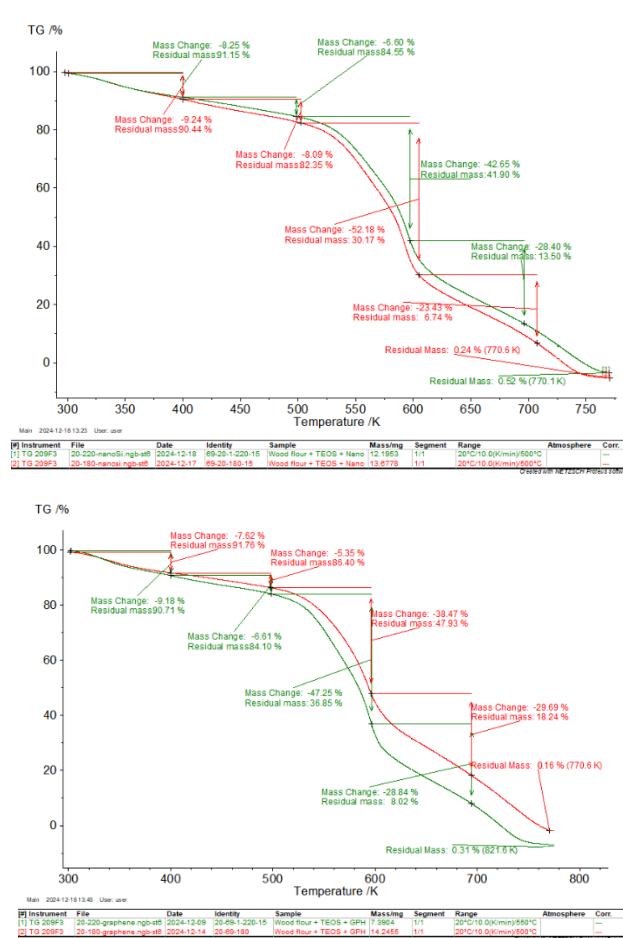
### 3.6. Thermogravimetric Analysis (TGA)

Thermal analysis can be defined as a test used to evaluate chemical, physical, and structural changes in a material due to a temperature change. In principle, temperature is a fundamental state variable that affects most chemical reactions, physical properties, and structural transformations. As a general concept, thermal analysis can be defined as any scientific or technological characterisation of a material in which temperature is varied as an experimental parameter.<sup>41,42</sup>

The thermal degradation and thermal oxidation stability of wood flour nanocomposites were investigated.

In the present study, composites with optimal mechanical properties and water absorption were selected for thermogravimetric investigations. The findings demonstrated that in comparison to thermal stability in

composites with equivalent chemical compositions but distinct nanofillers (graphene or nano-silica), nanocomposites incorporating graphene exhibited superior heat stability over those comprising nano-silica.

**Fig. 5.** Thermogravimetric curves of composites XIV, XV, XIX and XX

Approximately 10 % mass losses are seen in the temperature range 400–500 K. Rapid falls of the TGA curves are seen between 500 and 700 K. Full thermal degradation is seen between 740 and 800 K. Overall, the binders used do not affect the thermal stability of the composites.

## 4. Conclusions

In this study, composites based on wood flour, tetraethoxysilane (TEOS), and nanofillers (graphene, nanosilica (40)) have been obtained under conditions of optimal pressure and at several temperatures *via* the hot press method. The binder and nanofillers act simultaneously as reinforcing agents during the obtaining of composites, with the best results being achieved at relatively high concentrations of the binders and high thermal treatment temperatures. With regard to the nanoparticles employed in the composites, it was determined that graphene yielded the most favourable outcomes.

The investigative procedures that were conducted comprised the following: 1) structural analysis employing Fourier transform infrared spectroscopy (FTIR) and optical microscopy; 2) assessment of physical-mechanical properties; 3) determination of thermal stability by thermogravimetry; 4) evaluation of water absorption.

The structural and compositional characteristics of nanocomposites were examined through the analysis of FTIR spectra data. Research findings have demonstrated that enhanced mechanical, thermal, and hydrophobic properties are attained as a consequence of the formation of novel chemical bonds, which arise from chemical reactions between the active groups of the constituent elements.

Surface studies were conducted using an optical microscope to define the morphology of the composites and the character of the distribution of the ingredients within the composite body.

An investigation was conducted into the mechanical properties, with particular reference to the strengthening of the material under bending and its impact viscosity. In addition, the water absorption of the composites was studied by means of standard methods.

It has been demonstrated that the mechanical properties of composites are contingent on their production conditions and composition, particularly the kind and size of nanofillers utilised. For instance, the strength of composites containing binder in the range of 3%-20% increases with increasing technological temperature at constant pressure (15 MPa). Furthermore, the mechanical properties of the composites are shown to improve with the degree of dispersion of the sawdust particles of the nanocomposite.

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## НАНОКОМПОЗИТИ НА ОСНОВІ ДЕРЕВНОГО БОРОШНА, ЕТИЛСИЛКАТУ ТА РІЗНИХ НАНОЧАСТИНОК

**Анотація.** Це дослідження містить вичерпний огляд сучасних досліджень та методів характеристики нанокompозитів, а також ретельний аналіз останніх тенденцій у цій галузі. Нанокompозити представляють собою нову категорію матеріалів, що характеризуються наявністю наповнювачів нанорозмірного масштабу (графен 5–25 нм, нанокремнезем 70–90 нм). Ці матеріали мають значний

потенціал для застосування в різних галузях промисловості, включаючи автомобілебудування, авіакосмічну галузь, будівництво, електротехніку та харчову промисловість. Існує значний інтерес до використання наночастинок, таких як графен та нанокремнеземні наповнювачі, у розробці інноваційних природних композитів. Продемонстровано можливість отримання екологічно чистих нанокompозитних матеріалів з поліпшеними властивостями на основі відновлюваної природної сировини (деревного борошна), яка є легкодоступною та недорогою, а також наночастинок як важливих наповнювачів у полімерних композитах.

Розроблено ряд композитних матеріалів на основі деревного борошна з різними якість дисперсії та різним відсотковим вмістом біндера (3–20%), етилсилікату (40%), а також нанопоповнювачів, включаючи графен та нанокремнеземні частинки. Доведено, що додавання наночастинок покращує механічні властивості та загальні характеристики композитів.

З метою визначення складу нанокompозитів було проведено серію досліджень методом інфрачервоної спектроскопії з перетворенням Фур'є. Були досліджені фізико-механічні властивості та водопоглинання композицій, а також вивчена морфологія поверхні за допомогою оптичного мікроскопічного методу. Крім того, для спостереження за тепловими властивостями матеріалів були використані методи термогравіметричного аналізу.

**Ключові слова:** деревне борошно, нанокompозити, наповнювачі, нанокремнезем, графен, механічні та термічні властивості.