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Parametric Studies Regarding the Development of Alkali-Activated Fly Ash-Based Geopolymer Concrete Using Romanian Local Raw Materials †

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Abstract: Current research and development policies in the field of building materials, in the context of sustainable development, have the main objectives of increasing the safety and performance of the built environment at the same time as reducing pollution and its negative impact. Today, the idea that the sustainable city of the future should meet human needs and maintain a higher quality of life is worldwide unanimously accepted. The aim of this paper is to present results regarding the production of alkali-activated fly ash-based geopolymer concrete, a new, alternative material, produced using local available raw materials from Romania.

Keywords: fly ash; alkali-activation; geopolymer materials; sustainable development; advanced materials

1. Introduction

Both circular economy and sustainable development are concepts that focus on resource efficiency, which implies a complex waste management process with a high degree of recycling and recovery. Designing resilient, sustainable buildings, by using recycled materials that can replace some or all of the cement, leads to the development of a sustainable, low-carbon economy. Production of large volumes of cement/concrete is directly associated with environmental problems—cement production being responsible for about 5–8% of the total carbon dioxide emissions [1,2], therefore, awareness towards the possible production of alternative materials is rising. Demand for concrete, hence for cement, is constantly growing, especially in highly developed countries [3,4], which means that alternative binders are urgently needed to meet the needs of millions of people, without compromising the CO_2 levels of the atmosphere.

With the best available technologies for the production of Portland cement, industry estimates that currently, CO_2 emissions resulting from its production could decrease by a maximum of 17% (even through the use of alternative fuels, optimizing the amount of cement in concrete, recycling, etc.) [5]. As the population needs for energy continue to increase, it will lead, at global level, to an increase in the production of fly ash as by-product from the energy industry [4,6].

When partially used in the production of concrete, together with Portland cement, fly ash reacts with calcium hydroxide during the moisturizing process, in the presence of water. The use of materials derived from industrial by-products on a larger scale in the manufacture of concrete, has increased with the development and the increasing demand of producing special concrete (i.e., self-compacting concrete, high-strength concrete, high performance concrete, self-healing concrete) [7–10].

Studies shown that the large amount of fly ash resulting from the energy industry in Romania can create new opportunities to use this waste as a substitute for Portland cement in the production of new materials [11]. The alkaline activation of the fly ash as raw material represents a procedure able to generate its solidification when mixing it with a certain type of alkaline activator and creating a new binding material. When incorporating aggregates, new building materials could be obtained, as an alternative to traditional concrete and cement-based composites [12–14].

For a material used in the construction industry, mechanical behavior is a basic property, which makes it optimal for a specific application. Since geopolymer materials are a novelty in this area, and most of the studies are presented on a case-by-case situation, compressive strength is an important factor. The mechanical behavior of geopolymers varies depending on the used materials and the production methods they are subjected to [15]. In order to obtain a geopolymer material with a good compressive strength, the type and the molar ratios of the oxides in the source material, the pH of the alkaline solution, and the solubility of the source material in alkaline activator should be particularly taken into account [3,6,11,16].

It has been shown that the compressive strength of the alkali-activated geopolymer materials increases, in general, with the increase in the concentration of the specific alkaline activators [17–19]. A higher concentration of the NaOH solution may produce stronger Si-O-Si bonds and improve the dissolution of source materials in the presence of the activators [17,19,20]. The optimal alkaline concentration also varies by a large number of conditions and factors that must be taken into account in the mix-design.

The properties of alkali-activated geopolymer materials depend mainly on the important factors that could affect the development of this type of material and include: The characteristics of constituent materials, alkaline activators, heat treatment regimes, etc. It is obvious that, due to the multitude of factors that could influence the geopolymerization reaction, the specificity and conditions met for each proposed mixture generate case-by-case characteristics. However, in most cases, studies have shown that the properties of geopolymer materials were similar or even better than those of ordinary Portland cement concrete, when similar tests underwent [4,11,21,22].

Based on preliminary results obtained on alkali-activated geopolymer paste by using Romanian local raw materials [11,23], the aim of this paper is to present results regarding the production of alkali-activated fly ash-based geopolymer concrete, a new, alternative material, produced by using local available raw materials from Romania and to study the parameters that affect the mechanical properties of the material.

2. Materials and Methods

Worldwide, research on alkali-activated geopolymer materials exists, but their production differs greatly, due to factors that mainly influence this type of material. Alkali-activated geopolymer concrete mixtures were developed based on studying the literature and starting from a rigorous selection of source components that will be used to produce this type of material. By choosing relevant research in the literature, the preliminary mixtures were produced based on the variation of the parameters influencing them, from the point of view of the specific ratios, as well as their chemical composition. The evolution of the geopolymer concrete mix design was developed based on the observations collected in the experimental research and initial practical assessments, the fresh state appearance and the performance of the materials at a certain time. By modifying the variables and ratios of materials used in the production of geopolymer paste (alkaline liquid to fly ash ratio, molar concentration of the NaOH solution, Na_2SiO_3/NaOH ratio), several necessary data were obtained on the parameters that influence this type of material [11,23].

2.1. Fly Ash as Raw Material

Fly ash (FA) is the by-product obtained by electrostatic or mechanical precipitation of the pulverized particles resulting from the coal-fired combustion gases of the furnaces in power plants. As a fine powder, consisting mainly of spherical shaped glass particles [24], some certain characteristics show significant differences when coming to their chemical composition. The high variation of the chemical composition, leading to different performance when it is used as binding material for producing alkali-activated fly ash-based materials and represents an essential aspect for the general development of the concept [11,23,24].

In order to study the transition from alkali-activated geopolymer paste to geopolymer concrete, low-calcium fly ash from a Romanian power plant was used in this study, focused on the development and the comparative analyses of the mechanical properties of the material [11]. Its chemical properties are presented in Table 1.

Table 1. Chemical composition of fly ash used in the production of alkali-activated geopolymer materials, measured by X-ray fluorescence [wt.%].

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	P_2O_5	TiO_2	Mn_2O_3	L.O.I
FA	54.32	22.04	9.02	5.85	2.48	0.20	0.54	0.16	0.86	0.06	3.05

It can be seen from Table 1 that the fly used in the current study has low L.O.I. values, which means that it has low carbon content. As well, the Si/Al molar ratio of the fly ash batch is approximately 2, which makes it suitable for the production of alkali-activated geopolymer materials, due to the Si-O-Al-Si bonds that could be developed further in the mixture [25].

2.2. Alkaline Activator

The alkaline activator used for this experimental study was a combination of sodium hydroxide solution (NaOH) and sodium silicate solution (Na_2SiO_3). The sodium silicate solution was purchased from the local market with the following chemical composition: SiO_2 = 30%, Na_2O = 14% and H_2O =5 6%. Two types of sodium hydroxide solutions were prepared by dissolving NaOH flakes (98% purity–SH1) and NaOH pearls (99% purity–SH2) into water until the desired concentration of the solution was achieved (8 M and 10 M).

2.3. Aggregates

Natural aggregates, granular class 0/4 mm (S) and 4/8 mm (CA), (sand and coarse aggregate) were used for the production of the alkali-activated geopolymer concrete.

2.4. Preliminary Design of Alkali-Activated Fly Ash-Based Geopolymer Concrete

Geopolymer concrete mixtures were developed based on studying the literature and starting from a rigorous selection of source materials that will be used to produce this type of material. The preliminary mixtures were produced based on the variation of the parameters influencing them, from the point of view of the specific ratios, as well as their chemical composition. The evolution of the geopolymer concrete mix design (Table 2) was developed based on the observations collected in the experimental research and initial practical assessments, the fresh state appearance and the performance of the materials at a certain time [11]. The raw materials (fly ash and aggregates) were conditioned at (20 ± 2) °C, until constant mass was reached. The alkaline activator (Na_2SiO_3 and NaOH solutions) was prepared 24 h prior mixing. The mixing technology for the production of the AAGC samples included the following sequences (Figure 1): The sand (0/4 mm) and the coarse aggregates (4/8 mm) were mixed homogenously for 30 s; the established amount of fly ash was then added and the raw materials (fly ash + aggregates) were mixed together for another 30 s; the alkaline activator was added during 90 s over the homogenous mixture; after adding the total alkaline activator quantity, the mixing was continued at low speed for 3 min. During the entire mixing process, the workability of the mixture has been observed. The samples were then placed in 40 mm × 40 mm × 160 mm molds and heat cured at 70 °C for 24 h. A glass film was placed on top of every mold in order to prevent excessive water release from the mixtures; after demolding, the specimens were stored in the climatic chamber at the temperature T (20 ± 1) °C and relative humidity RH (60 ± 5)%.

Table 2. Alkali-activated geopolymer concrete mix-design (wt.%).

AA/FA	Fly Ash	$Na_2SiO_3/NaOH$	Na_2SiO_3	NaOH	NaOH Molarity	S 0/4 mm	CA 4/8 mm
1.0	1.0	1.0 1.5 2.0 2.5	0.25 0.30 0.33 0.36	0.25 0.20 0.17 0.14	8 M	0.5	0.5
1.0	1.0	1.0 1.5 2.0 2.5	0.25 0.30 0.33 0.36	0.25 0.20 0.17 0.14	10 M	0.5	0.5

Figure 1. Alkali-activated fly ash-based geopolymer concrete: (**a**) Fly ash; (**b**) Coarse aggregate–granular class 4/8 mm (CA); (**c**) Sand-granular class 0/4 mm (S); (**d**) Mixing of the aggregates; (**e**) Mixing of the raw materials (FA + aggregates); (**f**) Final mixing sequence; (**g**) Casting of the mixtures; (**h**) Geopolymer concrete samples after demolding.

2.5. Testing Methods

All the tests performed on the alkali-activated geopolymer samples were conducted at the age of 7 days. The flexural and compressive strength testing of the alkali-activated geopolymer concrete samples were performed using three prismatic specimens for each type of alkali-activated fly ash-based geopolymer mixtures; the mean value of the results was considered relevant for the data interpretation. The testing method was in accordance to EN 196-1 [26]. Early age testing at 7 days was considered relevant for the comparative evaluation, as previously experiments proved that generally, the fly ash-based geopolymer binder reached most of its compressive potential by this age [11,23,24].

3. Results and Discussions

In order to study the physico-mechanical parameters, the grouping of the alkali-activated fly ash-based geopolymer concrete mixtures was made according to the type of raw materials used and is symbolized as follows: AAGC FA SHx yM SS, where AAGC–Alkali-Activated Geopolymer Concrete; FA–fly ash-based; SHx–type of sodium hydroxide solution; yM–molarity of the sodium hydroxide solution; SS–sodium silicate solution.

3.1. AAGC Fresh-State Properties

The general observation was that when the Na_2SiO_3/NaOH solution ratio increased, the workability of the mixtures also increased. Instead, the functionality of the mixtures with NaOH solution 10 M was considerably lower than those using 8 M NaOH solution. The high content of NaOH solids in the solution directly affects the workability of the mixes. This does not mean that NaOH is the only parameter affecting the functionality of the fresh fly ash-based geopolymer material; however, it is of great importance. Another important observation during the preparation of the samples concerned their behavior during and after mixing. For the apparently dry mixtures, the ones with high Na_2SiO_3/NaOH ratios, at the beginning of the mixing, the material was not homogenous and the sample seemed to be an almost completely dry mixture. However, a significant aspect of these dry mixtures was that, by vibrations induced by a jolting table for compacting the samples, the mixtures formed a continuous and cohesive mixture.

3.2. AAGC Hardened-State Apparent Density

The apparent density of the derived AAGC mixtures was carried out in accordance with the standard EN 12390-7 [27] at the age of 7 days. The results obtained are presented in Figure 2.

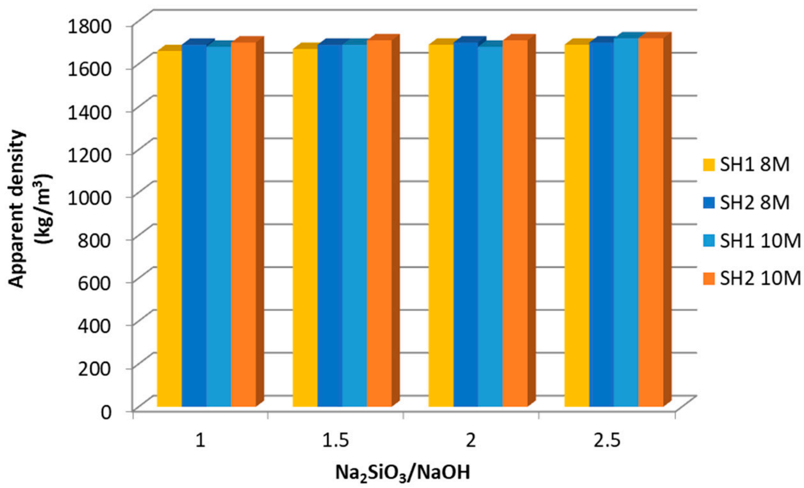


Figure 2. AAGC hardened-state apparent density.

Selection of the appropriate binder to aggregate ratio helps to ensure the proper workability and the performance characteristics of the hardened geopolymer material. As presented in Figure 2, the average values of geopolymer concrete apparent density are in the range between 1690 kg/m^3 and

1720 kg/m^3 for all the Na_2SiO_3/NaOH ratios, for both NaOH solution concentrations and for both NaOH types.

3.3. AAGC Mechanical Properties

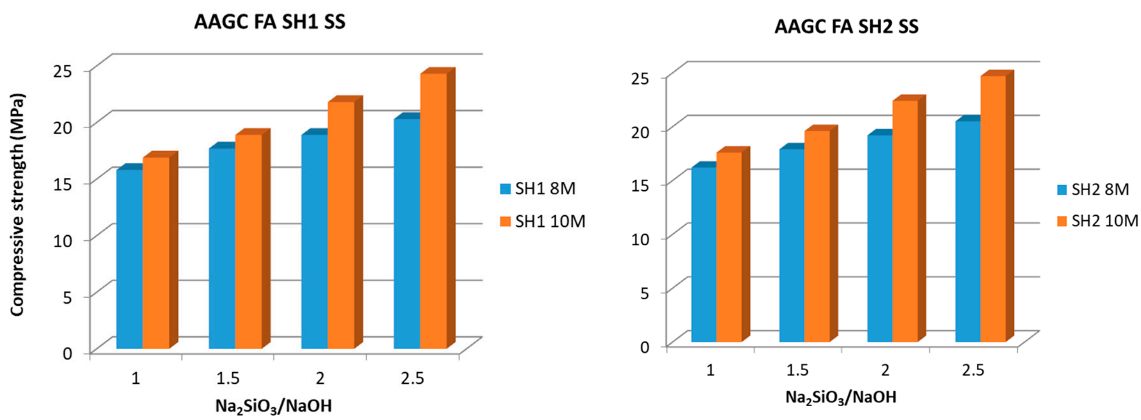
The flexural strength, respectively the compressive strength, of the derived AAGC mixtures was carried out in accordance with the standard EN 196-1 [26]. The test age of the specimens was 7 days, as it has been shown that, due to the heat treatment to which the mixtures are subjected, they reach mechanical properties at young ages [11,23,24]. The results obtained are presented in Table 3.

Table 3. Alkali-activated geopolymer concrete mix-design (wt.%).

Mixture	Na_2SiO_3/NaOH	Flexural Strength (MPa)	Compressive Strength (MPa)
AAGC FA SH1 8 M SS	1.0 1.5 2.0 2.5	2.9 3.0 3.7 4.2	15.8 17.7 18.9 20.3
AAGC FA SH1 10 M SS	1.0 1.5 2.0 2.5	3.0 3.2 3.9 4.4	16.9 18.9 21.8 24.3
AAGC FA SH2 8 M SS	1.0 1.5 2.0 2.5	2.8 3.1 3.8 4.1	16.2 17.9 19.2 20.5
AAGC FA SH2 10 M SS	1.0 1.5 2.0 2.5	3.1 3.8 3.8 4.2	17.6 19.6 22,4 24.7

3.4. Influence of the Alkaline Activator on the Mechanical Properties

The alkaline activator plays a vital role in initiating the geopolymerisation process. Generally, a strong alkaline environment is required to increase the surface hydrolysis of the aluminosilicate particles present in the raw material, while the concentration of the chemical activator has a pronounced effect on the mechanical properties of geopolymers [28]. Results regarding the influence of the alkaline activator on the mechanical properties of the geopolymer concrete, in terms of NaOH solution type and concentration, but also in terms of Na_2SiO_3/NaOH ratio, are presented in Figure 3.



(**a**) (**b**)

Figure 3. Influence of the alkaline activator on the compressive strength of the alkali-activated geopolymer concrete: (**a**) AAGC FA SH1 SS mixtures; (**b**) AAGC FA SH2 SS mixtures.

From the results obtained and presented in Table 3 and Figure 3, it can be seen that the compressive strength of the alkali-activated geopolymer concrete increased as the concentration of the NaOH solution increased, from 8 M to 10 M. In the case of AAGC FA SH1 SS mixtures, the increase in the compressive strength of the samples, as the molar concentration of the NaOH solution increased was between 7% (for mixtures with Na_2SiO_3/NaOH ratio of 1.0 and 1.5) and 20% (for mixtures with Na_2SiO_3/NaOH ratio of 2.5). For AAGC FA SH2 SS mixtures, the increase in the compressive strength was between 9% (for mixtures with Na_2SiO_3/NaOH ratio of 1.0 and 1.5) and 21% (for mixtures with Na_2SiO_3/NaOH ratio of 2.5).

Results also show that the type of sodium hydroxide (NaOH) did not have a major influence on the final compressive strength of the alkali-activated geopolymer concrete mixtures. To the extent that their purity is comparable, the results are similar, with less than 1.5% for all the Na_2SiO_3/NaOH ratios, and they fall under the measurement uncertainty. One of the reasons users could choose a lower purity of sodium hydroxide might be the economic ones, with a lower purity sodium hydroxide being cheaper than a higher purity one.

As an influencing parameter of the mechanical properties of the alkali-activated geopolymer materials, the Na_2SiO_3/NaOH ratio, the results show that, as the ratio increases, the compressive strength of the AAGC mixtures increased. This is corroborated with the fact that, although the workability of the mixtures was lower, their mechanical properties increased. The result regarding the evaluation of the influence that this parameter has on the mechanical properties of the AAGC mixtures is presented in Figure 4.

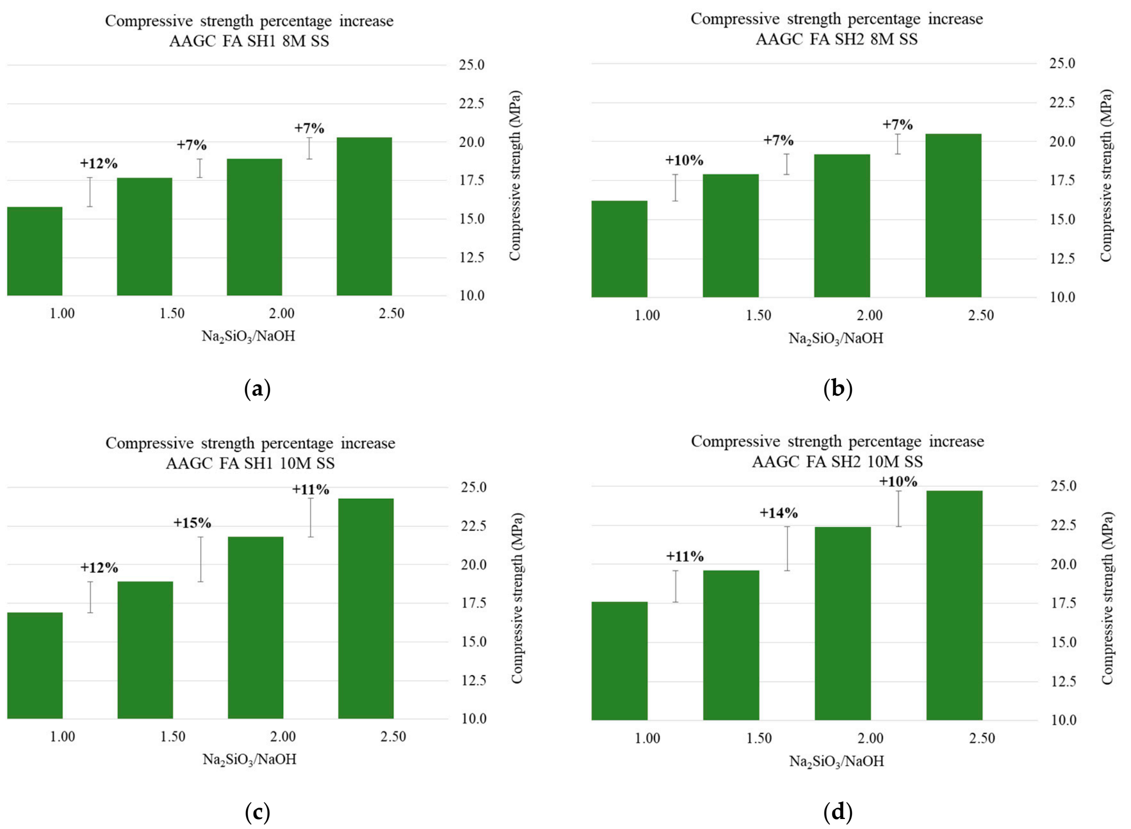


Figure 4. Na_2SiO_3/NaOH ratio influence on the compressive strength of the alkali-activated geopolymer concrete: (**a**) AAGC FA SH1 8 M SS mixtures; (**b**) AAGC FA SH2 8 M SS mixtures; (**c**) AAGC FA SH1 10 M SS mixtures; (**d**) AAGC FA SH2 10 M SS mixtures.

For all AAGC analyzed mixtures it can be seen that, for a Na_2SiO_3/NaOH ratio of 2.5, the highest compressive strength values are obtained with a percentage increase of 26–28% for AAGC FA 8 M SS mixtures and 40–44% for AAGC FA 10 M SS mixtures. The increase of the compressive strength of the samples can also be observed from the point of view of molar concentration of the NaOH solution. Results show that the increase in the compressive strength behavior is identical for a similar molar concentration of the NaOH solution. This remark is corroborated with previous statements that have shown that, since purity of the NaOH used to produce the solution is comparable, not only the increase in the compressive strength is similar, but also the behavior in the increase of this parameter is similar.

4. Conclusions

The aim of the current experimental research was to analyze the development of alkali-activated geopolymer materials produced by using raw materials from Romania and to study the parameters influencing the mechanical properties of the material, namely, the compressive strength. By using the same fly ash as raw material, by keeping the alkaline activator to fly ash mixing ratio constant and varying the Na_2SiO_3 to NaOH solution ratio and also the NaOH solution concentration, the effect of the two main parameters on the compressive strength of the alkali-activated fly ash-based geopolymer binder could be studied.

The results obtained regarding the influence of the sodium silicate solution to sodium hydroxide solution ratio (Na_2SiO_3/NaOH) highlight the importance of this parameter regarding the final results of the compressive strength of the alkali-activated fly ash-based geopolymer concrete. The evolution of the compressive strength values for the analyzed mixtures, at the age of 7 days, is different and, as this ratio increased, the values of the mechanical properties of the material also increased. For all AAGC mixtures that were analyzed, it can be seen that, for a Na_2SiO_3/NaOH ratio of 2.5, the highest values for the compressive strength were obtained.

Presented results also show that for the development of alkali-activated fly ash-based geopolymer materials, the influence of the sodium hydroxide solution concentration is a very important parameter, not only in the geopolymerization process, but also in the mechanical properties of the material. The solubility of fly ash increased as the concentration of the NaOH solution increased, therefore generating a denser material, with higher compressive strength results of the samples.

The workability of the mixtures was also assessed, considering the variation of the two studied parameters that influence the AAGC samples. Thus, it can be seen a clear interdependence between the variation of the workability and compressive strength of the mixtures. As the Na_2SiO_3/NaOH solution ratio increased, the workability of the mixtures also increased. Instead, the functionality of the mixtures with NaOH solution 10 M was considerably lower than those of 8 M NaOH solution, which, in the case of the compressive strength values, can be considered inversely proportional (higher ratio–lower workability–higher compressive strength).

As geopolymer concrete materials start to become more desirable in comparison to traditional OPC materials, both due to economic and ecological drivers and their demonstrated increased mechanical performances, the initial and long-term properties of these materials must be well understood, so they could be properly designed in order to obtain the effective mechanical properties. Results presented in the current paper emphasize that the specific evaluated parameters which could influence the compressive strength of the geopolymer materials, have an important effect on the behavior of the material.

Further studies will be performed using different types of raw materials and alkaline activators, in order to confirm the present conclusion. Furthermore, research regarding the viability of the material, through the optimization of the mixtures, will be carried out, thus opening up new opportunities for the development of this type of material in the future.

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