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# Influence of Microparticles on Setting Time and Micromorphology of Coal Ash Geopolymers

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**Abstract.** Geopolymers are inorganic materials with zeolites-like microstructure and mechanical properties similar to those of Ordinary Portland cement materials [1]. However, their properties are highly depending on the constituents (raw material and activator) characteristics, as well as, on the activation particularities (mixing parameters, curing time and temperature etc.). In order to explore the influence of partial replacement of coal ash with two types of fine aggregates (glass and sand microparticles) on micromorphology and setting time, four types of geopolymers were developed. The evaluations were performed by means of electronic microscopy and Vicat method. According to this study, the coal ash replacement with glass microparticles results in an increase in the initial and the final setting time, while the replacement of coal ash with sand particles show a significant decrease. Moreover, the microstructural analysis shows different behaviour, during activation, of the studied microparticles. The surface of the glass microparticles reacts in the alkaline environment, while the sand particles did not. Therefore, the increase of initial and final setting time can be correlated with the dissolution of Si-O from the glass particles, during geopolymerisation.

## 1. Introduction

Nowadays, the main challenge in new materials developing is strongly related to the environmental impact of raw materials exploitation and sustainability of obtaining technology. A promising class of oxidic materials, ideal to substitute Ordinary Portland Cement (OPC) materials and to eliminate the negative effects produced by those, are geopolymers [1, 2].

A geopolymer is rich in aluminium and silicon material with porous structure and characteristics similar to OPC materials [3]. However, since as source of raw material for geopolymers, any powder rich in aluminium and silicon oxides can be used, globally multiple types of wastes have been identified as compatible [4]. Furthermore, the lack of standardization and the wide range of parameters that can vary in each geopolymer has lead to multiple unknowns which are hard or impossible to predict or simulated. In order to develop specific standards for these materials, different scenarios (composition) must be designed and analysed. According to previous studies [5, 6], multiple factors influence the properties of geopolymers. As can be seen in Table 1, those are related to the raw



materials characteristics, the type of the activation solution, the curing stage parameters and the parameters of the mixing stage.

**Table 1.** Factors that influence the properties of geopolymers [5, 7].

<b>Raw material</b>	Particle size distribution
	Humidity
	Chemical composition
<b>Activator solution</b>	Solid to liquid ratio
	Sodium silicate to sodium hydroxide ratio
	Sodium hydroxide molar concentration
<b>Mixing stage</b>	Mixing time
	Mixing speed
<b>Curing stage</b>	Curing time
	Curing temperature

In case of geopolymers with reinforcing element few more factors must be taken into consideration, such as particles pozzolanicity or particles adhesion to the matrix [6].

According to Lateef N A et al. [8] the particle size distribution has a direct effect on the compressive strength, structure and permeable void ratio of geopolymers. For example when a fly ash source with the average particle size of 42.3  $\mu\text{m}$  has used a value for compressive strength of 22 MPa was obtained in case of 28-days age samples, while after the fly ash was milled and sifted (the average particle size decreases to 4.78  $\mu\text{m}$ ) the compressive strength increased up to 67.3 MPa. For the same type of particles, the absorption decreased up to 21% and the microcracks resulted from the curing process are fewer in number.

The quality of the final structure and its properties results mainly from the uniformity and strength of the tetrahedral structure formed by the Si-O-Al system [9]. Any impurity or chemical element (except Si, O and Al) from the raw material, especially the calcium content, can lead to the formation of defects in the structure that significantly influence the final properties of the geopolymers [10]. To evaluate the influence of calcium content on the setting time of geopolymers, P. Chindaprasirt et al. [11] replaced different percentages of ash with Portland cement type I, which contains a high content of calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) and calcium oxide ( $\text{CaO}$ ). According to the study, the increase in calcium content in the composition causes a sharp decrease in setting time regardless of the percentage of replacement.

The chemical activator or activation solution plays a vital role in initiating the geopolymerization process. In general, a strongly alkaline environment is required to increase the hydrolysis of aluminosilicate particles contained in the raw material, therefore its concentration has a pronounced effect on the mechanical properties of the geopolymers [12]. On the other hand, the dissolution of  $\text{Si}^+$  and  $\text{Al}^+$  species during geopolymer synthesis depends mainly on the NaOH concentration, where the dissolution capacity of silicon and aluminum oxides is controlled by the NaOH concentration and mixing parameters [13]. Considering the fact that for geopolymers manufacturing alkaline solution with high concentration are used, adequate health and safety conditions must be introduced during the obtaining process [14-16].

Somna et al. [17] studied the compressive strength of fly ash-based geopolymers, hardened at ambient temperature by changing the NaOH concentration from 4.5 M to 16.5 M. It was observed that by increasing the NaOH concentrations from 4, 5 to 9.5 M, there is a significant increase in the compressive strength of the samples. However, the increase of NaOH concentrations from 9.5 to 14 M positively influences the mechanical properties of the final product, but in a reduced. The influence on the compressive strength of the alkaline activator concentration is significant due to the degree of dissolution of  $\text{Si}^+$  and  $\text{Al}^+$  which directly affects the formation of sialates bonds. Above 16.5 M a decrease is observed due to the excess of hydroxide groups which determines the precipitation of the aluminosilicate gel at early ages.

Conventional methods of manufacturing geopolymers involve mixing the solid component, the raw materials rich in aluminum and silicon with the reinforcing elements if any, with a liquid component, a strongly alkaline solution. There are several studies about the ideal mixing procedure, however, the most common method involves mixing the solid components, if there are several, as well as the liquid ones, followed by mixing the liquid component with the solid one [18]. Another method is to mix the materials rich in silicon and aluminum with the sodium silicate solution, and after a few minutes, the NaOH solution is introduced in the binder while mixing. However, the geopolymers obtained by the second method have lower mechanical strength properties, except for those based on fly ash [19]. After mixing the components, the resulting geopolymeric paste is poured into moulds, where it is to harden at room temperature or at a slightly high temperature. At the same time, according to the study conducted by Z. Yahya et al. [20], if after filling the moulds, the surface in direct contact with the atmosphere is covered with a foil, the evaporation rate of the water in the mixture is reduced, so the geopolymer will have a smaller number of cracks.

Up to know, the geopolymers successfully participated in multiple domains such as civil construction, water filtration, heavy metals encapsulation, heat resistant shields etc. due to their novel properties including corrosion resistance, compressive and flexural strength and high-temperature resistance [21-23, 25-27]. However, there are still a few challenges in developing geopolymers with different types of particles in the matrix. Therefore, this study aims to develop and analyse four types of coal-ash based geopolymers which contain one or two types of reinforcing elements.

## 2. Materials and methods

The obtained geopolymer was manufactured by mixing sodium silicate and sodium hydroxide solution (alkaline activator) with coal ash and reinforcing particles (solid component).

### 2.1. Materials

**2.1.1. Coal ash.** Coal ash is the main waste which results from coal combustion in power plants, these mineral powder rich in aluminium and silicon oxides ends by being deposited on waste areas near the cities powerplants. The coal ash used in this study comes from CET II - Holboca Iasi Romania ash dumps, which occupied an area of approximately 50 hectares in 2013. Due to the fact that the performance of coal ash in geopolymers is influenced by its characteristics (chemical composition, humidity, particle size distribution etc.). In order to remove the negative effects produced by the impurities with high dimensions from the coal ash, only the particles with the diameter lower than 80  $\mu\text{m}$  have been used. Moreover, the powder was dried at 120 until its mass remains constant for 30 minutes, i.e. humidity  $\approx 0$ .

According to ASTM C618-92a, the coal ash used in this study belongs to class F fly ashes because the sum of the three main oxides (silicon, aluminum and iron) is higher than 70% (eq. 1), as can be seen in Table 2.

$$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = 47.8 \pm 0.32 \% + 28.6 \pm 0.27\% + 10.2 \pm 0.95 \% = 86.6 \pm 1.54 \% \quad (1)$$

**Table 2.** Indigenous fly ash oxide chemical composition.

Oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>x</sub> O <sub>y</sub>	Ca O	K <sub>2</sub> O	MgO	TiO <sub>2</sub>	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	Oth. 1
% , weight	47.8 0	28.60	10.20	6.40	2.40	2.00	1.30	0.60	0.40	0.30
Stat. error, %	0.32	0.27	0.95	0.77	0.71	1.09	1.81	0.63	0.24	-

<sup>1</sup> Sum of chemical elements lower than 0.1 %.

**2.1.2. Reinforcing particles.** Another mineral waste that results in large quantities, both from the food industry and from the civil construction industry is glass. This inert material does not decompose

naturally, producing proven negative effects on the environment following storage in waste dumps [5]. Therefore, the use of glass waste in the manufacture of environmentally friendly materials has become a global concern [4]. Due to the ability to incorporate geopolymer paste, by the introduction of glass powder in the composition of these materials, a geopolymer with higher compressive strength can be obtained while an environmental problem is solved. As can be seen from Table 2, the glass powder, with particles smaller than 10  $\mu\text{m}$  in diameter, shows higher  $\text{SiO}_2$ ,  $\text{CaO}$  and  $\text{Na}_2\text{O}$  content, but lower aluminium oxide concentration.

**Table 3.** Glass powder oxide chemical composition.

Oxide	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_x\text{O}_y$	$\text{CaO}$	$\text{MgO}$	$\text{Na}_2\text{O}$	Oth. <sup>1</sup>
%, weight	70-71	1.5-2	0.8-1	9-11	2-3	12-14	< 0.1

<sup>1</sup> Sum of chemical elements lower than 0.1 %.

In order to study the behaviour of natural aggregates (sand), compared with waste (glass), two types of geopolymers with different percentage of sand particles have been obtained. As natural aggregates, a class of river sand with particles lower than 4 mm in diameter has been used.

**2.1.3. Activator solution.** As an alkaline solution for coal ash-based geopolymers activation, a mixture of sodium hydroxide and sodium silicate solutions was used. The NaOH solution was prepared at a molar concentration of 10 by dissolving high purity NaOH flakes (98%) in distilled water 24 hours prior use. The sodium silicate solution with a density of 1.37  $\text{g}/\text{cm}^3$  and pH lower than 11.5 has been commercially acquired (Scharlad Company, Barcelona).

**2.1.4. Sample preparation.** The samples mixture was prepared according to the BS EN 196-1:1995 by means of a variable speed mixer. In order to increase the homogeneity of the samples, firstly, the solid component was poured into the mixer and stirred in a dry state for 4 min. Secondly, the liquid component was added gradually and mixed for 10 min. until a homogeneous binder was obtained. The mix proportion of liquid and solid component of each sample are presented in Table 4.

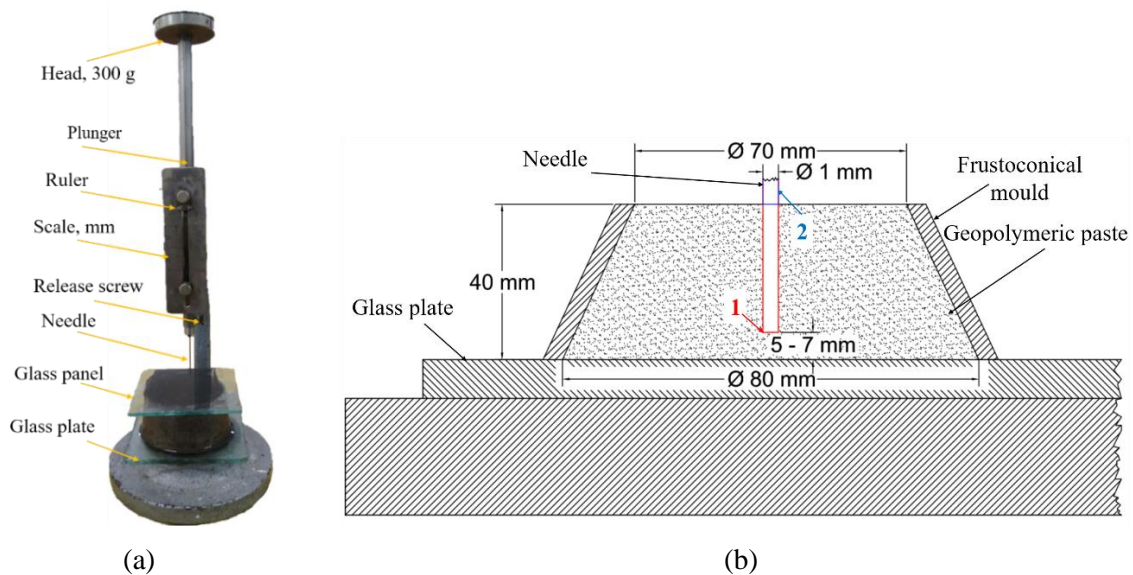
**Table 4.** Samples components mix proportion.

Sample	$\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio % weight	Liquid to Solid ratio % weight	Solid component, % weight		
			Coal ash (CA)	Glass powder (GP)	River sand (RS)
100CA			100	0	0
70CA_30GP			70	30	0
30CA_70RS	1.5	1.5	30	0	70
15CA_15GP_70RS			15	15	70

The micromorphology analysis was realized on samples cured at 70 °C for 8 hours and aged for 28 days in normal environmental condition.

## 2.2. Methods

The chemical composition of raw materials and obtained samples were measured by a Bruker S8 Tiger Wavelength Dispersive X-ray Fluorescence (Bruker, Karlsruhe, Germany) instrument. The samples micromorphology has been analysed using a scanning electron microscope FEI, model QUANTA 450 FEG (FEI company, WA, USA) in environmental SEM condition. The setting time of the geopolymer paste was evaluated by Vicat test according to ASTM C 191-04a. The test was conducted at room temperature ( $22 \pm 2$  °C) with a Vicat apparatus (Figure 1.a).



**Figure 1.** Vicat apparatus for setting time measurement: (a) components; (b) schematic representation of measurement principle.

The Vicat method consists in testing the penetration resistance, with a flat tip needle with a diameter of 1 mm, of a geopolymer sample that was poured into a frustoconical mould specific to the device used (Figure 1. b). After placing the mould on a glass plate, it is filled with freshly obtained geopolymer paste (immediately after mixing the components) and covered with a glass panel that prevents water evaporation. Every 30 minutes, the penetration resistance shall be checked by removing the glass panel and releasing the needle using the release screw. The needle is pushed into the sample surface under the action of its weight, the weight of the plunger and that of the head (300 g). Depending on the depth of penetration, it is determined whether the two points (initial or final) specific to the setting time have been reached. After checking the depth on the scale, the needle is removed from the paste by lifting the rod, further, the sample is covered with the glass panel for the next 30 minutes.

### 3. Results and discussion

#### 3.1. Chemical composition analysis

As can be seen in Table 5, the chemical composition of the obtained geopolymers shows high differences between the concentration of the main elements (Si, Na, Fe and Al). Except for oxygen, the element with the highest concentration in all samples is silicon.

**Table 5.** Chemical composition of the studied samples.

Sample	Element	Si	Na	Fe	Al	Ca	K	Ti	Mg	O
100CA	[%], wt.	21.46	10.98	3.14	8.86	2.25	1.05	0.20	0.73	balance
	error, [%]	0.33	0.79	0.27	0.70	0.49	1.10	1.83	1.72	
70CA_30GP	[%], wt.	22.06	12.27	2.47	6.83	3.24	0.99	0.27	1.00	
	error, [%]	0.34	0.74	0.28	0.75	0.52	1.14	1.89	1.91	
30CA_70RS	[%], wt.	25.72	10.83	3.08	4.79	2.20	1.13	0.29	0.80	
	error, [%]	0.36	0.65	0.26	0.80	0.72	1.08	1.72	1.81	
15CA_15GP_70RS	[%], wt.	26.41	11.01	2.30	4.42	2.83	0.81	0.73	0.90	
	error, [%]	0.35	0.69	0.27	0.77	0.62	1.11	1.79	1.72	

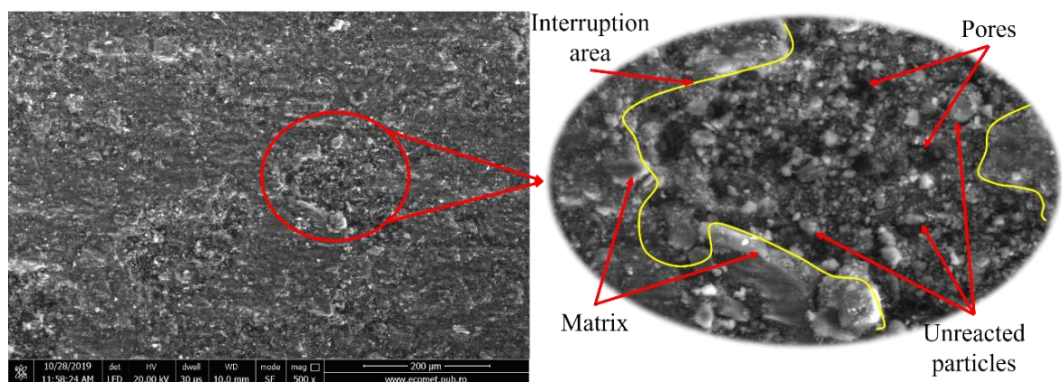
Sample 100CA contains the highest concentration of aluminum (Al) and iron (Fe), but the lowest concentration of silicon (Si). However, the sample 70CA\_30GP exhibit a decrease of more than 20% in

the concentration of Fe and Al, but due to the replacement of 30% of coal ash with glass powder, the calcium concentration in the sample increases up to 30%. The chemical composition of the 30CA\_70RS sample shows an increase of approximately 17% in the Si concentration, but by replacing 70% of the amount of coal ash with sand particles, the Al and Ca content decreases by 46%, respectively 3%. In the case of 15CA\_15GP\_70RS sample, the increase of Si concentration is of approximately 17% while the decrease of aluminum content reaches 50%. Therefore, by decreasing the coal ash content from the composition of the geopolymers, the aluminum content decrease while the silicon content increases.

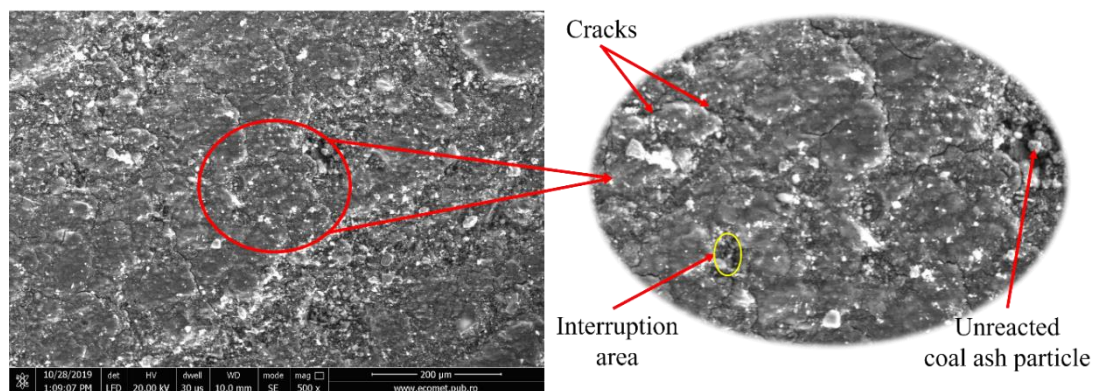
According to the chemical composition, the 100CA sample has better adhesion properties due to the ratio between the Si and Al concentration of approximately 2.4 which corresponds to a geopolymer with linear 2D structure [27, 32]. However, by increasing the percentage of reinforcing particles, the Si content increases, therefore, those sample will exhibit lower flexibility but higher hardness and compressive strength.

### 3.2. Micromorphology analysis

From the microstructural point of view, the samples show a homogeneous structure with high compactness of the matrix that depends on the type or the percentage of reinforcing particles. Moreover, all samples show unreacted coal ash particles, but the sample 100CA (Figure 2) shows the highest number, therefore, in some areas the matrix continuity is interrupted. Additionally, the SEM micrograph at 500X also highlights the cracks formed during the curing process, due to the fast evaporation of water. However, due to the self-healing capacity of geopolymers, which is related to the continued geopolymerisation reaction between unreacted particles and the activation solution from the gel pores, some cracks are repaired over time. At the same magnification, the sample 70CA\_30GP (Figure 3) shows a homogeneous structure, with a high degree of ash dissolution.

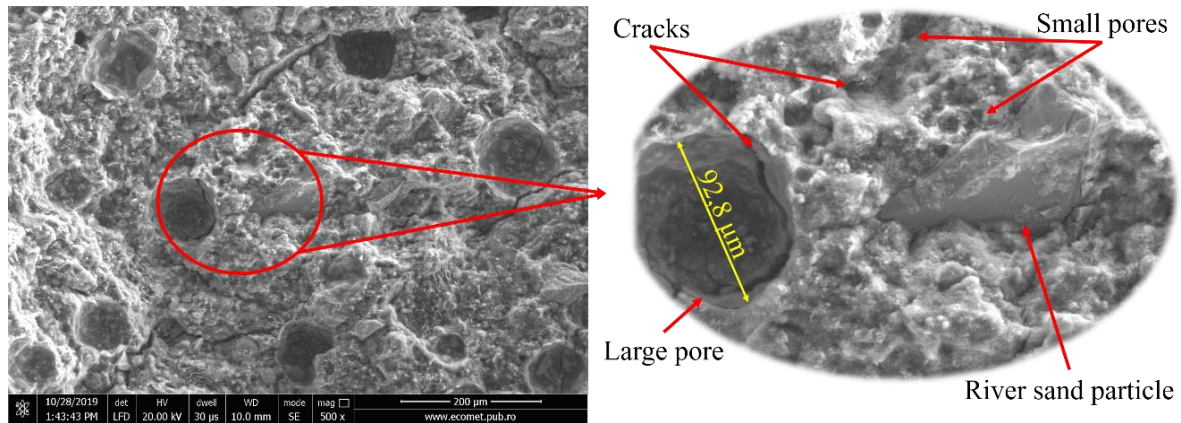


**Figure 2.** The micromorphology of 100CA sample.

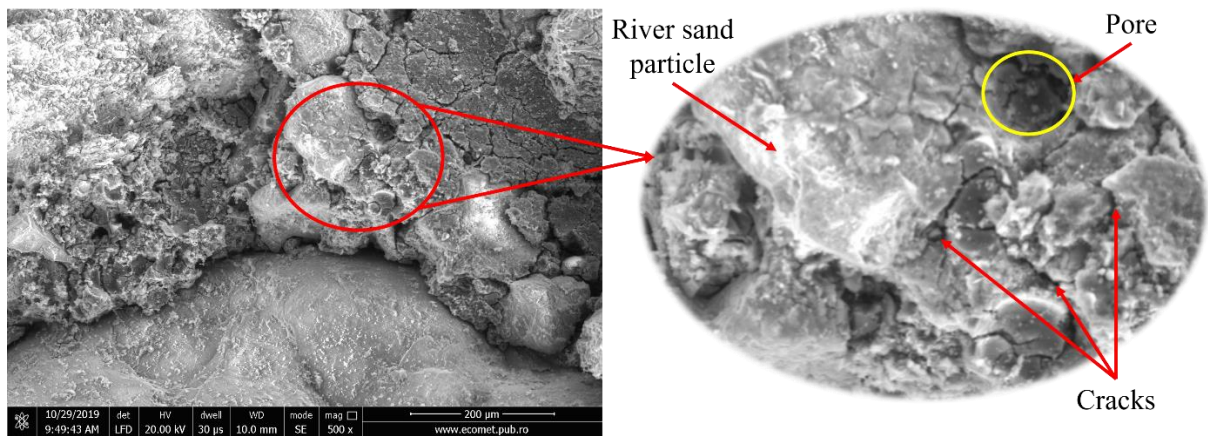


**Figure 3.** The micromorphology of 70CA\_30GP sample.

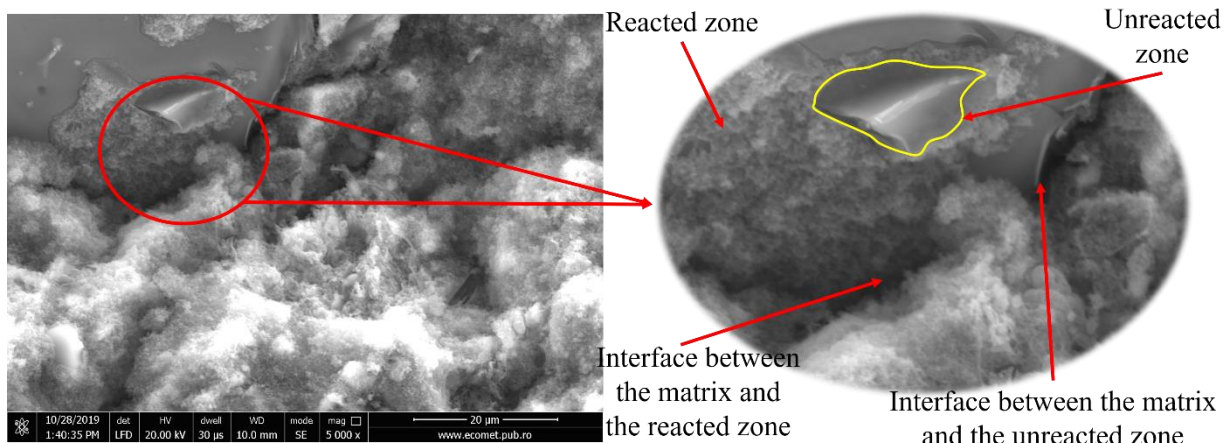
The micromorphology of the geopolymers with river sand (Figure 4) respectively river sand and glass powder (Figure 5) exhibit a high increase in the number of large pores, besides, the number of unreacted particles and small pores significantly decrease. The effect is produced by the high dimensions particles which block the air bubbles evaporation from the geopolymeric paste, during curing.



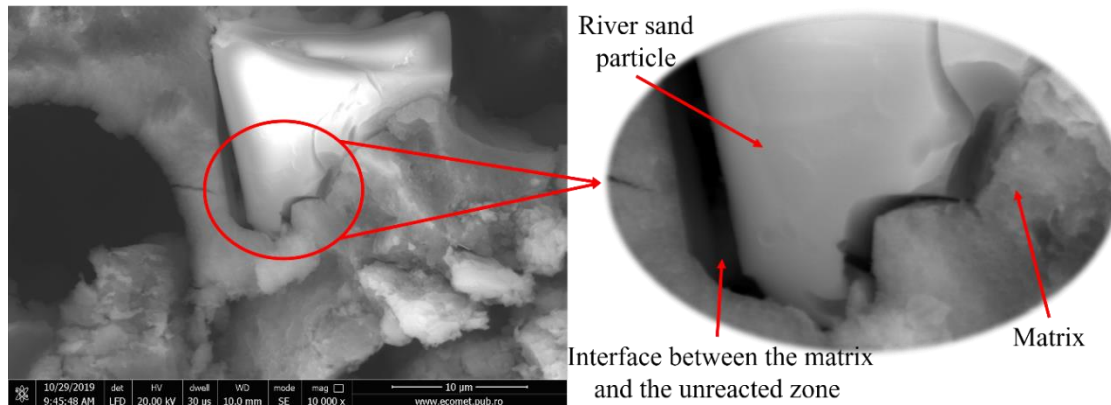
**Figure 4.** The micromorphology of 30CA\_70RS sample.



**Figure 5.** The micromorphology of 30CA\_70RS sample.



**Figure 6.** 70CA\_30GP sample micromorphology at high magnification (10KX).



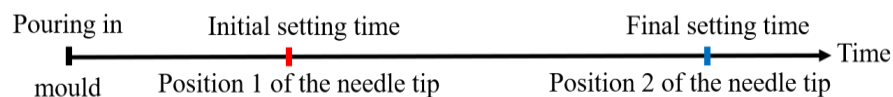
**Figure 7.** 30CA\_70RS sample micromorphology at high magnification (10kX).

In environmental SEM condition, the interface between the reinforcing particles and the matrix can be studied at high magnification. As can be seen from Figure 6, the surface of the glass particles reacted in the alkaline medium (reacted zone), therefore, the matrix adheres strongly to their surface. In the case of the samples with river sand as reinforcing elements (Figure 7) the matrix does not adhere to the surface of the particles, thus, a clear delimitation can be observed between the matrix and the sand particles.

### 3.3. Setting time

The interpretation of the setting time for these materials is similar to the equilibrium diagram specific to alloy systems, where the cooling liquid line provides information about the temperature at which the first crystallization germs appear, and the solidus line highlights the temperature at which the transition to solid-state occurs for the entire volume of material [24]. In the case of geopolymers, the first point (position 1 of the needle tip) refers to the initial setting time, which is specific to the loss of plasticity of the mixture and the passage of a certain period of time after mixing the components. The second point (position 2 of the needle tip) refers to the final setting time and this is characterized by the total time elapsed from the mixing of the components to the actual hardening of the sample (Figure 8). In other words, the initial setting time is determined by the time elapsed from the pouring in the mould of the geopolymeric paste until the needle penetrates to a depth of only (5 ÷ 7) mm in the geopolymer paste (Figure 1.b). The final setting time is specific to the moment when the needle no longer penetrates the surface of the sample.

The coal ash-based geopolymers analysed in this study shows an initial setting between 4.78 and 6.88 hours and a final setting time between 22.95 ÷ 25.00 hours at ambient temperature (Table 6).



**Figure 8.** Time distribution of the points specific to setting time.

**Table 6.** Setting time of coal ash based geopolymers.

Sample	100CA		70CA_30GP		30CA_70RS		15CA_15GP_70RS	
	a	b	a	b	a	b	a	b
Test 1	290	1420	410	1500	280	1380	350	1410
Test 2	300	1420	410	1490	290	1370	350	1400
Test 3	290	1430	420	1510	290	1380	370	1430
<b>Average, [min.]</b>	<b>293</b>	<b>1423</b>	<b>413</b>	<b>1500</b>	<b>287</b>	<b>1377</b>	<b>357</b>	<b>1414</b>

a – initial setting time, [min.]; b – final setting time, [min.].

It is essential that the initial setting time to be long enough to cover all the cast-in-place operations. However, according to the obtained results, the initial and final setting time of the samples with glass powder is higher than that of 100CA and 30CA\_70RS samples. In case of this materials calcium silicate hydrate (C-S-H), calcium aluminate silicate hydrate (C-A-S-H) and sodium aluminate silicate hydrate (N-A-S-H) are formed during geopolymerisation. As reported in previous studies the compounds that include calcium have lower setting time because the dissolution rate of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  species is lower compared to the dissolution rate of  $\text{Ca}^{2+}$ . Therefore, the setting time of the samples with glass powder should be lower, but this inversion can be explained through the dissolution of high content of  $\text{Si}^{4+}$ , as a consequence of the glass particles reaction in the alkaline environment, that results in a high initial and setting time.

#### 4. Conclusions

Four types of geopolymers have been obtained by activating the coal ash with a solution of sodium silicate and sodium hydroxide. In order to evaluate the influence of reinforcing elements of setting time and micromorphology, two types of particles have been introduced in the geopolymers matrix.

The microstructural analysis confirms a different behaviour, during geopolymerisation, between the glass powder and the river sand particles. According to this study, the glass particles react partially in the alkaline environment while the matrix adheres strongly. However, the river sand particles didn't react in the described condition showing a clear delimitation between their surface and the matrix of the samples.

Due to the fineness of the glass powder, the  $\text{Si}^{4+}$  content from their surface is dissolved in high concentration by the alkaline solution. Therefore, the setting time of the samples with glass particles (70CA\_30GP and 15CA\_15GP\_70RS) show higher initial and final setting time, even with a higher Ca content.

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