SYNERGETIC USE OF LIGNITE FLY ASH
AND METALLURGICAL CONVERTER SLAG
IN GEOPOLYMER CONCRETE

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Abstract: The application and utilization of the industrial wastes and by-products in the construction industry is a key issue from an environmental and economic point of view. The increased use of lignite has substantially increased the available quantities of lignite fired power plant fly ash, which can be mainly classified as class C fly ash. The utilization of such raw material however has some difficulties. In the present paper lignite fired power station fly ash and metallurgical converter slag were used for the production of geopolymer concrete. The fly ash was used as a geopolymer based binder material, and a converter slag as aggregate, thus created a geopolymer concrete which contains mainly industrial wastes. As preliminary test experimental series were carried out using andesite as aggregate. The optimal aggregate/binder ratio was determined. The effect of the amount of alkaline activator solution in the binder, the aggregate type on the geopolymer concretes' compressive strength and density was investigated. Furthermore, the physical properties - freeze-thaw resistance and particle size distribution - of the applied aggregates were measured as well. As a result of the experiments it was found that physical properties of the andesite and converter slag aggregate was close. Therefore andesite can be replaced by converter slag in the concrete mixture. Additionally, geopolymer concrete with nearly 20 MPa compressive strength was produced from class C fly ash and converter slag.

Keywords: lignite fly ash, converter slag, geopolymer concrete

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INTRODUCTION

In the last decade fly ash based geopolymer has emerged as a promising new cement alternative in the field of building and construction materials (Palomo et al, 1999; Rangan, 2007, Hardjito and Rangan, 2005). Geopolymers exhibit many excellent properties such as high compressive strength, low creep, good acid resistance and low shrinkage (Hardjito and Rangan, 2005). Davidovits (1991) showed that geopolymers can be produced by polymerization of alumino-silicate oxides with alkali polysilicates yielding Si–O–Al bonds. Based on Duxson et al (2007) the two main ingredients of geopolymer binder are alkali liquids and source materials. The alkali liquids are usually sodium or potassium based solutions. The source materials should be rich in silicon (Si) and aluminium (Al) from geological origin or by-product materials such as clays, metakaolin, fly ash, bottom ash, slag, and rice husk ash. The use of geopolymer involves a lesser amount of green house gas and is, therefore, a more environmental-friendly binding material compared to the conventional Portland cement. Hardjito and Rangan (2005) demonstrated that one tonne of low-calcium fly ash can be used to produce 2.5 m^3 of high quality geopolymer concrete that is cheaper than Portland cement concrete. Fly ash geopolymers have been prepared as geopolymer pastes (Temuujin et al, 2009), mortars (Lee and van Deventer, 2004) and concretes (Rangan, 2007; Hardjito and Rangan, 2005; Temuujin et al, 2010). For the production of mortar and concrete, natural or industrially manufactured aggregate is added as filler as it is less expensive than the paste. The inclusion of naturally occurring materials such as quartz, basalt, granite, sandstone and limestone as aggregates to geopolymer paste is not only economically favourable but also reduces pore density, reduces crack formation and improves durability (Subaer, 2004). Determination of the optimal binder aggregate ratio is important for resource efficiency and meeting specified mechanical properties of mortars (Temuujin et al, 2010). Based on Guo et al (2010) thus far, the work on fly ash geopolymer has been based on a precursor derived from class F fly ash produced by burning bituminous coals (Palomo and Grutzeck, 1999; Swanepoel and Strydom, 2002; Fernandez-Jimenez and Palomo, 2005; Kovalchuk et al, 2007). Recently, the increased use of lignite and subbituminous coals has substantially increased the available quantities of class C fly ash. However, there have been very few published papers on class C fly ash geopolymer (Guo et al, 2010). From a compositional viewpoint, the main difference between C and F type fly ash is the calcium content, while both typically contain substantial amounts of silica and alumina (Duxson and Provis, 2008). The class C fly ash based geopolymers were studied by Phoongernkham et al, 2014; Tho-in et al, 2012; Oh et al, 2012; Hanjitsuwan et al 2014, Phair and Van Deventer, 2001; Phair and Van Deventer, 2002).

The possibility of the synergetic use of the lignite-fired power plant fly ash and metallurgical converter slag is presented in this paper. The fly ash is used as a geopolymer based binder material, and a converter slag as aggregate used in a geopoly-

mer concrete. The subject of the paper - besides the utilization possibility – is the investigation of the effect of aggregate/binder ratio, amount of the activator solution and fly ash fineness in the binder, aggregate type on the geopolymer concretes' compressive strength and density. Furthermore, the physical properties - freeze-thaw resistance and particle size distribution - of the applied aggregates were measured as well.

MATERIALS AND METHODS

Lignite fired fly ash from Mátra power station was used for the production of the geopolymer binder material. Chemical composition of the fly ash can be found in Table 1. The chemical composition was measured by Rigaku Supermini 200 type x-ray fluorescence spectrometer. The mineral composition of the fly ash was investigated by Bruker D8 Advance type powder diffractometer. The main mineral phases in the fly ash were quartz, anhydrite and albite (Table 1).

Table 1. Mineral and chemical composition of the Mátra power station fly ash

Mineral composition, m/m%		Chemical composition, m/m%	
quartz	20.34	SiO_2	48.10
maghemite	4.22	Al_2O_3	14.42
hematite	3.91	MgO	3.34
anhidrite	7.08	CaO	11.76
albite	4.71	Na_2O	0.37
albite K0.16	5.58	K_2O	1.66
CaO	1.61	Fe_2O_3	10.97
amorph	52.5	MnO	0.171
		TiO_2	0.492
		P_2O_5	0.264
		Σ	91.547

Andesite (Colas Északkő Ltd, Tállya Mine) and metallurgical (steelmaking) converter slag (ISD Dunaferr Ltd., Dunaújváros) were used as an aggregate in the geopolymer concrete. Andesite was used in the size fractions (nominal) of -4 mm; –8 mm + 4 mm; –16 mm +8 mm and –22.4 mm +16 mm, while converter slag was used in the size fractions of –12 mm + 5 mm and –20 mm +12 mm.

Water glass (Wöellner Betol SB) and sodium hydroxide (8 M/l NaOH solution) was used as activator solution for the geopolymer production.

The uniaxial compression strength of the binder and concrete specimens were measured by Controls SERCOMP 7 (Modell 50 C7022) equipment.

The fine grinding of the fly ash was carried out in a ball mill. In case of the binder development the batch ball mill experiments was realized by 303x305 mm internal diameter smooth walled, cylindrical ball mill with steel ball grinding media. Grinding media is uniformly distributed with 40 mm of maximum diameter. The filling ratio of the grinding media was 30 V/V%, the material filling ratio (compared to the pore volume between the grinding media) was 110 V/V%. The operational revolution number to the critical revolution number was 80 %. Time duration of the fine grinding was 10, 20, 30, 60, 120 minutes. In case of the geopolymer concrete, because of the required higher amount of the grounded fly ash, a continuous working ball mill (L/D=800/400; grinding media diameter: d_{max}=40 mm, d_{min}=12 mm; grinding media filling ratio 0.3) was used. The mill is equipped with smooth steel liners and filled with steel balls as well. The operational revolution number to the critical revolution number was 80 %.

The ground materials' particle size distribution was investigated by a Horiba LA-950 V2 type laser particle size analyser. Specific surface areas had been calculated from the distributions, shape factor was 1. During the measurement processes to achieve the appropriate dispersity state 1 minute ultrasonic treatment and 0.5 ml sodium pyrophosphate dispersant was applied.

Stretching and bending vibrations of the chemical bonds in the samples induced by infra range electromagnetic waves was detected by JASCO FT-IR 4200 type Fourier Transformed Infrared Spectrometer in reflection mode, diamond ATR was used. During the measurements 5 spectra were measured from each sample.

EXPERIMENTAL

In case of the geopolymer binder development the following procedure was used. As a first step the fly ash was mechanically activated in a batch ball mill. The ground fly ash then was mixed with the activator solution (Betol SB water glass and 8 M/l NaOH solution) in a L/S = 0.82 ratio. The mixture then was placed in a 35×80 mm cylindrical mould and 2 min vibration was applied in vibro-table for the compacting. The specimens were heat treated on 30, 60 and 90 °C for 6 hours. The specimens were removed from the moulds after 24 h curing time. Five specimens were made in each condition for the strength investigation.

Two experimental series were carried out for the optimization of the geopolymer binders' composition. In the first case the effect of the fly ash fineness was investigated. In the second specimens with various water glass ratios in the activator solution (0, 25, 50, 75, 100%) and various heat treatment temperature (30, 60, 90 °C) were applied. The L/S ratio was 0.82 in both measurements (55 m/m% fly ash and 45 m/m% activator solution).

As a first step of the geopolymer concrete production the fly ash was mechanically activated in a continuous working ball mill. The ground fly ash then was mixed with the activator solution (75% Betol SB water glass and 25 % 8 M NaOH solution) and the aggregate. The mixture then was placed in a 100x200 mm cylindrical mould and 2 min vibration was applied on vibro-table for the compacting. The specimens were heat treated at 30 °C for 6 hours. The specimens were removed from the moulds after 24 h curing time. Three specimens were made in all cases for the strength investigation. The uniaxial compressive strength was measured at the 7 days age.

In case of the geopolymer concrete production the effect of the aggregate/binder ratio was investigated at two different activator solution/fly ash ratio (L/S=1 and 0.82). During the experiments the effect of the aggregate/binder ratio on the specimens' compressive strength and density was investigated. The uniaxial compressive strength of the geopolymer concrete was measured at the 7 days age. The experiments were carried out with 85/15; 80/20; 75/25; 70/30; 65/35 aggregate/binder ratios. The aggregate was -22.4 mm andesite, the particle size distribution was set properly, the –22.4 mm fraction was the mixture of the –4 mm; +4 mm –8 mm; –16 mm +8 mm; –22.4 mm +16 mm fractions. The mass ratios of the fractions are as follows: –4 36%; +4 mm –8 mm 17%; –16 mm +8 mm 12%; –22.4 mm +16 mm 35%. After the optimal aggregate/binder ratio was found the aggregate in the –22.4 mm +4 mm size fraction was replaced by metallurgical converter slag. The physical properties of the aggregate materials, like particle size distribution and freeze-thaw resistance was measured according to the European standards (EN 933-1, EN 1367-1, EN 1367-2) to measure their application as concrete aggregate.

RESULTS

The results are presented in four different sections: (1) result of the fine grinding of the fly ash (2) the experimental results of the binder development, (3) physical properties of the aggregates and (4) the experimental results of the geopolymer concrete development.

FINE GRINDING OF THE FLY ASH SAMPLE

As a first step for the geopolimer concrete production the fly ash was mechanically activated by dry milling. The effect of the milling time on the particle size distribution of the ground material and the particle size distribution of the raw fly ash can be seen in Fig. 1.

The 80 percent particle size of the raw fly ash was 134 µm. During the milling the particle size was significantly decreased, after 60 min milling more than 40 percent of the material was finer than 10 µm, and the 80 percent particle size in this case was 18 µm.
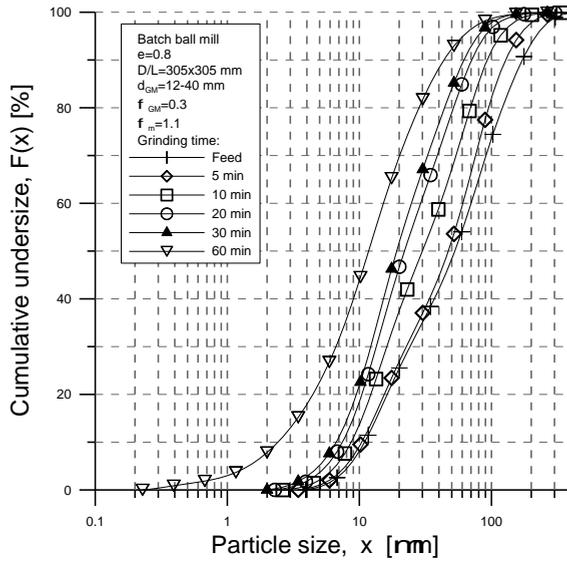
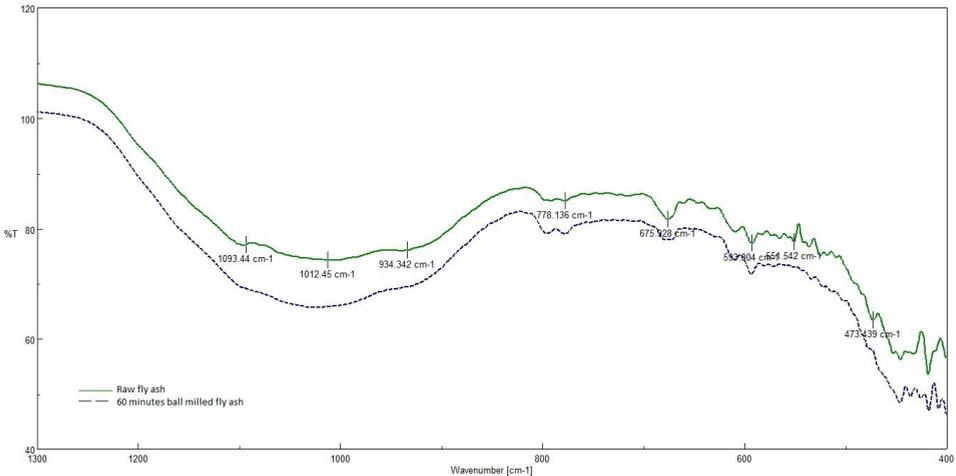Fig. 1. The particle size distribution of the ground fly ash

Fig. 2. FTIR spectra of the raw and grounded fly ash

Based on the raw and ball milled fly ash FT-IR spectra (Figure 2) significant absorbance can be observed at 1093, 1012, 934, 778, 676, 593, and 551 cm^{-1} related to T-O-Si (T=Si, or Al) bonds asymmetric and symmetric stretching vibration. Peak belongs to 473 cm^{-1}, related to bending vibration of Si-O-Si and O-Si-O. After 60 minutes grinding the peak intensities were higher, which can be associated to the higher infra absorbance due to the higher specific surface area of the fine particles

(Molnár et al, 2013). The specific surface area of the raw fly ash was 1152.07 cm^2/g, and after 60 min grinding it was 5425.55 cm^2/g. The peak at 1097 cm^{-1} can hardly observed in the case of ground fly ash spectra, at 473 cm^{-1} peak was disappeared respectively, these changes can suggest that mechanochemical, crystal structure changes was carried out in the material, as a result of the milling.

GEOPOLYMER BINDER MATERIAL

Numerous preliminary experiments were carried out for the development of the lignite fired fly ash based geopolymer binder, although in the present paper the effect of fly ash fineness, the effect of the Betol SB water glass and the effect of the heat treatment temperature are presented, as the highly important parameters of the geopolymer production.
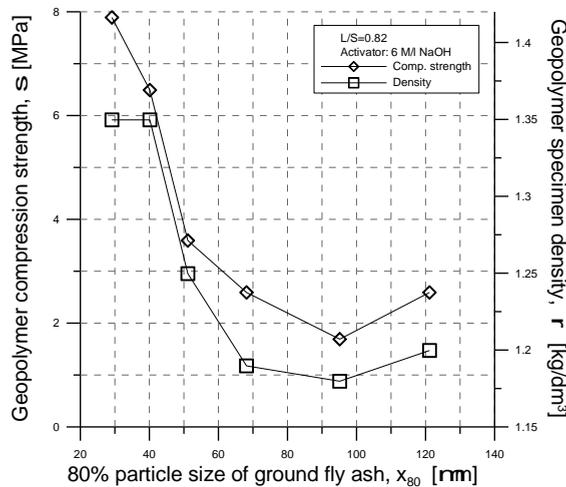

Fig. 3. Effect of the fly ash fineness on the geopolymer strength

Figure 3 shows the effect of the fly ash fineness on the geopolymer strength. Higher milling time and thus finer fly ash resulted in higher geopolymer strength. As a result of the milling, the specific surface of the material was increased as well, resulting more Al and Si was solved by the NaOH solution from the fly ash, and this was advantageous for the emergence of the geopolymer gel. Another explanation of the increasing compressive strength can be that the finer particles resulted in more compact microstructure.

Fig. 4 shows the effect of the water glass dosage and the heat treatment's temperature on the specimen's compressive strength. As it can be seen higher K-Na-silicate content (water glass) resulted in higher compressive strength of the specimen at all
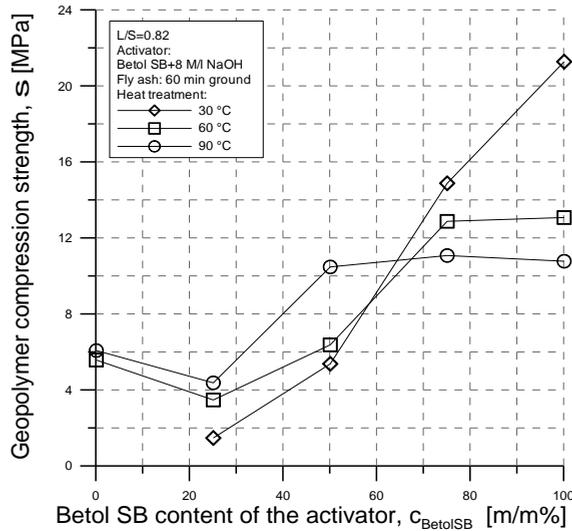
Fig. 4. Effect of the water glass dosage and heat treatment's temperature
on the specimen's compressive strength

temperature of the heat treatment. The decrease of the temperature of the heat treatment resulted in higher compressive strength at 75 and 100 percent water glass dosage. At lower water glass dosages the tendency is reversed, higher temperatures resulted in higher compressive strength. The highest compressive strength was obtained at 30 °C heat treatment with 100 m/m% Betol SB water glass in the activator solution. During these experiments the aim was to decrease the heat treatments' temperature and to make geopolymer at ambient temperature. As it can be seen, the applied temperature of the heat treatment can be reduced by the increasing Betol SB water glass content in the activator solution. At 75 and 100m/m% water glass content nearly ambient temperature geopolymer production can be reached.

The applied L/S ratios compared to the literature is much higher, in our case it was 0.82 or 1, but Hanjitsuwan et al (2014) used L/S = 0.4, Songpiriyakij et al (2010) L/S = 0.54, Somna et al (2011) L/S = 0.3. However in this case the high activator liquid content was necessary for the geopolymer concrete production, based on the earlier experiments of the binder development.

PHYSICAL PROPERTIES OF THE AGGREGATES

The rock-physical properties of the andesite and converter slag samples were measured. The aim of the measurements was to decide their applicability as aggregate material in the concrete. The particle size distribution of the aggregates was determined by wet screening according to the EN 933-1. The andesite is a standard certified product according to EN 13043. Their particle size distribution corresponds to

the standard one. The -12 mm +5 mm fraction grade is Gc85/20. The -20 mm +12 mm fraction shows the best agreement with the -22 mm +11 mm standard product; however its particle size distribution curve slightly sticking out from the specified range, but the difference is less than 1% at 22.4 mm. Based on these measurements it can be conclude that standard size fractions can be produced from the samples by further classification.

The freeze-thaw resistance measurements of the samples were carried out according to the EN 1367-1 standard by 10 cycle freeze-thaw measurement. The andesite sample was chosen as reference material. Water absorption and $MgSO_4$ crystallization measurement methods were carried out comparatively to the freeze-thaw measurement. The measurement results can be seen in Table 2.

Table 2. Freeze-thaw resistance measurements of the andesite and converter slag samples

Name of the measurement and certificate according to EN 12620	Andesite sample	Metallurgical converter slag sample
Water absorption, [%]	0.83	1.61
Magnesium sulphate soundness, MS	5.05 (MS_{18})	6.26 (MS_{18})
Freeze-thaw resistance, F	0.22 (F_1)	0.13 (F_1)

Water absorption capacity of the samples is practical to measure because it is provides results relatively fast. Less than 1% value means that the sample can be presumed as frost resistant. Higher than 1% water absorption capacity, however does not automatically mean that the sample is not frost resistant. In all cases the water absorption capacity of the samples was not significant, the andesite had less than 1%, but the metallurgical converter slag had water absorption capacity a bit higher than 1%. The higher water absorption of the slag can be explained by the porous structure. Both $MgSO_4$ soundness and 10 cycle freeze-thaw resistance measurements were resulted proper values, so both andesite and converter slag could be considered weather and frost resistant.

GEOPOLYMER CONCRETE

Fig 5 shows the effect of the aggregate/binder ratio on the compressive strength of the geopolymer concrete using andesite aggregate in case of L/S=1 in the binder. Based on the measurement result it can be concluded that the highest compressive strength (19.2 MPa) was measured at 80/20 aggregate/binder ratio. The compressive strength and density of the geopolymer concrete increased as function of binder content until a certain value which was 20 m/m% so it has an optimum in the applied measurement range. After the maximum point the compressive strength and density slightly decreases with higher binder content. In contrary the workability of the paste is becoming better by the higher binder content.
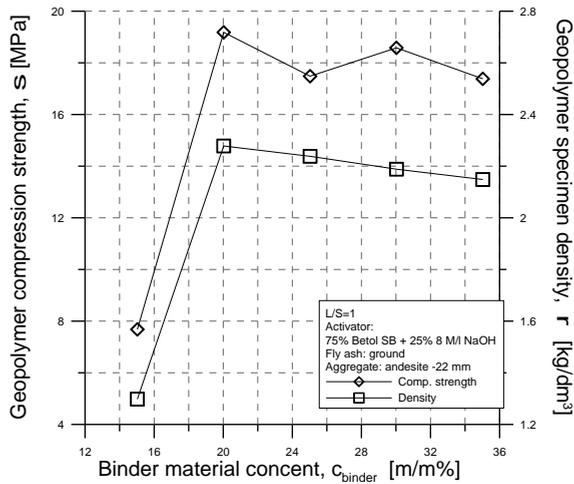
Fig. 5. Effect of the binder content on the compressive strength and specimen density
of the geopolymer concrete in case of L/S = 1 in the binder material

As it can be seen in Fig 6, in case of low binder content (15%) the amount of the binder material was not enough to fully fill the pours between the aggregates, so in this case so called water permeable (pervious) geopolymer concrete was produced. Higher binder material content resulted in denser specimen structure; the pores between the aggregates are fully filled, so denser geopolymer concrete was produced.
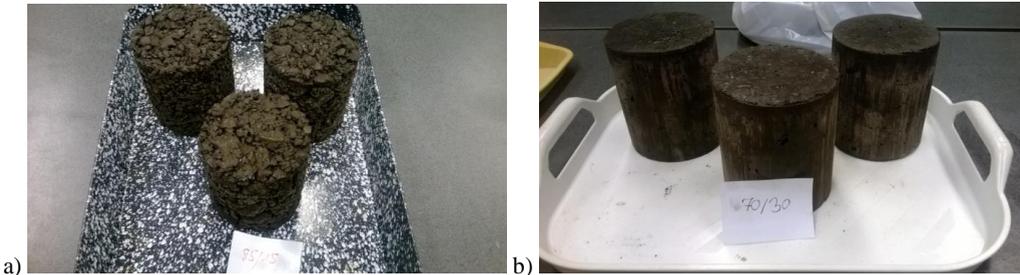

Fig. 6. Pictures of the geopolymer concrete specimens produced by 85/15 (a)
and 70/30 (b) aggregate/binder material ratios (L/S = 1 in the binder material)

Fig 7 shows the effect of the binder content on the compressive strength and specimen density of the geopolymer concrete in case of L/S=0.82 in the binder material.
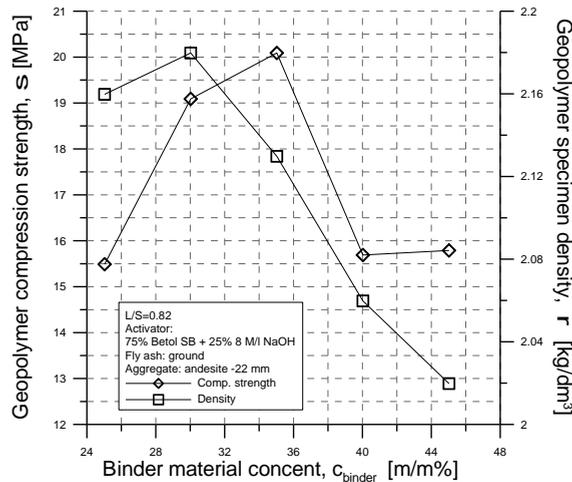
Fig. 7. Effect of the binder content on the compressive strength and specimen density
of the geopolymer concrete in case of L/S = 0.82 in the binder material

Based on the measurement results it can be seen that the highest compressive strength value was obtained at an aggregate/binder ratio of 65/35, its value is 20.1 MPa. Increasing binder material content resulted in higher compressive strength value until a limit – in this case it is 35 m/m% – and after this limit maximum value the compressive strength significantly decreased. The binder material content has an optimal value in the measurement range from a compressive strength viewpoint. Contrary, the geopolymer concrete specimens' density continuously decreases slightly by the higher content of binder material. Investigated the effect of the L/S ratio in the binder material, it can be established that decreasing liquid content in the binder material resulted in higher geopolymer concrete compressive strength, so the lower L/S ratio is favourable in the binder material. In case of lower liquid content in the binder material and thus lower L/S ratio higher binder material content is necessary to obtain the highest compressive strength. It was observed during the preparation of specimens that the workability of the geopolymer concrete was improved and the setting time was increased by the higher liquid and binder material content.

Based on the earlier described ideal aggregate/binder material ratio (70/30) and activator liquid concentration (L/S = 1) the –8 mm +4 mm; –11 mm +8 mm and –22 mm +11 mm andesite fractions were replaced by metallurgical converter slag fractions, to investigate the possibility of the synergetic use of the lignite-fired power plant fly ash and metallurgical converter slag in geopolymer concrete. Based on the previously described measurements the –8 mm +4 mm, –11 mm +8 mm and –22 mm +11 mm andesite fractions can be replaced by the appropriate converter slag fraction. The average value of the specimens' compressive strength – produced by the previously described way – can be seen in Table 3.

Table 3 Compressive strength and specimen density of the converter
slag aggregate based geopolymer concrete

Geopolymer concrete	Converter slag aggregate	Andesite aggregate
Compressive strength (MPa)	19.1	18.6
Specimen density (g/cm^3)	2.37	2.19

Based on the measurement result in Table 3, it can be established that the replace of the andesite by metallurgical converter slag resulted in a bit higher compressive strength, than in the case of andesite aggregate based geopolymer concrete. The metallurgical converter slag can be used as a geopolymer concrete aggregate, thus creating the possibility of the synergetic use of these industrial by-product as building materials. The developed fly ash and metallurgical converter slag based geopolymer concrete can be used in the building industry, for example for paving rocks production.

CONCLUSIONS

Based on the experimental results it can be conclude that the fine grinding of the lignite fly ash and the application of the water glass in the activator solution were advantageous to increase the strength of the geopolymer binder. The metallurgical converter slag has good weather and frost resistance, thus it can be well used as geopolymer concrete aggregate. The lignite fired power station fly ash and the metallurgical converter slag was successfully applied collectively in the geopolymer concrete, thus creating a binder material for building industrial purposes with a nearly 20 MPa compressive strength.

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