Effect of corundum sand proportion on strength properties geopolymer mortar based on fly ash

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Abstract. The main objective of this study is to investigate the possibility of replacing the traditional construction aggregate, river sand, with corundum material and to evaluate the effect of the proportion of corundum sand on the strength properties of fly ash based geopolymer mortar. Electrocorundum (white) and fly ash from a thermal power plant in Skawina were used to make the samples. Different fractions of electrocorundum were used to prepare the geopolymer mass. A geopolymer based on fly ash and river sand was used as reference material. The results showed that the average compressive strength of fly ash and alumina-based materials increased (results ranging from 25.9 to 47.0 MPa) compared to the reference samples (20.7 MPa). The flexural strength results showed a weakening of the strength properties of the electrocorundum and fly ash-based samples (results ranging from 6.1 to 4.8 MPa) compared to the reference sample (7.0 MPa). Only the geopolymer, which in its composition contained a mixture of electrocorundum with several grain gradations, showed an increase in flexural strength (7.3 MPa), compared to the reference sample. Additionally, surface topography analysis of the geopolymer samples was performed by scanning electron microscopy. The whole tests carried out on the above types of material indicate that it can be used as a substitute for sand in geopolymer mortars.

1. Introduction
The fundamental natural resource used by the construction industry is river sand, used as fine aggregate during concrete production [1]. More than 50 billion of this material is exploited annually [2], and the continuous development of the construction industry and other sectors of the economy related to natural resources makes it become a scarce commodity. Over the years, the ratio of the extraction of this raw material to the rate of replenishment of its deposits worldwide has been gradually decreasing [3]. Unfortunately, not all types of this material are suitable for use in construction mortars - desert sand, due to its streamlined grain shape is more difficult to mix and is more easily washed out by water [4]. Therefore, it is not suitable for construction applications. The increasing demand for river sand and environmental concerns about its complete depletion have prompted the investigation of other relative sources that could provide a substitute for the above raw material in construction materials [1,5]. Aggregate is a rocky loose material (coarse or fine grained) of organic or mineral origin, mainly used in the production of concrete, building mortar or as a base for road surfaces [6,7]. The geometrical features, among others, the shape or grain size of the aggregate determine the subsequent properties of the entire concrete mix. The mixture of aggregates for concrete should contain both coarse and fine fractions in appropriate proportions. The fine aggregate stabilizes the whole concrete mix (affects the...
viscosity and workability of the mass), and the addition of coarse aggregate is to fill the volume of the concrete mix. The optimal composition of fractions is to ensure the lowest possible demand of the concrete on the cement slurry. Aggregate grain size is also related to another property, namely water holding capacity. It depends on its surface, roughness and shape. The smaller the grain of the crumb pile, the higher the water demand of the concrete mix. In the development of concrete formulations, a balance is sought between low water-carrying capacity (to achieve the lowest possible shrinkage) and good rheological properties and surface quality of the produced elements [8].

Cement is another most widely used material in the construction industry worldwide [9]. Along with sand, it is the primary component of concrete. The construction sector is the largest explorer of natural resources, which is associated with driving the global production of cement, used as the base material in this industry [10]. The 2020 data on global cement fabrication, shows that the volume of cement production reaches values of about 4 billion tons per year [11], and the production process itself is associated with high carbon dioxide emissions. According to the International Energy Agency, the cement industry is responsible for about 7% of global emissions of this gas into the environment [12].

Research carried out on geopolymers is aimed at verifying their ability to replace Portland cement in the construction industry [13]. The design process of a geopolymer mix is mainly based on its intended use and related performance criteria. The workability of the mix during its preparation and the compressive strength value of the bonded mortar are used as the main determinant [10]. Geopolymers are a class of amorphous, inorganic and aluminosilicate polymers whose properties depend on the type of base material used and the type and amount of activator used in their production [14].

Secondary raw materials or by-products from industrial processes can be used to make geopolymer materials. One of these is fly ash, which is a waste product of the coal combustion process [15,16]. Currently, some of these ashes are disposed of as an admixture material for concrete production to reduce their environmental impact. The durability and mechanical properties of concrete with part of the cement replaced with fly ash have already been studied. This has been shown to produce a material with high durability and strength [17-20]. It should be noted, however, that the development of the technology of production of geopolymers on the basis of ash itself would affect the increase in the use of this raw material, which is a by-product of combustion. At the same time, it would be a positive environmental aspect.

The optimum aggregate content in geopolymers is not yet precisely specified and is mainly based on the results of laboratory scale research work [21,22]. Based on their analysis, Joseph and Mathew proved that geopolymer concrete with proper content and fixed proportion of fine aggregate can have better mechanical properties compared to those of ordinary cement concrete [23].

The most commonly proposed application for geopolymers is to obtain various types of products for use in construction, including pavers, flagstones, curbs, facade panels, or bricks. These materials are also suitable for immobilizing hazardous waste (radioactive waste, asbestos) or toxic waste (mercury, lead, arsenic) [20,24-27]. The above group of binders is characterized by, among others: high compressive strength [28-30], resistance to acids, chlorides and sulfates [31-34], thermal resistance (up to about 800°C) [35-37] or good frost resistance [38,39]. Moreover, there is no corrosion of steel reinforcement in geopolymers and the material shows good adhesion properties with steel. The synthesis process of geopolymer materials itself consumes 2-3 times less energy than the production of Portland cement [20,40].

In this article, the possibility of replacing river sand with another aggregate in the form of electrocorundum sand in fly ash based geopolymer mortar is analyzed. Strength tests were conducted on the fabricated geopolymer materials and the surface topography of the specimens was observed.
2. Materials and sample preparation
In the present work, activities related to the fabrication of geopolymers based on fly ash and an additional aggregate in the form of noble electrocorundum were undertaken. The fly ash was obtained from the Skawina CHP Plant and the abrasive material from one of the Polish suppliers - the "Herubin" company (Dobra, Poland). The table below (Table 1) shows the fractions of white electrocorundum ordered for the production of geopolymer materials. Figures 1a, 1b, and 1c show photographs of each type of abrasive, taken with a JEOL JSM-6390LV scanning microscope. This material was chosen for this study because of the shape and size of its grains (similar to river sand), its chemical composition (more than 98% pure Al2O3), and its excellent mechanical properties of high hardness and low abrasiveness.

| Grain number (F) | Particle size [µm] from to |
|-----------------|--------------------------|
| 24              | 710 850                  |
| 80              | 180 212                  |
| 220             | 53 75                     |

Table 1. Grit sizes for electrocorundum according to FEPA 42-D-1984 and PN/M-59107

![Figure 1](image1.png)

**Figure 1.** Aggregates used to produce geopolymers; a) F24 electrocorundum, b) F80 electrocorundum, c) F220 electrocorundum, d) river sand

In addition, one of the aggregates needed to make the reference sample mixture was river sand (Swietochlowice area). Figure 1d shows grains of the above material taken with the same scanning microscope model. River sand is a basic raw material, necessary for the production of concrete mortars and geopolymer pastes, due to the morphological nature of the grains and its general availability. Table 2 shows the proportion of the different grain fractions of the above river sand.

| Grain diameter [mm] | Fraction content [%] |
|---------------------|-----------------------|
| >1.6                | 0.65                  |
| 1.6-1.0             | 2.94                  |
| 1.0-0.3             | 1.50                  |
| 0.2-0.3             | 85.25                 |
| 0.2-0.1             | 9.18                  |
| <0.1                | 0.48                  |

Table 2. Percentage of individual grain fractions in river sand
To prepare a suitable alkali activator for the geopolymerization process, a source of strong base, an aqueous silicate solution, and water are required. In the above work, technical sodium hydroxide in flake form with a density of about 2.13 g/cm$^3$, sodium water glass R-145 with a density of about 1.48 g/cm$^3$, and tap water were selected. The preparation of the 10M alkaline activator involved weighing an appropriate amount of technical sodium hydroxide and dissolving it in water, followed by the addition of sodium silicate. Both processes were accompanied by an exothermic reaction, so the resulting solution had to be cooled to ambient temperature (about 25°C).

The design of the geopolymer mortar was mainly based on selecting the appropriate raw materials and determining their proportions in the overall mixture. The dry ingredients were mixed with a laboratory mixer for about 5 minutes and then the alkaline solution was added, mixing everything thoroughly for 15 minutes until a homogeneous mass was obtained. On average, about 400 ml of alkaline solution was added per 1kg of dry ingredients. The obtained plastic mass was filled into sets of prismatic molds of 50 mm x 50 mm x 200 mm and cubic molds of 50 mm x 50 mm x 50 mm, and then placed on a vibrating table. The geopolymer concretes prepared in this way were cured for 24 hours in a laboratory dryer (LW 750 STD, Pol-Eko-Aparatura, Wodzislaw Śląski, Poland) at 75°C. Then, the samples removed from the molds, cooled to ambient temperature, were seasoned for 28 days, after which the mechanical properties were tested and the SEM microstructure was observed. Table 3 shows the composition of the prepared samples and their mass ratios.

| Sample | Solution | Composition                                  |
|--------|----------|----------------------------------------------|
| R      | 10 M     | Fly ash + sand (1:1) - reference sample      |
| P24    | 10 M     | Fly ash + electrocorundum F24 (1:1)          |
| P80    | 10 M     | Fly ash + electrocorundum F80 (1:1)          |
| P220   | 10M      | Fly ash + electrocorundum F220 (1:1)         |
| K      | 10M      | Fly ash + electrocorundum F24 + electrocorundum F80 + electrocorundum F220 (5:1:1:1) |

3. Research methods
Compressive strength tests were carried out on a testing machine MATEST 3000 kN with a speed 0.5 MPa/s, according to EN 12390-3 standard „ Testing of hardened concrete. Compressive strength of samples “. The tests were carried out on cubic samples with dimensions of 50mm x 50mm x 50mm.

The flexural strength tests were carried out similarly to the compressive strength tests, according to the EN 12390-3 on the MATEST 3000 kN testing machine with a speed of 0.5 MPa/s. These tests were carried out on 50 mm x 50 mm x 200 mm. The distance between the support points was 150mm.

The observation of the microstructure of the geopolymers was made using the scanning electron microscope (SEM) of the type JEOL JSM-6390LV with EDS on the breakthroughs of the samples after the compressive strength tests. Preparation of samples for the observation was to their previous sputtering a thin layer of gold by vacuum sprayer JEOL – JEE-4X. The samples were observed at different magnifications.

4. Results and discussions
4.1. Compressive strength
The results from compressive strength tests are presented in table 4. The measurements for each series were made on 10 samples.
Table 4. The results of compressive strength

| Sample | Compressive strength [MPa] |
|--------|----------------------------|
| R      | 20.7 ± 3.6                 |
| P24    | 25.9 ± 6.2                 |
| P80    | 50.8 ± 8.2                 |
| P220   | 28.9 ± 5.7                 |
| K      | 47.0 ± 6.2                 |

The average compressive strength value for material R (reference specimen) is about 21 MPa. This is the lowest average value obtained compared to all types of compression specimens. Compared to sample P, an increase in average compressive strength value was recorded for all geopolymers with the addition of alumina - P24 by about 25% and P220 by about 40%. The highest fracture toughness was demonstrated for material K and P80, for which the average values were 47.0 MPa and 50.8 MPa, respectively, which corresponds to an increase in compressive strength of about 130% and 140%, compared to the reference sample. The reference sample had the lowest standard deviation, which indicates very good repeatability of the results during the experiment. The P80 sample had the highest measurement uncertainty. The standard deviation of the other test materials remained constant.

4.2. Flexural strength

The results from flexural strength tests are presented in table 5. The measurements for each series were made on 5 samples.

Table 5. The results of flexural strength

| Sample | Flexural strength [MPa] |
|--------|-------------------------|
| R      | 7.0 ± 1.8               |
| P24    | 6.1 ± 0.3               |
| P80    | 5.2 ± 1.5               |
| P220   | 4.8 ± 0.6               |
| K      | 7.3 ± 0.5               |

The differences between the average values of the reference sample (P) and the composite material (K) are really small, up to a limit of 10%. However, the K material has the highest flexural strength of more than 7 MPa. The reference sample came in second with a flexural strength of 7 MPa. The average flexural strength of the geopolymer material with the addition of F24 electrocorundum is about 6 MPa. In specimens P80 and P220, the addition of electrocorundum affected the deterioration of flexural strength. Compared to the reference sample, the flexural strength decreased by about 35 % for the P80 material and by about 45 % for the composite based on fly ash and F220 electrocorundum. The P24 samples had the lowest standard deviation and kept the measurement results constant. The reference sample and sample P80 had the largest spread of measurement values. Material K and P220 had a similar trend of results - the standard deviation oscillated at a similar level in both cases.

4.3. Microstructural investigation

The basis for the study of the surface topography of the specimen and its chemical composition, was the material from specimens destroyed during the performance of strength tests.

Figure 2a and figure 2b shows the morphology of the reference material sample. The fly ash and sand based geopolymer is characterized by a compact and homogeneous structure with no obvious pores. The light-colored and irregular structures visible on the surface of the material are probably remnants of unburned coal. The spherical, regular particles embedded in the geopolymer matrix are fly ash grains that have not dissolved.
Figure 2. SEM images of geopolymer R: a) magnification 100x, b) magnification 600x

Figure 3a and figure 3b shows the microstructure of P24 geopolymer. The surface of the material is irregular, with numerous pores, whose size oscillates between 30 and 40 µm. At 600x magnification - Figure 3b, spherical, regular forms, connected with the matrix, can be seen. These are fly ash particles which have not degraded. Additionally, feathery shapes can be seen, located in numerous clusters on the surface of the material. It is likely that these are some kind of crystalline forms, but at present we do not have information on the structures formed.

Figure 3. SEM images of geopolymer P24: a) magnification 100x, b) magnification 600x

Figure 4a and figure 4b present the surface image of P80 geopolymer. The microstructure of P80 geopolymer is characterized by numerous pores, which oscillate around 50 µm. Irregular, sharply pointed structures of several tens of micrometers in size are the grains of electrocorundum. Undissolved fly ash grains with a rounded shape tend to bond poorly with the geopolymer matrix and, consequently, to lower mechanical properties.

Figure 4. SEM image of geopolymer P80: a) magnification 100x, b) magnification 600x

Figure 5a and figure 5b show the surface topography of sample P220. Taking a closer look at the microstructure itself, one can see numerous small pores of 5 - 10 µm, as well as their larger counterparts of around 50 µm. In addition to the spherical ash particles, which are bonded to the material, we can also see sharp-edged corundum grains measuring approx. 30 µm. At higher magnification, the feathery structures, which appeared earlier in the case of P24 material, are revealed. However, there are far fewer of them.
The last type of material analyzed is geopolymer K, shown in figure 6a and figure 6b. The structure of the material is more homogeneous than in the case of P24 or P80. However, a few pores of about 40 µm in size are present. In the case of this sample, the ash particles appear to be well solidified with the geopolymer matrix, although they have not dissolved. There is no gap (lack of cohesion) around them, which compromises the mechanical properties.

Microstructure and properties of geopolymers strongly depend on the nature of the initial raw materials even though the macrostructure characteristics of alumino-silicate-based geopolymers may appear similar, since it is related to the same silicon and aluminum bonding and the same gel phase binder [41]. Compressive strength measurements are used by many researchers as a tool to assess the success of geopolymerisation process. This is due to the low cost and simplicity of compressive strength testing, as well as due to the fact that strength properties is a primary measure of the utility of materials used in different applications of the construction industry [42].

5. Conclusions
Based on a review of the collected literature and an analysis of the results of individual experiments, the above work draws important conclusions about the properties of a fly ash-based geopolymer mortar and noble electrocorundum:

1. The good strength properties of electrocorundum and fly ash based geopolymers are related to the varying degree of aggregate grain size and grain shape.
2. The addition of aggregate in the form of homogeneous fraction of electrocorundum as a substitute for sand affects the deterioration of strength properties when tensile forces are applied to the material. This can probably be related to the failure to form a tight crumb stack in the geopolymer.
3. Addition in the form of homogeneous fraction of electrocorundum as a substitute for sand affects the improvement of strength properties when compressive forces are applied to the material. However, the best results are obtained by adding a mixture of noble aluminium oxide with different grain size (composed of F24, F80 and F220) - MK material or F80.
4. Addition in the form of differentiated fractions of electrocorundum material (composed of F24, F80 and F220 electrocorundum) contributes to a more uniform material structure. This is probably related to the formation of a tight crumb stack in the geopolymer.
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