POROUS FLY ASH-BASED GEOPOLYMER USABLE AS AN UNCONVENTIONAL CONSTRUCTION MATERIAL

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Abstract: Geopolymer foam with thermal insulation properties was made by foaming the alumino-silicate mixture composed of fly ash and clay brick waste activated with alkaline activator (water glass and sodium hydroxide in aqueous solution). Other mixture components were expanded perlite as siliceous additive, usual fine aggregate (quartz sand), and less frequently used surfactant (olive oil). The main characteristics of the geopolymer foam were: low density, low thermal conductivity, and relatively high compressive strength. The residual materials contributed to low costs and the complete replacement of ordinary cement led to the significant reduction of CO_2 emissions.

Keywords: geopolymer foam, alumino-silicate binder, coal fly ash, hydrogen peroxide, alkaline activator.

1. INTRODUCTION

Under the conditions of the world ecological and economic crisis exhibited since the end of the last century, the conception of a new type of unconventional building material using as raw material different alumino-silicate by-products of industrial processes for complete substituting the Portland cement is justified. The industrial manufacture of cement involves great fossil fuel consumptions and implicitly, a high amount of carbon dioxide (CO_2), the main component of greenhouse gases, released into the atmosphere. The inventor of this new type of material called "geopolymer", J. Davidovits defines it as "an inorganic alumino-silicate polymer" [1] manufactured by synthesizing either natural materials (clay, kaolinite, and calcined kaolinite) or industrial by-products (coal fly ash and granulated blast furnace slag) with alkaline activators (sodium hydroxide-NaOH, sodium silicate, also known as water glass-Na_2SiO_3, sodium sulfate-Na_2SO_4, etc.) in aqueous solution [2] for the polymerization reaction to take place.

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Except for excellent mechanical, physical, and thermal properties (high mechanical strength, low shrinkage, resistance to acid-corrosion, fire, and high temperature) [2], the CO_2 emission in the making process of geopolymer is much lower compared to the manufacture of the common Portland cement [3, 4]. By increasing the temperature of the process of making the geopolymer material, the polymerization reaction is intensified influencing the improvement of its mechanical properties [1].

In numerous international papers [5-10], increasing the mechanical strength of geopolymer concrete was the main objective. Coal fly ash, granulated blast furnace slag and rice husk were used as alumina-silicate binders completely replacing the ordinary Portland cement. Also, natural alumina-silicate materials (mainly metakaolin) were used in experiments presented in these works. To activate the binder and facilitate the polymerization reaction, alkaline activators (water glass and NaOH aqueous solution) were added to the material mixture. The fresh geopolymer workability was improved by using a superplasticizer based on naphthalene. The compressive strength of geopolymer concrete had higher values compared to the conventional cement concrete. Curing techniques (at room temperature, with hot air or with steam at temperatures below 80 ºC) consisting of keeping the fresh geopolymer under these conditions for 3-12 days have contributed to the further increase of the mechanical strength reaching values of over 30 MPa.

The papers mentioned above were focused on aspects related to obtaining geopolymer concretes with high level of compression resistance. Making cellular geopolymers with low heat conductivity and high heat insulation features represent less frequent concerns highlighted in the literature. The present paper aimed precisely at these technical objectives, so that further, information provided by the literature in this field is presented. The making techniques of high-strength or porous geopolymers are based on similar natural or residual inorganic alumino-silicate materials that completely replace ordinary Portland cement, activated with alkaline solutions of water glass and NaOH and contain fine and coarse aggregates. Both techniques have low CO_2 emissions in the atmosphere during the manufacture of raw materials, being environment friendly. The main difference is the addition of a foaming agent or the use of mechanical foaming in order, to form a porous geopolymer structure in the case of manufacturing geopolymers with thermal insulation properties.

Fly ash-based porous geopolymers were produced using hydrogen peroxide (H_2O_2) as pore supplying agent [11]. This type of material can contribute to reducing the loss of thermal energy from the inside of building to the outside using residual materials. The addition of H_2O_2 influences the properties of the porous geopolymeric material (porosity, thermal conductivity, and compressive strength). The geopolymer specimens had thermal conductivity below 0.107 $W \cdot m^{-1} \cdot K^{-1}$ and the corresponding density of 560 $kg \cdot m^{-3}$, i.e. thermal insulation properties. The geopolymer was prepared with a mixture composed of 67 % metakaolin (as a natural alumino-silicate material) and 33 % coal fly ash (as an industrial alumino-silicate by-product). A mixture of hydrated water glass and NaOH aqueous solution was used as alkaline activators.

The paper [12] analyzed the influence of raw material mixing parameters (alkaline concentration, activator ratio, metakaolin/activator ratio) on the heat conductivity of geopolymers based on metakaolin. The mixed effect of the blowing agent (H_2O_2) and the surfactant known as Tween 80 (polyethylene glycosorbitan monooleate) on physical features, compression resistance and peculiarities of cells was researched. According to these scientific investigations, it has been found that the metakaolin-based geopolymer with the highest compression resistance of 33 MPa, relatively low bulk density of 1680 $kg \cdot m^{-3}$, porosity of 18 %, and heat conductivity of 0.40 $W \cdot m^{-1} \cdot K^{-1}$ was made using 10M-concentration of alkaline solution, the ratio of activator around 1 %, and metakaolin/activator ratio at the value of 0.8. Experimentally, it has been shown that the decisive factor of resistance was the activator proportion, while the metakaolin/activator ratio decided the value level of the heat conductivity. By adding H_2O_2 and surfactant, the cellular geopolymer reached compression resistance with values from 0.4 to 6 MPa. Bulk density was within the limits of 471-1212 $kg \cdot m^{-3}$, porosity in the range of 36-80 %, and heat conductivity had values between 0.11-0.30 $W \cdot m^{-1} \cdot K^{-1}$. The microstructural aspect was determined by the amounts of H_2O_2 and surfactant used in the same time. The variation range of H_2O_2 clearly influenced the dimension and cells distribution uniformity. By adding the surfactant, cells dimension stabilization has been observed at low H_2O_2 proportions (< 0.75 wt.%), which faded at its high proportions. Even by increasing the porosity in the case of using high contents of H_2O_2 and surfactant, the heat conductivity did not obviously decrease due to the coalescence of pores.

The biomass ash constituted by calcium phosphate was applied as a reactive filler in a geopolymer composite based on metakaolin to produce strong and light boards with thermal insulation properties [13]. The material was

obtained by alkaline activation of a mixture of metakaolin and biomass ash in the mass ratio 1:1 and foamed with H_2O_2 (5 wt. %). The results showed the density of 310 $kg \cdot m^{-3}$ and the thermal conductivity of 0.073 $W \cdot m^{-1} \cdot K^{-1}$ at 30 ºC, the compressive strength being 0.6 MPa. The material is applicable for self-bearing thermal insulation partitions or lightweight cores for thermal-structural sandwich panels.

The work [14] followed the effect of polypropylene fibers on the mechanical strength and thermal conductivity of geopolymer foams based on coal fly ash. Water glass and NaOH in aqueous solution were used as an activator of the alumina-silicate binder (coal fly ash). The preparation of geopolymer foams was mechanically made by mixing the foaming agent with distilled water at high pressure. The foams were added to the geopolymer mixture in proportions of 40 % and 60 %, respectively. Low amounts of polypropylene (between 0-0.50 % of the amount of coal fly ash) were added. The strength of geopolymer foam increased with the increase of polypropylene content. It has been observed that the polypropylene fiber can improve the tensile strength of the geopolymer. The addition of fibers affected the thermal conductivity however it had values similar to those of the conventional lightweight concrete.

Another recipe for the experimental preparation of metakaolin geopolymer concrete is shown in [15]. The metakaolin as a natural alumino-silicate material was used as a binder and was activated with 10M sodium hydroxide and sodium silicate solution in the mass ratio 2:3. Sodium perborate (NaH_2BO_4) was chosen as a foaming agent (in a variable proportion between 0.5-2.0 wt. %) being firstly mixed with metakaolin and then with the alkaline activator aqueous solution forming a geopolymer paste, which was poured into silicon molds. The curing process at temperatures below 60 ºC took place for 24 hours, followed by aging the samples at room temperature and 75 % humidity for 28 days. Geopolymer foam features varied depending on the NaH_2BO_4 proportion between 0.5-2.0 % as follows: bulk density decreased from 1077 $kg \cdot m^{-3}$ up to 750 $kg \cdot m^{-3}$, porosity reached 67.6 % from 54.7, compression resistance registered decreasing from 6.7 to 5.2 MPa, and heat conductivity decreased up to 0.218 $W \cdot m^{-1} \cdot K^{-1}$ starting from 0.325 $W \cdot m^{-1} \cdot K^{-1}$.

According to [16, 17], the porous structure of geopolymer can be obtained by different foaming methods: the addition of a surfactant (rosin, sodium dodecyl sulfate, sodium lauryl ether sulfate, proteins, etc.), the use of foaming agents (H_2O_2, NaH_2BO_4, sodium hypochlorite, etc.) and other methods. Different additives can be added to the geopolymer matrix (polypropylene fiber, carbon nano-tubes, nano-silica etc.). Also, organic materials as expanded perlite or cellulose fibers are ecologically and economically adequate for this purpose. The predominant raw material used in [16] was metakaolin. To increase the mechanical strength and workability, 10 % of sand for aggregate was introduced into the metakaolin mass and mixed. Aqueous solution of water glass was added over 8M NaOH (2.5:1 ratio) and mixed. H_2O_2 (3 wt. %) and aluminum powder (low proportion) were used as foaming agents. The use of an additive in the form of expanded perlite led to a significant decrease of the thermal conductivity and density, practically without changing the compressive strength.

Experimental results of making cellular geopolymer based on coal ash using sodium silicate (Na_2SiO_3) known also as water glass as a blowing agent and washing powder as a surfactant were presented in [18]. The surfactant addition in very low weight proportions (between 0.1-0.5 %) influenced the fineness of geopolymer porous macrostructure, the pore size decreasing with the surfactant amount increasing. Thermal conductivity was reduced from 0.32 $W \cdot m^{-1} \cdot K^{-1}$ to 0.27 $W \cdot m^{-1} \cdot K^{-1}$, while compressive strength of the cured (for 28 days) porous material increased from 4.21 MPa to 4.82 MPa. The increase of the surfactant ratio up to 0.5 % led to increasing the fire resistance of the geopolymer compared to its resistance with only 0.1 % surfactant or without this addition.

The current paper aimed to make geopolymer foams with excellent thermal insulation properties and acceptable compressive strength values using alumino-silicate materials from industrial by-products (coal fly ash) and recovered wastes from building demolitions (residual clay bricks) activated with alkali solution, the foaming agent being hydrogen peroxide (H_2O_2). The work originality is the exclusivity use of the combination of recycled alumina-silicate materials as a binder substituting the common Portland cement.

2. METHODS AND MATERIALS

2.1. Methods

Two main principles were the basis of applying the geopolymer foam making method: (i) activating the alumino-silicate binder mixture with an alkaline solution to facilitate the polymerization reaction and (ii) foaming the mixture with a foaming agent. The alkaline solution was chosen by the authors from several variants mentioned above, being composed of aqueous solution of water glass and 8M NaOH, the weight ratio between the two components being 2.5:1. To obtain the porous structure of the geopolymer, H_2O_2 was adopted as a foaming agent, used in most of the experiments presented in the literature. For the same purpose, perlite existing in nature in the form of siliceous amorphous volcanic rock was added. The expanded perlite [19] used in this experiment was obtained in Cosfel Actual SRL Company by expanding the rock rapidly heated in an adapted microwave oven to over 850 ºC [16] and has a significant influence in the foaming process of geopolymer.

By decomposition of the H_2O_2 solution in H_2O and O_2 according to the reaction (1), gases are released inside the alumino-silicate mixture mass, causing the formation of bubbles then turned into pores. The decomposition takes place slowly when exposed to light [20].

$$2H_2O_2 = 2H_2O + O_2 \quad (1)$$

From the category of surfactants recommended for geopolymer foam making processes, olive oil [21] was chosen. Added to the geopolymer paste in small proportions (0.1-0.5 %) it can improve the structure and fineness of the pores.

The preparation method included the following operations. The solid components (coal fly ash, clay, expanded perlite, and fine sand as aggregate) were dry mixed until a homogeneous mixture was obtained, then the alkali solution was added, the mixture being further mixed in a laboratory mixer for 20 min, forming a paste. The liquid foaming agent H_2O_2 (30 % concentration) and also the surfactant (olive oil) for the stabilization of foam were added to the geopolymer paste and mixed. The fresh mixture was poured into stainless steel molds and cured at room temperature for 24 hours. At the end of this time interval, the samples were removed from the molds.

2.2. Materials

The materials used in this experiment and their role have already been mentioned above. Coal fly ash, a by-product of coal combustion in thermal power station captured in the electro-filters, was provided by Paroseni-Romanian thermal power plant with the grain dimension under 200 μm, diminished by the grinding process and granulometrically separation by sieving at very low size (100 μm).

Residual clay brick was recycled from building demolition. Expanded perlite powder was prepared by thermal expansion of volcanic rock of perlite through heating at above 850 ºC in an adapted microwave oven and grinding in ball mill of expanded material at grain size below 130 μm. Quartz sand purchased from the market had a fine size of grain below 200 μm. The oxide compositions of fly ash, clay waste, expanded perlite, and quartz sand (wt. %) are shown in Table 1.

Table 1. Oxide composition of raw materials.

Composition	Coal fly ash	Clay brick	Perlite [19]	Sand [22]
SiO_2	49.8	59.0	70.7	97.9
Al_2O_3	23.5	28.9	13.0	1.1
Fe_2O_3	6.1	1.7	1.0	0.5
CaO	3.6	1.0	2.1	-
MgO	3.1	0.9	1.7	-
K_2O	4.0	2.3	4.3	0.3
Na_2O	1.6	1.2	3.5	-
TiO_2	1.1	1.3	-	-

According to the data in Table 1, coal fly ash and clay brick are silica and alumina-rich, therefore arealumino-silicate materials suitable for their transformation into geopolymer (according to Davidovits). Perlite is a silica-rich material, like the sand.

The H_2O_2 solution is available on the market in concentrations between 30-70 %. The concentration chosen by the authors was 30 %.

2.3. Characterization methods of geopolymer foam samples

All tests for characterizing the geopolymer samples were performed after finishing the curing process. The gravimetric method [23] was applied for measuring the density. The determination of heat conductivity at low temperature (30 ºC) was made using HFM 446 Lambda apparatus by applying the heat flow method (SR EN 1946-3:2004). 100 kN-hydraulic axial press machines (EN 826:2013) was used for measuring the compression resistance. Absorbing the water test (ASTM C1585-20) was applied by immersing the sample into distilled water. The microstructural feature of specimens was investigated with ASONA 100X Zoom Smartphone Digital Microscope. Determining the crystalline phases of the cellular geopolymer was made using X-ray diffractometer Bruker-AXS D8 according to EN 13925-2:2003 standard. Porosity was calculated by measuring true density (without pores) with the pycnometer and the apparent density [24].

3. RESULTS AND DISCUSSION

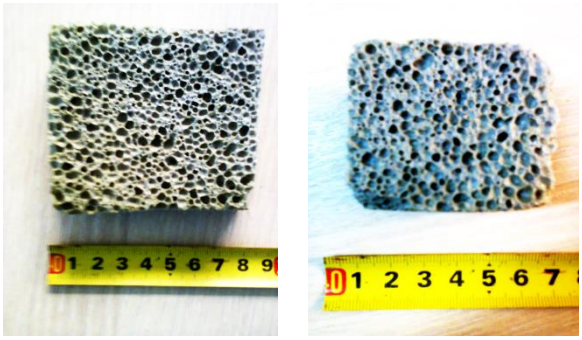
3.1. Results

Four making recipe variants were prepared for this experiment, in which coal fly ash was the basic alumino-silicate material together with clay waste, and perlite as a siliceous additive. River sand was the fine aggregate of the mixture. To these solid materials were added: olive oil as a surfactant, alkaline activator, and H_2O_2 as a foaming agent, all in a liquid state. Among all the mentioned components, sand, perlite, and the alkaline activator were kept at constant absolute values in the weight ratio 1.96: 0.56: 1, the others being variable in the four variants tested. Table 2 shows the material mixture composition (wt. %) corresponding to the four experimental variants.

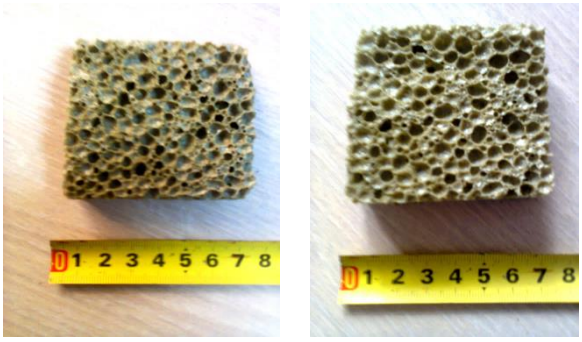
Table 2. Composition of experimental versions.

Material	Version (wt. %)			
	1	2	3	4
Fly ash	30.38	30.15	29.92	29.67
Clay	1.60	1.77	1.95	2.12
Sand	37.31	37.25	37.18	37.09
Perlite	10.66	10.64	10.62	10.60
Olive oil	0.04	0.07	0.11	0.18
H_2O_2	0.96	1.10	1.24	1.41
Alkaline activator	19.04	19.01	18.98	18.93

The appearance of the four geopolymer foams made in the current experiment is exposed in Figure 1. According to these images, the content increase of foaming agent (H_2O_2) and surfactant (olive oil) had obviously the effect of increasing the macroporosity of specimens and implicitly, their pore size. The specimens (c) and especially (d), have this feature.



a b



c d

Fig. 1. Appearance of the geopolymer foam samples:
a—variant 1; b—variant 2; c—variant 3; d—variant 4.

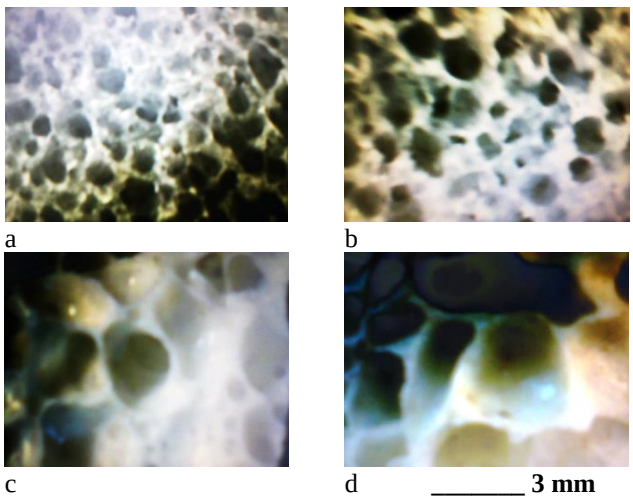
The result of measurements performed through the methods noted above are shown in Table 3.

Table 3. Physical-mechanical and thermal features of cellular geopolymer specimens.

Version	Apparent density ($kg \cdot m^{-3}$)	Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	Compression resistance (MPa)	Absorbing the water (vol. %)	Porosity (%)	Pore size (mm)
1	577	0.126	5.7	3.6	71.1	0.1-0.8
2	551	0.115	5.2	3.7	72.3	0.4-1.4
3	516	0.109	4.7	3.3	74.0	0.7-1.9
4	480	0.098	4.0	3.0	75.7	1.2-2.6

The characteristics presented in Table 3 indicate achieving specific samples of geopolymer foam. The apparent density has sufficiently low values (480-577 $kg \cdot m^{-3}$) and also the heat conductivity falls within a range of low values (0.098-0.126 $W \cdot m^{-1} \cdot K^{-1}$). The two characteristics confirm the existence of good heat-insulating properties of the making material. The expanded perlite addition in geopolymer did not influence significantly the compressive strength confirming the results presented in [16]. According to the data in the table, the porosity is acceptable under the conditions of the density level of the specimens and contributes to ensuring their heat-insulating character. The porous structure of the specimens shown in Figure 1, at least at the macro level, shows a very good homogeneity and uniformity of the pore distribution, which contributes to obtaining homogeneous thermal insulation.

The microstructural aspect of cellular geopolymers is presented in Figure 2.



a b

c d **_____ 3 mm**

Fig. 2. Microstructural aspect of cellular geopolymer specimens:
a—version 1; b—version 2; c—version 3; d—version 4.

The images in the above figure confirmed that the four samples microstructure is characterized by closed pores without the tendency to intercommunicate with neighbouring pores. The pore size is that indicated in Table 3, i.e. very low sizes in case of samples (a) and (b) and higher sizes in case of specimens (c) and especially (d).

The analysis of the crystalline phases in geopolymer foam identified mainly quartz and to a small extent mullite. The two crystalline phases which do not easily dissolve in the alkaline activator reduce the effectiveness of the polymerization process.

3.2. Discussion

The cellular geopolymer made in the current work was based on the innovative technique recently developed by the French researcher J. Davidovits, i.e. the use of alumino-silicate waste or industrial by-products having important pozzolanic properties, which allowed the complete abandonment of cement, considered for a very long time the perfect binder of concrete and mortar in technological terms, and the adoption of alumino-silicate materials as its substitutes. Davidovits' remarkable invention was made under the conditions of a deep ecological and energy crisis in which the production of cement provided excessive emissions of CO_2 into the atmosphere and required an huge consumption of fossil fuel.

The cellular geopolymer achieved in the present paper was produced with residual materials of alumino-silicate type: fly ash as a by-product of energy industry and clay brick waste recovered from buildings demolition. Perlite as a natural siliceous additive was processed into foam through the natural rock expansion process by the own technique of unconventional microwave heating carried out at Cosfel Actual SRL. Compared to other processes of making the geopolymer foam, the exclusive use of wastes and the perlite processing, which contributed to reducing costs, were original elements of the work.

The main raw materials used in the experiment presented in the paper were a by-product of the energy industry (coal fly ash) and a waste recovered from the demolition or remodeling of buildings (residual clay brick). Their activation as alumino-silicate materials was carried out (according to the invention) with an alkaline activator containing water glass (Na_2SiO_3) and NaOH in liquid solution. Additionally, expanded perlite powder was used as a silica-supplier. This commercially available material was previously produced by the authors' team through a own conception method by thermal expanding at 850 °C of volcanic rock of perlite in a ground state by applying the microwave irradiation heating technique. The efficient use of the conversion of microwave power into heat is a known technique, but practically not applied in the world at high temperatures, being jointly developed after 2016 by the Romanian companies Daily Sourcing & Research SRL and Cosfel Actual SRL and being one of the original elements of the current work.

Considering the objective of producing a cellular geopolymer, the H_2O_2 solution was adopted as a usual pore-supplying agent as well as olive oil with the role of surfactant, rarely used in the geopolymer manufacturing process. The application of clay brick waste as well as that of olive oil, little used in the usual manufacturing recipes, were other elements of the work originality.

Experimental results showed that the adopted solutions were viable for obtaining cellular products with thermal insulation properties for building walls (density between 480-577 $kg \cdot m^{-3}$, thermal conductivity between 0.098-0.126 $W \cdot m^{-1} \cdot K^{-1}$, and porosity between 71.1-75.7 %) and simultaneously with compression strength having sufficiently high values (4.0-5.7 MPa) similar to those of other geopolymer foams known from the literature. Also, the water absorption (between 3.0-3.7 vol. %) was within acceptable limits for a porous product of this type.

4. CONCLUSIONS

Given that the manufacture of concrete for construction includes materials whose processing involves high consumption of fossil fuel and high emissions of greenhouse gases (mainly CO_2), making geopolymers has become a necessity. The production of geopolymer is based on alumino-silicate materials (rich in silica and alumina) in the group which includes both natural materials and residual materials in the form of industrial by-products or wastes. The current work aimed at the production of a porous geopolymer with adequate heat-insulating properties using as an alumino-silicate binder a mixture of coal fly ash from energy industry and clay brick waste from building demolition activated with an alkaline activator composed of water glass and NaOH.

An additive with a major role in foaming the geopolymer and increasing the thermal conductivity (expanded perlite) was processed through an own unconventional heating method with microwaves in order, to foam the natural rock and then grind it. The mixture for the production of geopolymer foam was completed with a commonly used foaming agent (hydrogen peroxide H_2O_2), a common fine aggregate (quartz sand), and a surfactant known in the literature, but less often used (olive oil). The main features of the geopolymer foam samples were: density between 480-577 $kg \cdot m^{-3}$, thermal conductivity between 0.098-0.126 $W \cdot m^{-1} \cdot K^{-1}$, porosity between 71.1-75.7 %, and compressive strength in the range of 4.0-5.7 MPa.

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