



Basic Physical – Mechanical Properties of Geopolymers Depending on the Content of Ground Fly Ash and Fines of Sludge

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Abstract

The binding potential of fly ash (FA) as a typical basic component of building mixtures can be improved in mechanical way, which unfolds new possibilities of its utilization. This paper presents the possibilities of preparing the geopolymer mixtures based on ground ($d_m = 31.0 \mu\text{m}$) FA, used in varying percentages to the original (unground; $d_m = 74.1 \mu\text{m}$) one. As a modification, fine-grain sludge from the process of washing the crushed aggregates was used as filler in order to obtain mortar-type material. The basic physical-mechanical properties of mixtures are presented and discussed in the paper, focusing on time dependence. The following standard tests were executed after 2, 7, 28, and 120 days: density, total water absorption, flexural strength, and compressive strength. Ground FA provided for positive effect in all tested parameters, while incorporation of fine portion of sludge into the geopolymer mixture does not offer a significant technical profit. On the other hand, it does not cause the decline in the properties, so the environmental effect (reduction of environmental burden) can be applied through its incorporation into the geopolymer mixtures.

Key words: geopolymer; fly ash; washing aggregate sludge; grinding; strength; water absorption.

1 Introduction

Many attempts have been made to minimize the use of cement as a binder in concrete production. The use of geopolymer as a binder in concrete production not only results in reduced CO₂ emission because of the elimination of cement, but also valorizes the alumino-silicate industrial by-products to produce environment-friendly construction material [1].

Geopolymers are members of the inorganic polymers family. The chemical composition of the geopolymer material is similar to natural zeolitic material, but the microstructure is amorphous. The polymerization process involves a substantially fast chemical reaction of Si-Al minerals under alkaline condition, resulting in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds [2]. This alumina-silicate binding material is synthetized by alkali activation of solid alumina-silicate raw materials such as fly ash (FA),

ground granulated blast furnace slag, metakaoline, rice husk ash, etc. The alkali metal hydroxide and silicate solution are the standard activators [3–6].

The utilization of FA in the development of geopolymeric materials for construction purposes has been and continues to be the subject of many research studies [7–11]. The works done on geopolymer technology show a significant potential for its utilization in construction, particularly low-calcium FA [12–13]. The possibility of using low-calcium FA to produce geopolymers also has environmental benefits. Quite a large amount of FA is utilized for cement concrete production, but the high-calcium type of FA is required in this case. The calcium content of the FA is perhaps the best indicator of how the FA will behave in concrete, although other compounds such as alkalis (Na_2O and K_2O), carbon (usually measured as LOI), and sulphate (SO_3) may also affect the performance of the FA. High-calcium FA (> 20% CaO) may be produced from lignite or sub-bituminous coals and are comprised of calcium-alumino-silicate glass and a wide variety of crystalline phases in addition to those found in low-calcium FA. Some of these crystalline phases react with water. This, coupled with the more reactive nature of the calcium-bearing glass, makes such types of FA react more rapidly than low-calcium types and renders the FA both pozzolanic and hydraulic in nature. These FA types will react and harden when mixed with water due to the formation of cementitious hydration products. If the calcium content of the FA is high enough, it is possible to make concrete of moderate strength using the FA as the sole cementing material.

On the other hand, low-calcium FA (< 8% CaO) is invariably produced from anthracite or bituminous coals and is predominantly composed of alumino-silicate glasses with varying amounts of crystalline quartz, mullite, hematite and magnetite. These crystalline phases are essentially inert in cement concrete and the glass requires a source of alkali or lime (for example $\text{Ca}(\text{OH})_2$) to react and form cementitious hydrates. Such FA types are pozzolanic and display no significant hydraulic behaviour [14–15]. Therefore, it is very significant that some possibility of utilizing the low-calcium FA exists. Low-calcium FA has been successfully used to manufacture geopolymer concrete, wherein the silicon and aluminium oxides constituted about 80% of the mass, with a Si-to-Al ratio of about 2.0. The content of the iron oxide usually ranged from 10–20% by mass, while the calcium oxide content was less than 5% by mass [16].

The effect of the fineness of FA on geopolymer's properties is also widely discussed. Changes induced in materials during the mechanical treatment process include reduction in particle size, changes in particle morphology, increase in specific surface area, structural defects formation, decrease in crystallinity, and implied structural rearrangement. The most important consequence of transformation in material during the mechanical activation is its enhanced reactivity. Improved reactivity is primarily the result of increased particle fineness and formation of amorphous regions in structure. Mechanical activation, as a method that can improve FA reactivity, is often applied in different applications of this material. It is known that particles with diameter below 45 μm , obtained through various methods of mechanical treatment, tend to improve mechanical strength of mortar when used as a supplementary cementitious material [7]. According to Jamkar et al. [16], the compressive strength results show that the FA fineness plays a vital role in the activation of geopolymer concrete. An increase in the fineness improved both workability and compressive strength. It was also observed that finer particles increased the rate of reaction, and led to requirement of less heating time to achieve a given strength. Moreover, Gunasekara et al. [13] describe the

highest compressive strength of geopolymer mortar resulting from the highest fineness ratio of FA used.

Washing aggregate sludge is a waste produced during the classification of sand and gravel in gravel pit, or while washing crushed aggregates in quarry (quarry washings). Its use in production of building materials (concrete or mortar) is rare. According to Cresswell [17], crushed rock fines are the material that cannot find a use in the local market, but has the potential to be used for a number of applications other than as fine aggregate. They may have compositions suitable for incorporation into brick manufacture or cement production. Together with González-Corrochano et al. [18], they prove that finer portions may also be suitable for the production of manufactured (light-weight) aggregates.

This paper is aimed on proving the application of mechanically treated (ground) FA for production of FA-based geopolymer. As a modification, fine-grain sludge—a by-product of washing the crushed aggregates—was used as filler for obtaining a mortar-type material. Finally, standard cement mixture was prepared using the same fillers for comparison. The experiment was also intended as environmental support in terms of utilization of specific/regional kinds of waste—low-calcium type of FA, ground granulated blast furnace slag, and washing aggregate sludge.

2 Materials and methods

The experiment is focused on the testing of standard FA-based geopolymer prepared by the acceptance of referenced recommendations for composition, while using regional materials. In addition to the recommended ratio of components of the geopolymer mixture, the influence of the treatment of two basic components was additionally tested: grinding of FA and separation of fine sludge. In this paper, basic physical-mechanical properties of mixtures are presented and discussed with emphasis on time development, while cement-based mixture was tested for comparison.

The materials for tested mixtures were as follows:

- Original fly ash: PVT-FA (coming from Heating plant, class F) and
Ground fly ash: GPVT-FA - as basic component of geopolymer,
- Ground granulated blast furnace slag: GGBFS (Steel making factory, Slovakia) - as intensifier,
- $\text{Na}_2\text{SiO}_3 + \text{NaOH}$ - as activator,
- Sludge - the material from processing (washing) the crushed aggregates, coming from an Eastern Slovakia producer of crushed aggregates - as microfiller
- Ordinary Portland Cement – OPC (CEM I 42.5 R, Slovakia) - as binder

The chemical composition of PVT-FA, GPVT-FA, GGBFS, cement and sludge are given in Tab. 1. The parameter of grain size distribution is also given by distribution values on the x-axis $d(0.1)$, $d(0.5)$ - median and $d(0.9)$, and by the mean value dm .

As for fly ash, the influence of fine-grain form was tested. For this purpose, results of the previous experiment which was focused on the finding the grinding conditions for reducing its particle size were taken into account [19]. Accordingly, sample prepared by following grinding conditions was selected for this experiment: grinding time 60 minutes; grist:grinding

media proportion: 2:1 (GPVT-FA). The mean size d_m was reduced by grinding from 74.1 to 31.02 μm , which is practically the mean size of cement particles. Chemical composition of PVT falls in the values given in [16]; the calcium oxide content of PVT-FA is less than 10% (2.58), the silicon and aluminium oxides constitute about 74% by mass, and Si-to-Al ratio is 2.1. Only the content of the iron oxide is a bit lower (6.4% by mass).

The sludge was withdrawn from the dewatering pond and dried, after which a portion $< 125 \mu\text{m}$ was separated using standard sieve. The aim was to get a micro-filler. The value of $d(0.9)=149.8 \mu\text{m}$ comes from the accuracy limit of such a mechanical process. However, the material can be classified as having $d_m = 83.55 \mu\text{m}$.

Table 1: Chemical composition and grain size distribution parameters of input materials

| Materials | Oxide Composition [%] | | | | | Parameters of grain size distribution [μm] | | | |
|--------------------|-----------------------|-------|-------|--------------------------------|--------------------------------|---|--------|--------|----------------|
| | SiO ₂ | CaO | MgO | Fe ₂ O ₃ | Al ₂ O ₃ | d(0.1) | d(0.5) | d(0.9) | d _m |
| PVT-FA | 51.11 | 2.58 | 1.18 | 6.40 | 23.21 | 3.97 | 20.44 | 84.73 | 74.10 |
| GPVT-FA | 49.72 | 2.49 | 1.09 | 6.37 | 22.32 | 3.48 | 14.53 | 52.81 | 31.02 |
| GGBFS | 41.28 | 35.98 | 12.85 | 0.39 | 6.30 | 3.61 | 19.03 | 111.5 | 49.46 |
| OPC | 19.87 | 64.36 | 4.61 | 3.18 | 3.99 | 3.20 | 17.69 | 49.57 | 29.90 |
| Sludge (separated) | 42.66 | 5.83 | 5.04 | 6.54 | 11.19 | 17.49 | 82.50 | 149.8 | 83.55 |

Table 2 illustrates the principle of tested mixtures. For mixtures T1 – T3 (geopolymer pastes), FA is intended as basic geopolymer filler, while the unground and ground forms are used in three combinations. Mixtures T4 and T5 (mortars) contain ground FA only (GPVT-FA), while 30% is replaced by sludge microfiller. T4 is based on geopolymer reaction similar to the previous mixtures, while T5 contains cement binder only and is considered as a comparative sample.

Table 2: The composition of experimental geopolymers mixtures

| Composition of fillers [%] | Mixtures | | | | | |
|----------------------------|-----------------------|-----|----|-----|----|----|
| | T1 | T2 | T3 | T4 | T5 | |
| GPVT-FA | 46% of mixture volume | 100 | 50 | - | 70 | 70 |
| PVT-FA | | - | 50 | 100 | - | - |
| Sludge < 125 μm | | - | - | - | 30 | 30 |

| Presence of other components | | | | | |
|----------------------------------|-------------------|---|---|---|---|
| GGBFS | 20% of fly ash | • | • | • | - |
| Na ₂ SiO ₃ | 16% of fly ash | • | • | • | - |
| 8M NaOH | 16.5 % of fly ash | • | • | • | - |
| OPC | | - | - | - | • |
| Water | | • | • | • | • |
| Plasticizer | | • | • | • | • |

Key parameters of geopolymer mixtures (ratio between the components) were selected as usual and their typical values were collected from a few publications [8, 9, 10, 20, 21], giving some standard starting level of experiment. The basic geopolymer mixture was designed according to these recommendations (see Table 3). Alkaline-activating agent for mixtures T1 – T4 was prepared by mixing the Na_2SiO_3 and NaOH. The SiO_2 to Na_2O ratio (Ms Modulus) in the alkaline activating agent was adjusted by addition of 8M NaOH to Na_2SiO_3 with a Ms = 2.5.

Table 3: Parameters of geopolymer mixtures

| Parameters | | Values of parameters | | | | | |
|------------|---|-------------------------|-----------------------|------|------|------|------|
| | | According to references | Investigated mixtures | | | | |
| | | | T1 | T2 | T3 | T4 | T5 |
| 1 | Water from the alkaline activators /geopolymer solids | 0.19 | 0.19 | 0.19 | 0.19 | 0.25 | / |
| 2 | Alkaline solution/FA | 0.25–0.45 | 0.38 | 0.38 | 0.38 | 0.56 | / |
| 3 | $\text{Na}_2\text{SiO}_3/\text{NaOH}$ | 1–1.25 | 1.18 | 1.18 | 1.18 | 1.18 | / |
| 4 | Water/solids | 0.23–0.51 | 0.24 | 0.26 | 0.30 | 0.33 | 0.37 |

The samples (beams 40x40x160 mm) were cured at the laboratory temperatures covered by foil sheet at first, then under ambient humidity (50%). Following standard tests (density, total water absorption, flexural strength and compressive strength) were executed after 2, 7, 28 and 120 days according to [22-24].

For providing information about molecular structure and the presence of specific functional groups in geopolymer samples based on various fly ash additions (T1-T3), infrared spectroscopy in transmission mode was used. FTIR measurements were carried out using Bruker Alpha Platinum-ATR spectrometer (BRUKER OPTICS, Ettingen, Germany). A total of 24 scans were performed on each sample in the range of 400–4000 cm^{-1} .

3 Results and discussion

The results of tests on density, total water absorption, flexural strength, and compressive strength are reported in the following figures. For discussion of the mentioned properties, results are summarized in the graphs in two groups:

- T1, T2, and T3, where the influence of ground FA can be discussed
- T1, T4, and T5, where the effect of replacing a part of ground FA with the sludge, as well as effect of using cement binder instead the geopolymer reaction can be discussed

Values of individual properties in 120 days of setting and hardening range as follows: 1730–2050 kg.m^{-3} (density), 12.6–18.9 % (water absorption), 2.6–3.6 MPa (flexural strength), and 9.1–17.2 MPa (compressive strength).

The results presented in Figures 1 to 4 indicate that all four presented parameters have a good relationship with each other, as it is a standard consequence of microstructure of matters: With decreasing density of sample, the strength decreases and total water absorptivity increases. The development of the properties in time is also clear: With the time, changes in the microstructure cause a reduction in the water absorption and increase in both the strength and density.

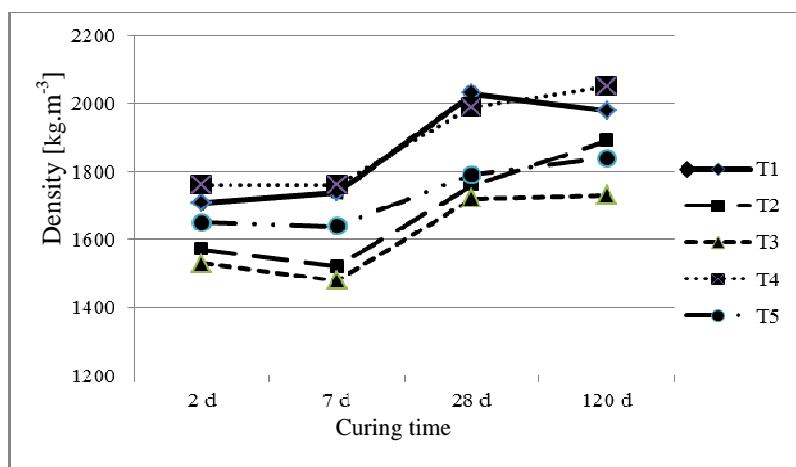


Figure 1: Density of samples T1-T5

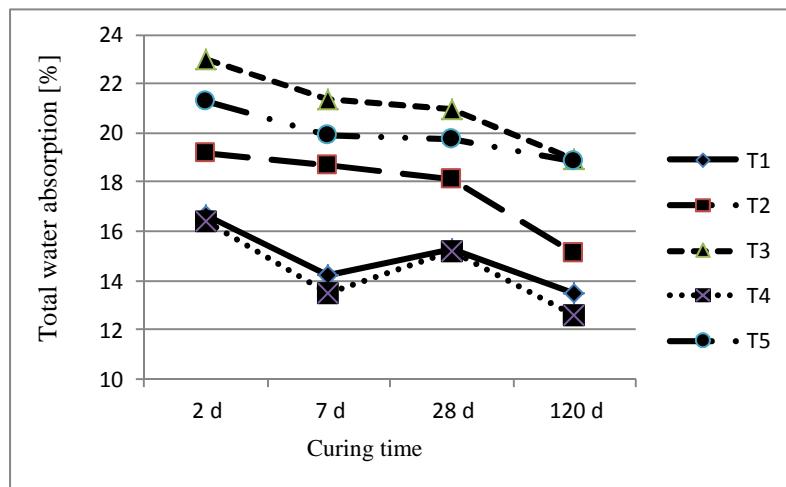


Figure 2: Total water absorption of samples T1-T5

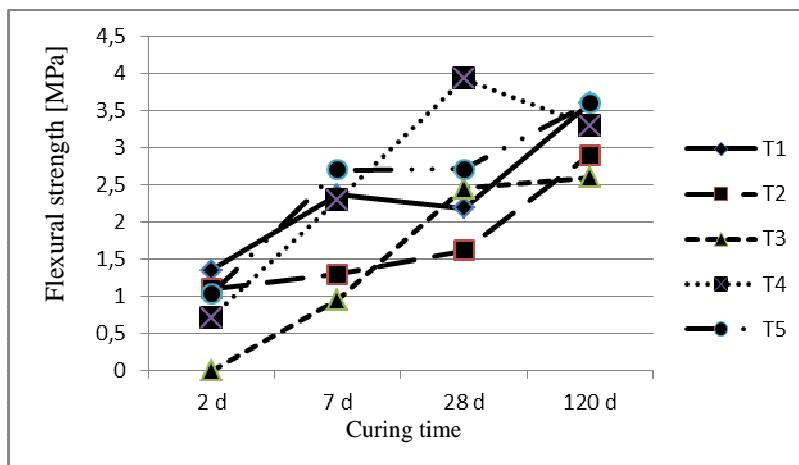


Figure 3: Flexural strength of samples T1-T5

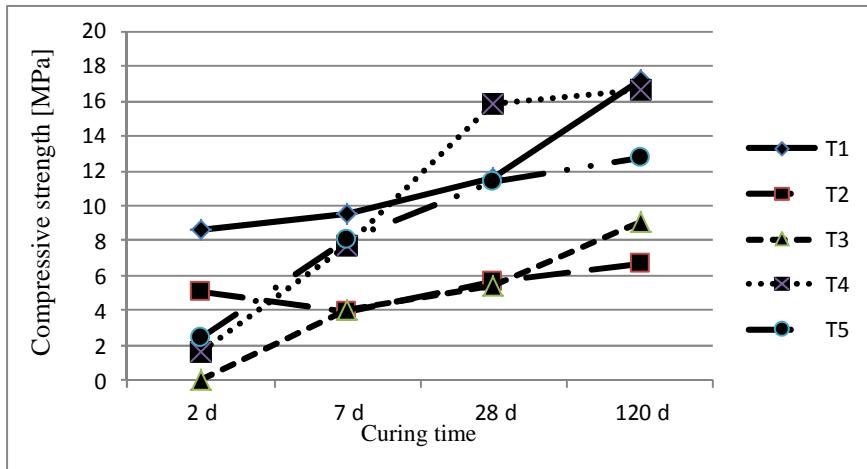


Figure 4: Compressive strength of samples T1-T5

Group T1, T2, and T3: The influence of FA granularity is confirmed in Figures 1 – 4 comparing samples T1-T3. The best results (highest strength and lowest water absorption) are detected for sample T1 (100% ground FA), while the worst results can be attributed in principle to T3 (100% unground FA). It is in agreement with the [25], where geopolymers produced with ground fly ash showed the increase in strength for finer fractions obtained by grinding. In fact, in terms of the density and strength, the results of samples with 100% unground FA (T3) and 50% unground FA (T2) are very close. What is technically important is that the difference between T1 and other samples is significant with respect to compressive strength, including short-term two-day values. Values of presented properties of T1 in 28 days of setting and hardening are as follows: 2030 kg.m⁻³ (density), 15.3% (total water absorption), 2.2 MPa (flexural strength), and 11.6 MPa (compressive strength).

Group T1, T4, and T5: The effect of replacing a part of ground FA with the sludge seems to be insignificant (see samples T1, T4 and T5 in Figures 1-5). There are a few differences between T1 and T4 in terms of flexural and compressive strength at certain points of time, but ultimately the results are settled. Results of density and total water absorption are very close. Differences between the geopolymer mixtures (T1 and T4) and cement based mixture (T5) are evident in the case of density and total water absorption: Positive changes were recorded for geopolymers, while results are in good relationship (lower is density, higher is water absorption). Tendencies in strengths are not quite clear. Results in flexural strength are close to T1 while results in compressive strength are close to T4, but only in the early ages. Taking into account total water absorption and later strengths, samples based on geopolymer reaction seem to be more favourable than samples based on cement.

The infrared spectroscopy analysis records are given in Figure 5 for original fly ash (PVT-FA) and Figure 6 for geopolymer mixture T1 (100% of ground fly ash: GPVT-FA).

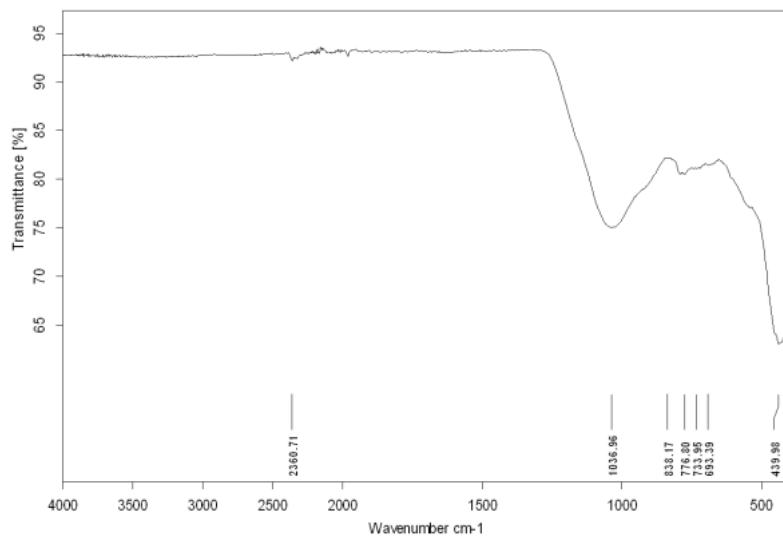


Figure 5: The infrared spectroscopy analysis of original fly ash PVT-FA

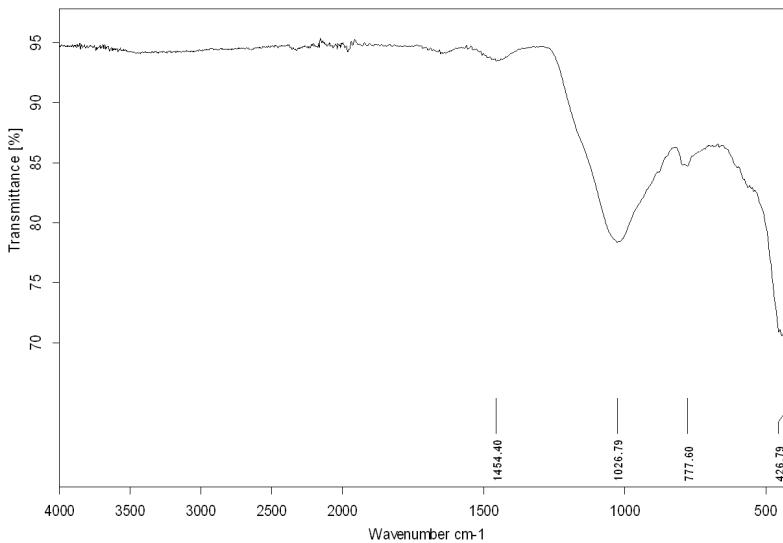


Figure 6: The infrared spectroscopy analysis of geopolymer mixture T1

As shown in Figures 5 and 6, the absorption peak positions observed by the FTIR spectra of the fly ash and the geopolymer sample T1 are almost identical. The major bands in the FTIR spectra of these substances and their assignments to vibrations of functional groups are summarized in Table 4. Measurements of another geopolymer concrete samples T2 (combination of ground and unground fly ash) and T3 (unground fly ash) showed the same IR bands in investigated region of wavenumber.

The broad absorption band at 1037 cm^{-1} , peak of significant intensity at 440 cm^{-1} , very weak peak at 2360 cm^{-1} and four peaks visible in the region of $839 - 690 \text{ cm}^{-1}$ are recorded in the spectrum of fly ash (Fig. 5). In addition, spectrum of sample T1 (Fig. 6) contains two peaks at 1454 and 1652 cm^{-1} , respectively. The band at 1454 cm^{-1} indicates stretching vibration of the O-C-O bond in calcite and could also contain a contribution of atmospheric carbonation of high alkaline NaOH. The band at 1652 cm^{-1} comes from trace amount of water (bending

vibration of H-O-H). The strong peak at 1037 cm^{-1} is due to the Si-O-Si and Al-O-Si asymmetric stretching vibration [26]. In the case of geopolymers sample T1, it is shifted to lower frequency value (1026 cm^{-1}). The more significant shift of this band to 976 and 969 cm^{-1} was identified in fly-ash-based geopolymers lightweight concrete using foam agent [27]. Based on investigations in [28, 29, 30], this shift was explained by the formation of amorphous aluminosilicate gel phase due to dissolution of aluminium and silicon species from the fly ash surface in alkaline activator, followed by the polymerization of active surface groups and soluble species to form gel and, subsequently a hardened geopolymers structure. Principal band of Si-O stretching vibration in hydrated phase of C-S-H at 974 cm^{-1} and Si-O asymmetric stretching vibration of silicate, centered at 925 cm^{-1} attributed to $[\text{SiO}_4]^{4-}$ vibration can be also included in the broad peak with maximum intensity at 1037 cm^{-1} [31, 32]. The bands at 877 and 777 cm^{-1} are assigned to O-C-O bond vibration in carbonate. Strong band in the region of 427 - 520 cm^{-1} seen in the spectra corresponds to the Si-O bending modes (452 cm^{-1} - Si-O in-plane bending vibration; 522 cm^{-1} - Si-O out-of-plane bending vibration) in SiO_4 tetrahedra of silicate.

The peak of very weak intensity at 2360 cm^{-1} represents the stretching and deformation vibration of O-H and H-O-H groups from weakly-bound water molecules adsorbed on the surface or situated between the rings of the geopolymers products [33].

Table 4: Identification of FTIR bands in PVT-FA and geopolymers sample T1

| Wavenumber [cm^{-1}] | Vibration of function group |
|---------------------------------|-----------------------------|
| 3400-3100 | O-H and H-O-H |
| 2360 | O-H |
| 1652 | H-O-H |
| 1454 | O-C-O |
| 1037, 1026 | Si-O-Si and Al-O-Si |
| 877 | C-O |
| 777 | C-O |
| 427-520 | Si-O |

4 Conclusion

This paper was focused on the utilization of regional alternative (waste) materials (FA and sludge) as components of standard geopolymers mixture. Their treatment (grinding in the case of FA and sorting in the case of sludge) represents a valuable input, leading to better influence on the technical parameters of final mixtures. The following conclusions can be formulated from the presented experiments:

- for proposed composition of experimental geopolymers mixtures based on ground fly ash, a formation of amorphous aluminosilicate gel phase, followed by the polymerization of active surface groups to form gel and, subsequently a hardened geopolymers structure was confirmed using FTIR analysis;

- for FA based geopolymer, the results confirm the ground form of FA is being favourable for basic technical properties such as density, water absorption, flexural strength, and compressive strength;
- mixture using ground FA with $d_m = 31.0 \mu\text{m}$ instead of unground one with $d_m = 74.0 \mu\text{m}$ is significantly better in all parameters. Comparing 28-day values, the density is higher by about 18%, total water absorption is better by about 25%, flexural strength is better by about 25%, and compressive strength is better by about 10%;
- incorporation of fine portion of sludge into the geopolymer mixture does not lead to a significant technical profit. However, the sludge can be utilized on the basis of the comparable properties. Here, an environmental point of view can be taken into account (amounts of FA and the sludge to be utilized);
- binders based on geopolymer reaction seem to be more favourable than samples based on cement, in terms of total water absorption and long-term strength.

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