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# Impact of Alumina Particles on the Morphology and Mechanics of Hybrid Wood Plastic Composite Materials

Srdjan Perisić<sup>1\*)</sup>, Marija M. Vuksanović<sup>1</sup>, Milos Petrović<sup>1</sup>, Andjela Radisavljević<sup>1</sup>, Aleksandar Grujić<sup>3</sup>, Radmila M. Jančić - Heinemann<sup>2</sup>, Vesna Radojević<sup>2</sup>

<sup>1</sup>University of Belgrade, Innovation Center of Faculty of Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia

<sup>2</sup>University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia

<sup>3</sup>University of Belgrade, Institute of Chemistry, Technology and Metallurgy, Belgrade, Serbia

### Abstract:

Hybrid composite panels of Wood-Plastic Composites (WPC) consisting of wood and poly (methyl methacrylate) (PMMA) were reinforced with alumina particles and made by "hot pressing" method. Alumina-based particles were made by sol-gel technique. The particles were characterized by the X-ray diffraction (XRD). The resulting alumina particles were modified with (3mercaptopropyl) trimethoxysilane (MPTMS), in order to obtain better mechanical properties of the composite relative to the composite with unmodified alumina particles. The aim of this work was to study the influence of composite structure and the moisture absorption on bending and the impact properties of the hybrid composite. The bending and impact tests revealed that modulus of elasticity and absorbed energy of deformation increased with modification of alumina and slightly decrease after moisture absorption.

Keywords: Alumina particles; Wood; PMMA; Water absorption; Mechanical properties.

### 1. Introduction

Along with the development and increasing use of bioplastic composite materials, there was a need for various mechanical improvements. Alumina particles prepared by different techniques have been used as reinforcements in order to improve particular properties of composite structures [1-3]. Hybrid wood-plastic composites (WPC) with biodegradable natural fibers have many advantages, such as low density, high specific strength value, good impact and flexural properties. They are also eco-friendly and have cost-effective processability [4]. For the preparation of composites, wood fibers and particles are commonly used (as) biodegradable materials in thermoplastic polymer matrices. They have found application in various industrial fields, such as civil engineering, interior and exterior design and present excellent alternatives to inorganic fiber polymer composites. The main disadvantage of these composites is an incompatibility between hydrophilic wood fillers and hydrophobic polymer matrix, especially because of a high content of wood reinforcement [5-9]. The creep behavior of WPC originates from their viscoelastic nature [10] and depends on

<sup>\*)</sup> **Corresponding author:** natasha@nanosys.ihtm.bg.ac.rs; natashajvc@gmail.com

composition, moisture and compatibility of components. Low interfacial adhesion between wood and polymer matrix induces creep behavior and low performance [11]. This incompatibility can be upgraded with the addition of functional polyolefin as a coupling agent, providing a better stress transfer between the polymer matrix and wood filler [12-14]. The selection of thermoplastic matrices is crucial for the thermal stability of hybrid WPC and consequently thermal degradation of lignocelluloses in wood [15]. At the elevated temperature, chemical chains of thermoplastic polymer become movable, so the matrix become softer until melting point is reached. This qualifies thermoplastics to be suiTab. for reuse and recycling. In other hand, thermoreactive polymers do not melt at the higher temperature. The behavior of thermoreactive net structure is strongly connected with thermal degradation region at higher temperatures and could not be rearranged [16]. The most common polymers used as matrix in WPC production route are polyethylene (PE) [17-20], polyethylene (PE), polypropylene (PP), polystyrene (PS), poly (vinyl chloride) (PVC) and acrylonitrile butadiene styrene (ABS). From all of the polymers used as matrix in WPC, the most promising candidate which could importantly repair impact properties of wood is PMMA. In order to improve compatibility between PMMA and wood fibers, different bonding agents could be used. Based on previous research, it can be concluded that modification of wood fibers was better when (3mercaptopropyl) trimethoxysilane (MPTMS) was used compared with 2,4-toluene diisocyanate (TDI) [21].

The balance between the mechanical performance and environmentally friendly behavior is an important issue for hybrid WPC applications [22]. In order to obtain desired mechanical properties of WPC, the appropriate reinforcements should be applied. Among other elements such as glass fibers, metal wires or grids [23-25], alumina reinforcements are commonly used ceramic material for polymer matrix composites. Mechanical properties of the composite could be further defined by synthesis of alumina which enables a great number of possibilities to obtain crystal structures. The heat treatments which can produce different structures of alumina are of special interest in the preparation of composite materials. The use of ceramic materials in PMMA-based WPC is desirable because of their excellent properties, including chemical durability and better mechanical properties of obtained composite [26].

Many different routes for determination of water absorption by WPCs exist: exposing to the air on defined air humidity conditions, immersing in water or in weather chambers where weather conditions can be simulated. Many research activities in the field of building materials are directed toward to control of the moisture absorption properties and to characterize the impact of coupling agents on dimensional stability of applied composites [27, 28].

Taking these facts into account, the aim of this work was to study the influence of processing parameters and moisture absorption on the impact properties and flexural strength of synthesized hybrid composite materials. The correlation between processing parameters and final properties of WPC reinforced with alumina was analyzed and discussed.

# 2. Materials and Experimental Procedures

Poly (methyl methacrylate) (PMMA) (Chi-Mei, China) was used as the matrix material, while acetone supplied by Zorka Pharma, Serbia was used as organic solvent. Mechanically groundwood, 100 % *Abiesalba Mill*. "flash dried" refined mechanical pulp (RMP), i.e., paper pulp (Metso Paper Oy, Sweden) were used as wood fiber reinforcement in PMMA matrix. Cross-linking bonding agent was (3mercaptopropyl) trimethoxysilane (MPTMS) (Sigma-Aldrich, Co.-Germany). Aluminum hydroxide chloride (Locron L; Clariant company) was used for the preparation of alumina based particles.

Initially, the solution for modifying wood paper pulp (mass 20 g) was made as described earlier [21]. As-received wood paper pulp was immersed in this solution and stirred

for 30 min at room temperature. At the same time, PMMA solution (10 wt.%) in acetone was prepared. The modified wood paper pulp was added directly to PMMA solution (PMMA to the wood pulp mass was 2:1) and dried at 40 °C for 24 h.

Alumina-based particles were synthesized using the sol-gel technique. The aluminum hydroxide chloride (Al<sub>2</sub>Cl(OH)<sub>5</sub> x 2.5 H<sub>2</sub>O) and demineralized water were mixed on the magnetic stirrer until the aluminum hydroxyl chloride was completely dissolved and then put into a petri dish and allowed to dry. The gel was milled in laboratory mortar and the powder was calcinated at 900 °C during 2 h. Distribution of the diameter of synthesized particles measured in our previous work and the mean diameter was about 2  $\mu$ m [26]. In order to improve the bonding between reinforcement and matrix, functionalization of alumina particles was carried out before preparation of composite. For functionalization, MPTMS was used, which has an epoxide and a silanol group at the ends of the chain. The silanol end can form a bond with inorganic phase whereas the epoxide end can participate in cross-linking. The entire functionalization procedure was explained in an earlier study [29].

Prepared samples were mixed with the vertical blender "IKA RW 20 digital" at a set speed of 350 rpm. The samples were dried at 40 °C for 24 h, in order to remove residual humidity. After that, they were exposed to hot pressing on a hydraulic press (Sojuzzagranpribor P-125, Moscow).

Before testing, the thickness of the samples was  $5 \pm 0.1$  mm. To prevent sticking of the sample, the mold was firstly lubricated with Teflon "MS 122 dry lubricant". The suiTab. doses of WPC were placed in the demounTab. steel mold (100 x 100 mm), with applied pressure and heating. The processing parameters of WPC samples are given in Tab. I.

**Tab. I** Processing parameters of WPC samples obtained at T=180 °C and t=60 min: 1) PMMA-wood-MPTMS; 3) PMMA-wood-MPTMS with 3 wt.% Al<sub>2</sub>O<sub>3</sub>; 5) PMMA-wood-MPTMS and 5 wt.% Al<sub>2</sub>O<sub>3</sub>.

Samples	PMMA, wt.%	Wood+MPT MS, wt.%	Al <sub>2</sub> O <sub>3,</sub> wt.%	Al <sub>2</sub> O <sub>3</sub> + MPTMS	$m_{wpc,}\ g$
1	65	35	0	-	60
3N	65	32	3	-	60
5N	65	30	5	-	60
3M	65	32	3	<b>MPTMS</b>	60
5M	65	30	5	MPTMS	60

<sup>\*(</sup>N-unmodified, M-modified Al<sub>2</sub>O<sub>3</sub> with MPTMS).

FTIR spectra were recorded in the range 4000-400 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup> using a FTIR spectrometer (BOMEM Michelfan MB-102 FTIR). All samples were crushed into a powder, mixed with KBr and the resulting powders were compressed into Tab.ts for the infrared test.

Fracture of the samples was observed by Field Emission Scanning Electron Microscope (FESEM) (JSM 5800, Tescan Mira 3, Brno, Czech Republic) using a voltage of 5-10 kV, with magnifications of 500 x and 1 kx.

The crystalline phase of the synthesized particles is determined by X-ray diffraction analysis. XRD pattern was recorded with an Ital Structure APD2000 X-ray diffractometer in a Bragg-Brentano geometry using CuK $\alpha$  radiation ( $\lambda$  = 1.5418 Å) and step-scan mode (range: 20–75° 20, step-time: 0.50 s, step-width: 0.02°). The corundum structure of the alumina particles is confirmed by comparison of the XRD data to the standard card 93096-ICSD.

Controlled impact energy tests were performed on high-speed puncture impact testing machine Hydroshot HITS-P10 (Shimadzu, Japan). The striker, with a hemispherical head and a diameter of 12.7 mm, was loaded with programmable velocity, height and attained a value

of depth. The impact speed and force were set at 2 m/s and 10 kN, respectively. In this manner, it is possible to control the impact energy.

The flexural (three-point banding) tests were conducted on a computer controlled model "INSTRON" Fast Track 8800machine. The speed of crosshead was set at 2.0 cm/min.

The microhardness of the WPC composite systems was characterized using micro Vickers hardness (HV) tester Leitz, Kleinharteprufer DURIMETI. For each sample, three indents were performed at room temperature applying a load of 500 g for 25 s duration according to the standard [30]. The samples were recorded on an optical microscope (Carl Zeiss – Jena, NU2) and the diagonals of indent were measured with software for image analysis (Image Pro Plus). Micro hardness is calculated using the following equation:

$$VHN = 2\cos\theta \frac{22^{\circ} p}{d^2} = \frac{1.8544 p}{d^2}$$

where P (kgf) is the applied load and d (mm) is the average length of the indentation diagonal [31]. Water absorption was calculated according to the difference in weight of immersed samples and was estimated by using equation (1):

$$WA[\%] = \left[\frac{(W - W_D)}{W_D}\right] \times 100 \tag{1}$$

where  $W_D$  is the oven-dried sample at 60 °C for 24 h which was immersed in deionized water at 20 °C for 28 h, while W presents the weight of the sample after water immersion [32].

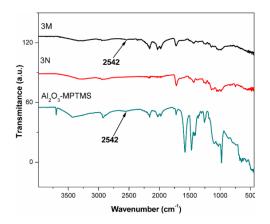
## 3. Results and Discussion

Fig. 1 present the FTIR spectra of modified Al<sub>2</sub>O<sub>3</sub> with MPTMS, as well as WPC (PMMA-wood-MPTMS) with unmodified and modified alumina.

The spectra show a peak in the range around 3400 cm<sup>-1</sup>, originating from the hydroxyl group and around 1040 cm<sup>-1</sup>, which is assigned to C–O vibrations in cellulose. Spectrums of PMMA-wood-MPTMS show peaks of 1070, 1039 and 994 cm<sup>-1</sup> assigned to the Si–O–H and Si–O–Si groups [21].

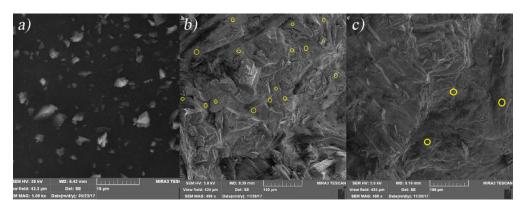
The peaks at 822, 763 and 628 cm<sup>-1</sup> are attributed to the Al–O bonds that are supposed to arise from a pseudo-boehmite structure. Frequencies reported at 630 cm<sup>-1</sup> and at 763 cm<sup>-1</sup> correspond to the Al–O stretching and the torsional mode, respectively [26].

The peak at  $2542 \text{ cm}^{-1}$  in spectra of  $Al_2O_3$ -MPTMS belongs to the –SH bond, indicating successful preparation of thiol-modified alumina ( $Al_2O_3$ –SH) [33]. The same peak is present on the spectrum of the WPC with modified alumina.



**Fig. 1.** FTIR spectra of: Al<sub>2</sub>O<sub>3</sub>-MPTMS, 3N) WPC with 3 wt.% unmodified alumina, 3M) WPC with 3 wt.% modified alumina.

The FESEM micrographs of fracture surfaces of the synthesized alumina particles sample and WPC samples with and without modification are presented in Fig. 2.



**Fig. 2.** SEM analysis of (a) agglomerates of synthesized alumina particles (b) PMMA with wood and unmodified alumina, (c) PMMA with wood and modified alumina.

By addition of alumina particles to MPTMS, it was found that the particles are uniformly dispersed in the sample (Fig. 2c) and bonded with the whole composite volume, opposite to the sample without MPTMS (Fig. 2b). The alumina particles are surrounded by polymer phase much better than in the sample without the bonding agent, thus contributing to the compatibility between modified alumina and rest of the sample. The mechanical properties of composites are in direct dependence on the bond stretching and bond strength at the interface which allows stress transfer from the matrix to the filler and vice versa.

The X-Ray diffractograms of the synthesized alumina-based particles after heat treatment at 900 °C with identified phases is shown in Fig. 3.

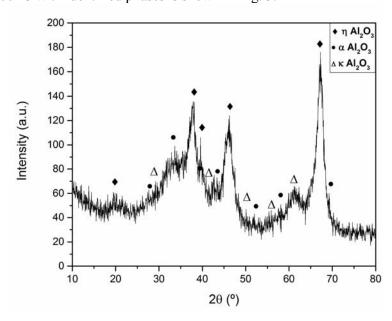
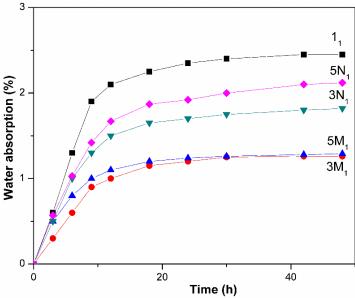


Fig. 3. XRD of the synthesized alumina-based particles after heat treatment at 900 °C.

The dominant structure in the synthesized alumina based particles sintered at 900 °C is  $\eta$ – Al<sub>2</sub>O<sub>3</sub> (PDF-2 77-0396).

From application point of view important information is materials behavior under humid and water environment. In that sense, water absorption test was carried out. The results for the samples with alumina and without alumina reinforcement are shown in Fig. 4. The water resistance that samples with modified alumina showed after immersion for 48 h was significantly higher compared to the WPC sample. The decrease in water uptake was determined for all samples with alumina treated or untreated with a bonding agent (Fig. 4).

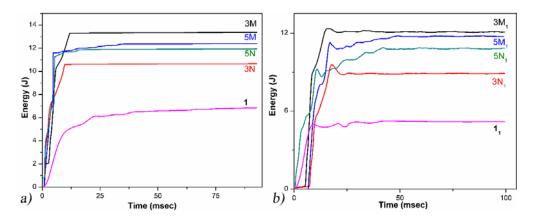


**Fig. 4.** Water absorption results for: 1) wood-PMMA-MPTMS, 3N) WPC with 3 wt.% unmodified alumina, 3M) WPC with 3 wt.% modified alumina, 5N) WPC with 5 wt.% unmodified alumina and 5M) WPC with 5 wt.% modified alumina.

The water absorption–time curves present two well-divided regions. At beginning of the process, until 10-12 hours, the water uptake kinetics were very fast, eventually becoming slow, resulting in almost complete congestion of the samples. Hydrophobic PMMA was the reason for reduced water uptake in samples with bonding agents. The cause stands in the fact that polymer overspread all wood fibers inside of composite. It is necessary to reduce at minimum the voids in the processing of composites because high void percentage may initiate the low mechanical properties and fatigue resistance under water sorption. The voids were minimized by modifying matrix/particles interface, and it was found that there exists a lower porosity and higher force transfer capability at the matrix/particles surface in the composites with alumina treated by pre-impregnation and a silane coupling agent. As it is shown, in Fig. 4, slightly better resistance to water adopt has modified sample with 3 than 5 wt.%. Also, samples with a bonding agent (3M and 5M) are more homogeneous than the wood–PMMA sample and consequently absorb less water than other samples.

Mechanical testing cover impact, flexural strength and hardness measurements for both set of samples, before and after water immersion. In order to investigate the impact behavior of composite materials, the resistance of the materials to high-speed impacts was tested. The obtained results gave the information about the behavior of the WPC composite in the situation of impact induced by a sudden hit and toughness. The results are shown in Fig. 5. Energy–time curves for samples with alumina particles show the expected significant increase in the absorbed energy (70-120 %) then WPC sample without them (Fig. 5, curve 1). Sample with 3 wt.% modified alumina absorbed the highest amount of energy during impact, followed by sample with 5 wt. % modified alumina. The lowest absorbed energy was for the samples of 3 wt.% untreated alumina. An increase in the maximum absorbed energy was observed in the samples with modified alumina. The addition of modified alumina particles to the composite gives higher impact strength of samples compared to samples with unmodified

particles. WPC with 3 wt.% modified alumina (curve 3M) has absorbed approximately 20 % more energy than the sample with 5 wt. % modified alumina (curve 5M), and the difference between sample 3M and the samples without modification 3N is 30 %. The overall results obtained from impact test showed that the addition of alumina particles to WPC improved impact energies of WPC samples. The results show that reinforcement modification, particles size and particle content have a great influence on the fracture mechanics and plane strain fracture toughness. The presented results of puncture test for WPC with alumina suggest that higher wt.% of alumina made uncertain samples. This is consequence of agglomeration of alumina particles which leads to formation of weak points in sample structure.



**Fig. 5.** Energy-time diagram from impact test: a) before and b) after water immersion (3N) WPC with 3 wt.% unmodified alumina, 3M) WPC with 3 wt.% modified alumina, 5N) WPC with 5 wt.% unmodified alumina and 5M WPC with 5 wt.% modified alumina).

It is obvious from Fig. 5 that water uptake has less influence on samples with modified than unmodified particles. WPCs after water immersion absorbed 20 % less impact energy then water untreated samples. The obtained values of flexural strength, flexural modulus, and Vickers micro hardness for samples with different reinforcements before and after water immersion are presented in Tab. II.

**Tab. II** Flexural strength (Rb), flexural modulus (E), Vickers microhardness of produced WPCs.

Samples	Rb (MPa)		E (MPa)		HV (GPa)	
	before	after	before	after	before	after
1	43.43	39.85	405	389	0.141	0.118
3N	44,28	42.89	424	399	0.186	0.179
5N	45,70	44.59	432	408	0.218	0.204
3M	53,47	51.32	482	475	0.193	0.190
5M	50,65	49.12	435	420	0.223	0.218

Analyzing the results presented in Tab. II, it is obvious that all parameters (*Rb*, *E*, and *HV*) have higher values before water immersion than after water immersion. The difference between Rb and Rb<sub>1</sub> for sample 1 is 3.58 MPa, for sample reinforced with 3 and 5 wt.% of Al<sub>2</sub>O<sub>3</sub> (modified and unmodified) is much lower. This occurs due to lower mass fraction of wood and higher quantity of Al<sub>2</sub>O<sub>3</sub>. The very similar behavior of flexural modulus is obtained. The values of Rb and E are the highest modified 3M sample with 3 wt.% of alumina before water immersion. The WPC composites with the higher mass fraction of alumina show slightly poorer properties compared to 3M due to structural properties such as the formation of weak bonds between polymer matrix and reinforcements, interactions between

reinforcement particles and bad sectors. The measurements of hardness are predominantly oriented to the surface properties of WPC including homogeneity, distribution of fillers and final treatment of the surface. The hardness increases with higher content of alumina reinforcement in both cases, modified and unmodified. As expected, the values of HV after water immersion are lower.

### 4. Conclusion

Comparative analysis presented in this work towards conclusion that reinforced WPC with alumina particles of approx. 2  $\mu$ m in diameter provide great influence on upgrading the wather resistance and mechanical properties. Beter influence on composite strength has been achieved with a bonding agent for surface treatment of alumina. The water absorption was lower in samples with modified then unmodified alumina and in samples with the higer content of wood. Mechanical tests of hybrid WPC with alumina particles shows expected significant increase in the mechanical properties.

Samples with 3 % modified alumina (3M) reinforcement showed the highest value of absorbed energy and flexural strength compared to other series. Combination of wood-PMMA interface modification and the addition of modified alumina particles in small weight percentage (up to 3 %) results in composites with advanced mechanical and swelling properties and gives opportunity for further developments of alumina incorporation.

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Садржај: Хибридни композитни панели дрво-пластика композит (ДПК) који се састоје од дрвета и поли(метил метакрилата) (ПММА) ојачани честицама алуминијум оксида прецесирани методом "врућег пресовања". Честице засноване на алуминијум оксиду направљене су сол — гел техником. Карактеризација честица вршена је рендгенском анализом. Добијене честице алуминијум оксида модификоване су помоћу (меркаптопропил)триметоксисилана, како би се постигла боља механичка својства композита у односу на композит са немодификованим честицама алуминијум оксида. Циљ овог рада је проучавање утицаја структуре хибридног композита и апсорбоване влаге на механичка својства, савијање и контролисани удар. Тестови савијања и удара показали су да се модул еластичности и апсорбована енергија

деформације повећава модификацијом алуминијум оксидних честица, а незнатно самањује након апсорпције влаге.

**Кључне речи** Алуминијум оксидне честице, Дрво, ПММА, Апсорпција воде, Механичка својства

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