

THE IMPACT OF ADDING TIMBER WASTE ON THE MICROSTRUCTURE OF FLY ASH-BASED GEOPOLYMER COMPOSITES

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Abstract

The increasing demand for building materials has significantly heightened the consumption of virgin raw materials, particularly sand and gravel. As a result, there is an ongoing effort to identify alternative products that can be integrated into building material formulations. This initiative aims to enhance the properties of these materials while reducing the quantity of aggregates used. One promising alternative is timber waste or furniture scraps, which can serve as lightweight aggregates in building materials. Current literature discusses the influence of these products on the mechanical properties of geopolymers and conventional concrete. However, the interaction and effects of these organic particles on the microstructure of geopolymers have not been extensively studied. Therefore, this study aims to evaluate the impact of wood particles on the morphology of fly ash-based geopolymers. To achieve this, mixtures containing 10%, 20%, and 30% wood content by weight were prepared. These mixtures were subjected to compressive strength tests, and the destruction zones were analyzed using scanning electron microscopy to observe the interface transition zone between the matrix and the reinforcing particles. The microstructure analysis revealed that, at certain percentages of wood waste, the particles were homogeneously distributed within the matrix and positively contributed to the mechanical properties of the composite. Furthermore, the type of particle was also significant; larger particles can slow down crack propagation, while smaller particles can fill pores, leading to a denser matrix. However, at high percentages, the wood particles tend to absorb large amounts of activator, negatively affecting the dissolution of fly ash particles, which ultimately results in a weaker matrix in terms of mechanical properties.

Keywords: *geopolymers composites, microstructural analysis, SEM, EDX, fly ash, wood addition.*

Introduction

The growing demand for eco-friendly building materials has fueled the development of sustainable geopolymers that can serve as alternatives to Ordinary Portland Cement (OPC)-based materials [1]. Consequently, various mixtures of aluminosilicate minerals and recycled products have been researched to identify the optimal combination that provides the necessary mechanical properties for the construction sector [2]. Fly ash is one of the most commonly used materials in the synthesis of geopolymer matrices [3]. To enhance these matrices, multiple types of waste materials have been identified as suitable reinforcements [4]. Glass fibers or glass particles,

marble, and various metallic particles have all been successfully incorporated [5]. Additionally, to achieve high performance and lightweight characteristics, wood in different forms—such as flour, fibers, or chips—has proven to be an excellent choice [6]. Given that natural wood is already valued for furniture or energy production, the use of lignocellulosic wood waste in geopolymer matrices has recently been investigated [7]. This approach addresses two significant challenges in the sector: valorizing waste from furniture (mainly wood mixed with wax and resins) and reducing the consumption of virgin raw materials, thereby supporting the goals of a circular economy.

Wood-fly ash geopolymer composites benefit from the good mechanical properties conferred by the fly ash matrix, while also offering reduced weight and improved thermal insulation due to the incorporation of wood particles [8]. Current literature suggests that the mechanical properties of these wood-geopolymer composites are affected by the type of wood used and any treatments applied to it [9]. Treated wood particles can improve the interfacial bonding within the composite, resulting in better performance [10].

The compressive and flexural strengths of geopolymers can be optimized by incorporating a limited amount of wood, typically between 10% and 25% by weight, particularly when using wood fibers [11]. However, increasing the wood content may lead to higher porosity, reduced bulk density, and decreased mechanical durability [12]. These issues often stem from microstructural incompatibilities between the organic and inorganic components of the composite [13].

Previous studies focusing on microstructural analysis have revealed common challenges, such as the formation of cracks at the wood-matrix interface and water-induced swelling and shrinkage during curing cycles [14]. These factors can significantly impact the long-term durability of geopolymer-wood composites. Despite some promising advancements in thermal performance, the durability of these composites, especially under freeze-thaw conditions, remains a critical challenge. Furthermore, the effects of wood on the dissolution capacity of the activator have not yet been addressed in the literature. Nevertheless, life cycle assessments indicate that these materials possess a lower environmental footprint compared to conventional composites [15].

Other studies indicate that the performance of wood particles in these composites is also affected by the type of aluminosilicate raw materials used to synthesize the matrix [16]. The availability of various activators and raw materials makes it more challenging to find the right combination of factors that can maximize the effectiveness of geopolymer composites.

To improve the compatibility between the inorganic matrix and the wood particles, different treatments can be applied to the lignocellulosic component [17,18]. Among the most common options are surface treatments with sodium hydroxide solutions and thermal treatments [19,20]. An increase in surface roughness will enhance the bonding strength, while thermal treatments can yield wood particles with lower porosity and reduced absorption of activator. Overall, these surface treatments can improve the performance of geopolymer composites; however, they may also add additional costs and environmental impacts to the final product.

This study aims to evaluate the impact of wood particles on the morphology of fly ash-based geopolymers. To achieve this, mixtures containing 10%, 20%, and 30% wood content by weight were prepared. These mixtures were subjected to compressive strength tests, and the destruction zones were analyzed using scanning electron microscopy to observe the delamination zones between the matrix and the reinforcing particles.

Materials and Methods

To identify sources of raw materials for geopolymers, it is essential to analyze their chemical composition. The strength and homogeneity of the Si-O-Al tetrahedral structure significantly affect the final geopolymer's quality [21]. Impurities can create structural defects, impacting the material's properties [22]. Therefore, assessing the chemical and phase composition of the raw

material is vital for determining its geopolymerization potential and the appropriate activation solution.

Materials

The properties of the solid component, including chemical composition, moisture content, and particle size, are essential for determining mechanical strength, durability, and microstructure in the final material. Different sources of raw materials with geopolymerization potential were identified locally for a project aimed at developing geopolymeric composite materials with the addition of wood waste, cured at room temperature. Selected materials include fly ash, blast furnace slag from the LIBERTY Galati Combine, and wood waste (wood chips and powder).

Blast furnace slag positively influences the properties of cement-based materials and improves freeze-thaw resistance in geopolymers made from thermal power plant ash [23].

The liquid component consists of sodium silicate and sodium hydroxide. Sodium silicate is produced by fusing sand with sodium carbonate at high temperatures and is used in combination with sodium hydroxide for geopolymerization. A commercially bought sodium silicate solution with a density of 1.52 g/cm³ and pH 11.5 will be used, containing at least 44.8% sodium silicate and other components [24]. The concentration of the sodium hydroxide solution significantly influences the geopolymers' properties, with higher NaOH concentrations enhancing early strength and stability in acidic conditions. NaOH was dissolved in water 24 hours before mixing with other components.

Methods

On the basis of preliminary experiments and literature, wood waste was added additionally in the solid component by referring to the mass formed by fly ash (FA) and blast furnace slag (ZF), thus in order to compare the obtained results, FA or ZF was not replaced by wood waste (DL), because it does not react in alkaline medium. Also, the same compositions were studied for both types of woody waste; therefore, DL refers to either woodchips or wood pulp, depending on the type of waste studied in the respective shard. Therefore, the following geopolymer design factors (parameters) were chosen in the Pre-Geo project: i) three mixtures comprising different percentage of fly ash (FA), blast furnace slag (ZF) and woody waste (DL) named as B1 = 60% FA + 40% ZF + 0% DL, B2 = 60% FA + 40% ZF + 10% DL and B3 = 60% FA + 40% ZF + 20% DL (Table 1), (ii) three different liquid-to-solid ratios (0.40, 0.45, and 0.50), (iii) three different Na₂SiO₃/NaOH ratios (1.0, 1.25, and 1.5), and (iv) three different molar concentrations of NaOH solution (8, 10, and 12). The design of the experiments with three factors and three levels is shown in Table 2. Consistent with the Taguchi method with L9 orthogonal matrix (36), 9 different mixtures were required to establish the influence of all factors involved.

Table 1. Geopolymer mixtures

Solid component code*	Mixture content, % wt.		
	FA	ZF	DL
B1	60	40	0
B2	60	40	10
B3	60	40	20

*Per 1 kg of solid component in the case of mixture B1 (600 g FA and 400 g ZF), B2 will contain 600 g FA, 400 g ZF and 100 g DL and B3 will contain 600 g FA, 400 g ZF and 200 g DL.

Table 2. Experimental factors and different levels

Experimental factor	Level 1	Level 2	Level 3
A. Solid component mix	B1	B2	B3
B. Mass ratio of liquid to solid*	0,7	0,75	0,8
C. Mass ratio between Na ₂ SiO ₃ /NaOH	1	1,25	1,5
D. Molar concentration of NaOH solution	3	6,5	10

*In the case of DL samples, additional water had to be added to ensure the workability of the mixture.

The production of geopolymers using three types of solid raw materials follows these steps:

1. Prepare the solid component by drying and dosing each mineral waste or waste wood according to the recipe.
2. Mix the dry solids to enhance homogeneity.
3. Prepare the liquid component by mixing sodium hydroxide and sodium silicate solutions, allowing the NaOH to dissolve for at least 24 hours to prevent interference during geopolymerization.
4. Combine the solid and liquid components using a variable speed mixer until a homogeneous paste is achieved.
5. Discharge the mixture into molds for testing.
6. Vibrate the samples on a vibrating table to remove air bubbles.
7. Cure the samples in the molds for 24 hours, covering the surface with plastic film to minimize evaporation before de-molding.

Results and discussions

Raw materials analysis

Fly ash

A representative sample was analyzed using X-ray fluorescence (XRF) with S8 Tiger equipment (Bruker) to assess the chemical composition of the raw materials. This analysis is crucial for evaluating the geopolymerization potential and characteristics of the activator. The concentrations of aluminum, silicon, iron, and calcium oxides significantly impact the final product's properties. The fly ash from the CET II thermal power plant's exhaust filters shows high levels of SiO₂, Al₂O₃, Fe_xO_y, CaO, K₂O, TiO₂, P₂O₅, SO_x, and other elements, as detailed in Table 2.

Table 2. Oxide chemical composition of fly ash

Chemical element	Si	Al	Fe	Ca	K	Ti	P	S	Oth. elem.
%, wt.	20.28	11.95	5.34	3.09	0.79	0.68	0.54	0.51	<0.3
St. dev., %	0.12	0.14	0.03	0.02	0.01	0.01	0.01	0.01	-

Thermal power plant fly ash is the mineral waste captured by the exhaust filters positioned at the top of the blast furnace. Because it is formed in suspension (the molten minerals in small droplets are entrained by the exhaust gases), the particles in its composition are in the form of hollow spheres or spheres containing smaller spheres (porous particles). SEM analysis at different magnifications (Figure 1) shows a relatively homogeneous composition of a mixture with a high content of fine particles (< 100 μm in diameter).

The particle size distribution of the raw material particles has a significant effect on the microstructure and mechanical properties of geopolymers. In general, geopolymers made from fine particles will, after curing, have superior mechanical properties and a denser microstructure than those made from predominantly millimeter-sized particles.

In order to improve the freeze-thaw resistance as well as the environmental friendliness of the obtained materials, natural aggregates were replaced by granulated blast furnace slag. The blast furnace slag used was supplied by Liberty Galati, Romania. Due to the high content of Ca (about 40%) and Al (about 10%) oxides in its composition, blast furnace slag can improve the freeze-thaw resistance of geopolymers.

The blast furnace slag used in this project contains Ca as the main chemical element in its composition, except for O. It also has a considerable content of Si, Al and Mg. As can be seen from Table 3, in addition to the 4 chemical elements, this waste also contains S compounds, but in a low amount. The presence of S in cementitious materials can negatively influence their durability, therefore it is desirable that the mineral wastes identified as raw materials contain sulfur in a very low amount [33].

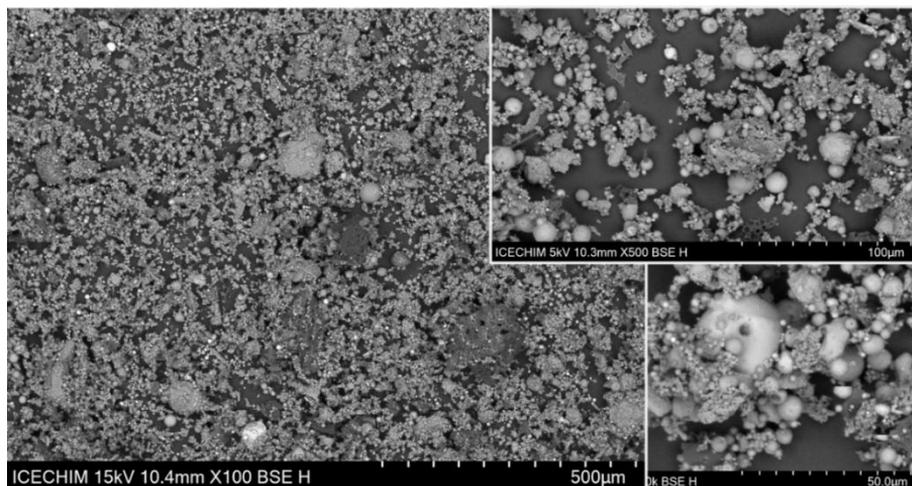


Figure 1. Particle morphology of fly ash used as raw material

Table 3. Elemental chemical composition of blast furnace slag

Chemical element	Si	Al	Ca	K	Mg	S	Oth. elem.	O
% wt.	13.3	4.7	22.9	0.4	3.9	0.2	<0.3	balance
St. dev., %	0.2	0.1	0.3	0.1	0.1	0.1	-	0.5

The microstructural analysis of the slag particles shows the presence of spongy formations, but also irregularly shaped compact structures (Figure 2). At high magnification powers, it can be observed that the surface of these particles is typical of materials formed by solidification from the liquid state. Thus, in some areas, there are microparticles partially embedded in the larger particles, whose morphology is different. Therefore, slag particles are formed from a mixture of minerals of different composition and morphology.

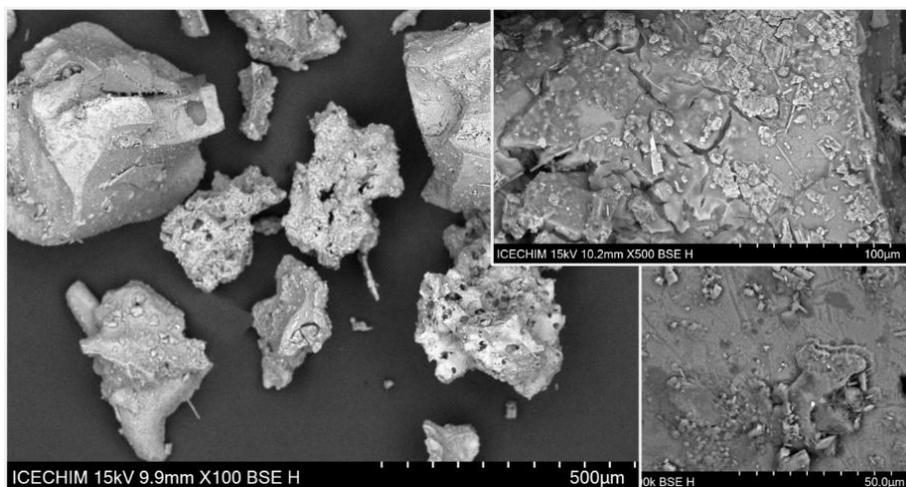


Figure 2. Particle morphology of blast furnace slag used as raw material

Wood waste

One of the most recent approaches for replacing aggregates or natural fibers in geopolymers is the use of wood waste. Flours, wood chips or fibers from discarded furniture or construction and demolition waste can be incorporated into geopolymers to produce lighter products and, most importantly, to reduce the use of virgin raw materials. However, when combining inorganic and organic materials into a product that should last for thousands or hundreds of years, it is very difficult to fully assess the behavior of the resulting composites.

The microstructural SEM analysis of the two types of woody waste used is shown in Figure 3. Both types of woody waste contain particles with irregular geometry and porous and layered structure (Figure 4).

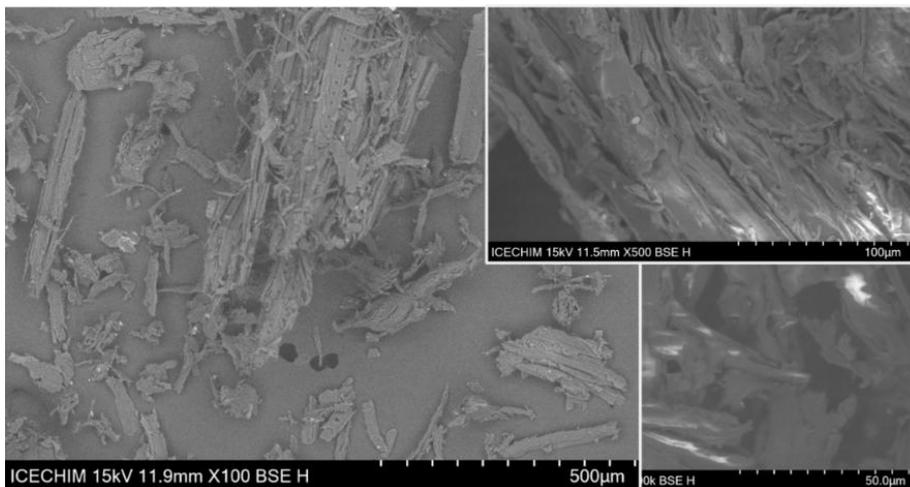


Figure 3. Morphology of the wood chips particles

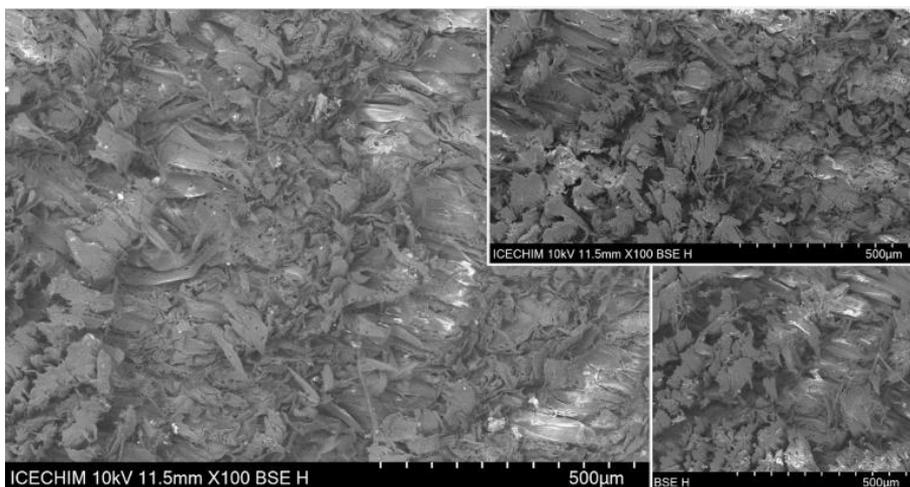


Figure 4. Wood powder particle morphology

Two types of wood waste were used in this study, namely woodchips and wood powder (flour) resulting from the processing of untreated wood with the rind. Their chemical composition is presented in Table 4.

Table 4. Elemental chemical composition of wood chips and wood powder

Timber waste Chemical element	Chips			Wood dust		
	C	O	Oth. elem.	C	O	Oth. elem.
%, wt.	62.1	37.6	<0.3	56.5	41.8	<0.5
St. dev., %	0.5	0.5	-	0.6	0.6	-

Microstructural analysis of geopolymer composites

Morphological analysis of the obtained samples and microstructural features were analyzed by scanning electron microscopy (SEM) using a FEI Quanta FEG 450 type SEM (FEI Company, Washington, DC, USA). The study of surface characteristics was performed on the samples with the most relevant mechanical characteristics, i.e., the blends that exhibited the best and the lowest mechanical characteristics determined by preliminary experiments. The mechanical performances of the obtained geopolymers were evaluated considering the requirements of SR EN 196-1:2016. Thus, preliminary tests identified the following compositions as relevant for comparative microstructural analysis (Table 5). The mechanical performance of all compositions will be presented in the report of the second phase of the project.

Table 5. Mechanical properties of some geopolymer samples

Sample code	A	B	C	D	FA [%]	ZF [%]	DL [%]	R _i [MPa]	R _c [MPa]
S1-M	B1	0.4	1	8	60	40	-	1.1	12.44
S1-Ma	B1	0.5	1	8	60	40	-	1.0	11.43
S6-PL	B2	0.5	1	12	60	40	10	0.77	11.52
S8-PL	B3	0.45	1	10	60	40	20	0.52	3.24
S6-Ta	B2	0.5	1	12	60	40	10	1.75	12.90
S8-Ta	B3	0.45	1	10	60	40	20	0.68	4.50

Where: M - geopolymer matrix with a liquid to solid ratio of 0.4; Ma - geopolymer matrix with a liquid to solid ratio of 0.4; PL - wood powder; Ta - wood chips; DL - wood powder or wood chips; R_i - 28-day flexural strength; R_c - 28-day compressive strength.

The microstructural analysis of the obtained geopolymer composites was carried out on surfaces resulting from mechanical determinations (in case-hardness) in order to highlight the fracture mechanism of these materials and their microstructural particularities. Therefore, the presence of cracks may also be due to the destruction mechanism of the samples during the compressive strength test.

SEM microstructural analysis of sample S1-M (Figure 5) shows partial activation of the raw materials by the alkaline activator. As a result, compact and homogeneous zones specific to complete dissolution can be observed, but also partially dissolved or undissolved thermal power plant ash particles. Also, some slag particles are unreacted and embedded in the geopolymer structure. At high magnification powers, it is observed that cracks advanced through the interface between the matrix and the unreacted particles. This aspect confirms a low adhesion of the matrix to the surface of the unreacted particles, especially in the case of the slag particles, where an almost complete separation can be observed. Therefore, under these conditions, unreacted slag particles behave as material defects having a negative influence on the mechanical properties of these composites. Increasing the liquid-to-solid ratio significantly influences the morphology of geopolymers. As can be seen in Figure 6, the sample with a liquid to solid ratio of 0.5 exhibits a much higher number of cracks as well as a morphology specific to a material formed from a mixture of crystals. The presence of unreacted particles and voids is also more evident. By comparing the samples with the addition of wood waste with those without waste, it can be observed that the introduction of particles in the form of a log (Figure 7) leads to the formation of a structure with high compactness and a very low number of unreacted particles. At the same time, the study of the S6-Ta sample at high magnification powers (Figure 8) confirms the presence

of homogeneous/compact zones that do not block the advancement of cracks through the geopolymer structure.

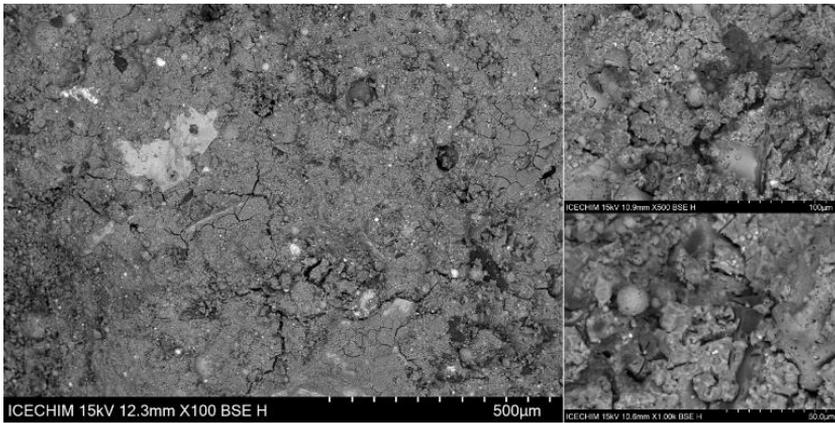


Figure 5. Microstructural analysis of sample S1-M

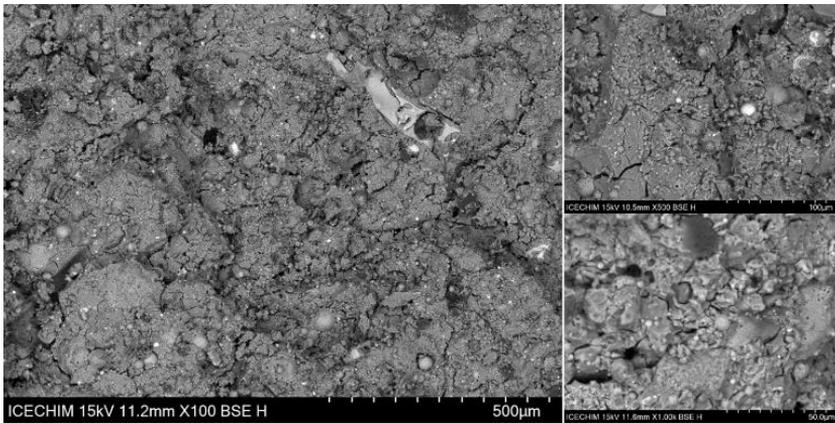


Figure 6. Microstructural analysis of sample S1-Ma

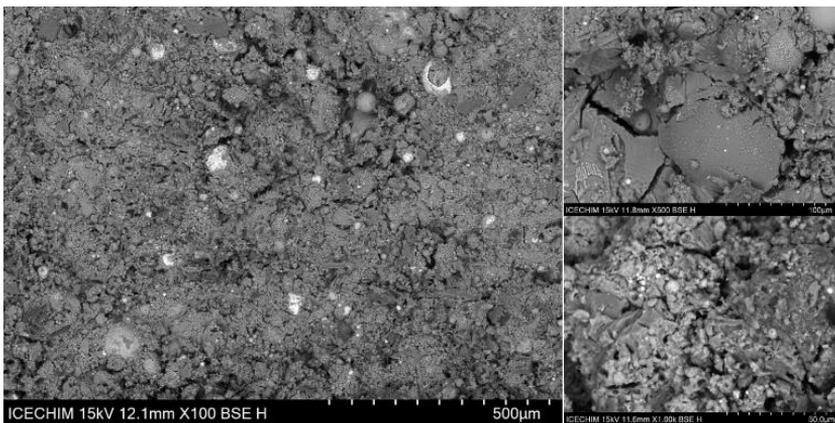


Figure 7. Microstructural analysis of sample S6-Ta

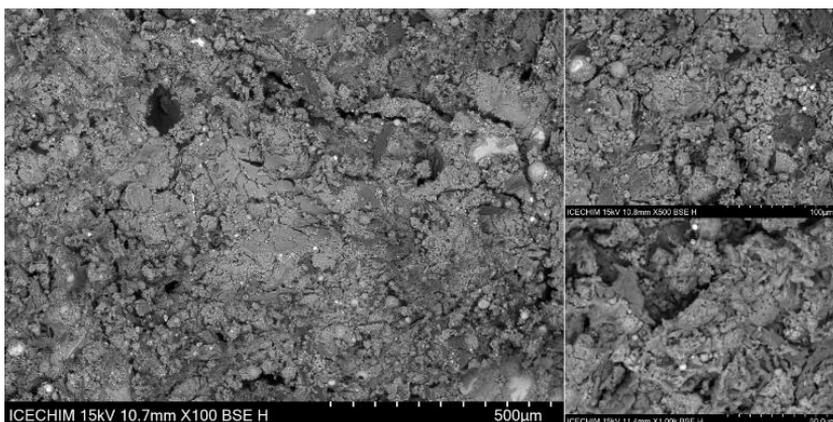


Figure 8. Microstructural analysis of sample S6-PL

In the case of composites with added wood waste, the microstructure of the samples is significantly influenced by the amount of wood. As can be seen, increasing the amount of wood from 10% to 20% resulted in a matrix with reduced compactness, consisting of grains of partially activated material embedding larger particles of thermal power plant ash or slag. However, each pellet has a compact and homogeneous structure specific to the complete activation of the particles in contact with the activator. Therefore, the wood particles have absorbed the activator in the early stages of the geopolymerization reaction, thus reducing the possibility of the composite forming a homogeneous structure during the gel phase. At the same time, this phenomenon may also explain the poor mechanical performance of wood-rich composites.

By comparing the mixture S8-PL (Figure 9) with S8-Ta (Figure 10), it can be seen that the grains in the structure are much finer in the wood powder sample. Therefore, the wood particles function as activation centers, since the activator absorbed into their structure, which is released during the drying of the sample, will cause the activation of the raw material particles in the vicinity of the wood particles, thus causing their dissolution and their adhesion to the wood particle.

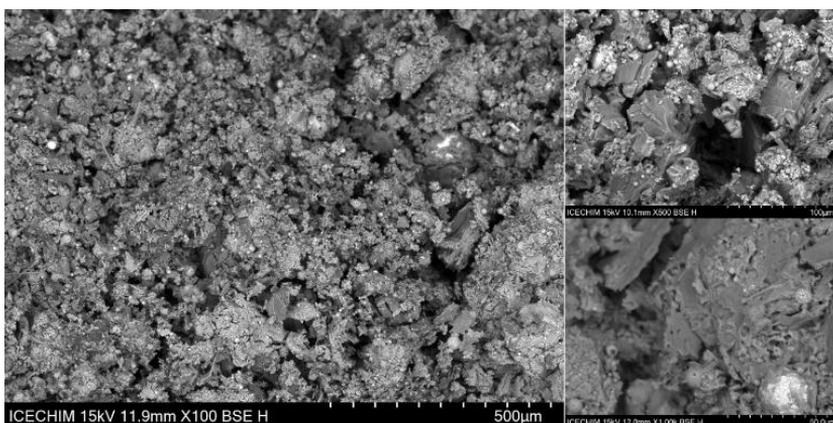


Figure 9. Microstructural analysis of sample S8-PL

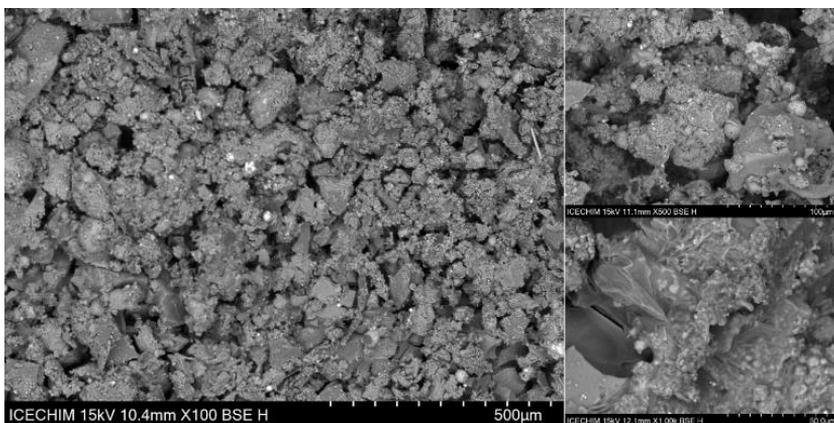


Figure 10. Microstructural analysis of sample S8-Ta

The microstructural analysis of the obtained samples confirms the positive influence of the addition of waste wood particles on the structure of these composites. However, amounts higher than 10% lead to the formation of a granular, inhomogeneous structure with reduced mechanical properties.

Conclusions

This study indicates that incorporating wood particles can enhance mechanical properties under certain conditions. It is recommended to maintain the wood content at 10% by weight. Besides the quantity of wood, the particle size also plays a significant role. The composite containing 10% wood demonstrated better homogeneity compared to those with higher wood content.

Excessive amounts of wood negatively impacted the integrity of the matrix, as the porous wood particles absorbed the alkaline activator, resulting in a weaker structure and inadequate dissolution of the raw materials. Additionally, fine particles, such as wood flour, contribute to denser composites by filling in pores, while larger particles help reduce the occurrence of cracks due to a bridging effect. However, if the bonding between components is weak, higher porosity may be observed.

Microstructural analysis at high magnifications revealed that composites with a 10% wood addition exhibited improved interfacial bonding and a lower number of microcracks. In contrast, composites with 20% wood showed multiple delamination zones and weak bonding between the matrix and the reinforcing particles, which accounted for their reduced mechanical performance.

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