UNIVERSITY OF MISKOLC
FACULTY OF EARTH SCIENCE AND ENGINEERING
MIKOVINY SÁMUEL DOCTORAL SCHOOL OF EARTH SCIENCES

Development of geopolymers with specific properties, with special regard to geopolymer foam products

Thesis booklet of Doctoral (PhD) dissertation

Author:

Roland Szabó

process engineer

Scientific supervisor:

Gábor Mucsi, PhD

associate professor

Co-supervisor:

Sándor Márton Nagy, PhD

associate professor

MIKOVINY SÁMUEL DOCTORAL SCHOOL OF EARTH SCIENCES

Head of the doctoral school:

Péter Szűcs, PhD

professor

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1. The scientific background and aims of the dissertation

Continuous decrease in the amount of primary minerals, and rise in their price all over the world are encouraging professionals to find solutions that realize in addition the environmentally friendly mining activities the more efficient use of raw materials. Furthermore, the recycling of the generated by-products, as secondary raw materials, should take place as widespread as possible.

Mining, metallurgy and construction also are producing large amounts of waste and by-products, which can be classified as aluminosilicates in terms of their mineral composition. However, large-scale recycling of these materials, like mining wastes, metallurgical slags, fly ash and slags, is far from resolved, and often even difficult to store (*Komnitsas és Zaharaki 2007, Kumar et al. 2007, Mucsi 2016*). According to the literature, the annual amount of coal combustion products generated reaches 1100 million tons worldwide (*Feuerborn et al. 2019*).

Most of the above-mentioned industrial by-products are currently deposited, which is a disadvantageous solution. On the one hand, waste stockpiles reduce valuable agricultural and industrial areas and in the case of improperly deposition also pose environmental risks (water, soil and air pollution) (*Komnitsas és Zaharaki 2007*). In contrast, the utilization of these by-products as raw materials also allows sustainable management of the primary raw material.

In recent years, there has been considerable research worldwide to develop new, environmentally friendly building materials that can reduce the emissions of greenhouse gases (primarily carbon dioxide from manufacture of Portland cement) in the Earth's atmosphere. Production of every tone of cement produces about 0.815 tons of CO_2 (*Gartner 2004*).

One way the use for aluminosilicate-containing by-products may be their usage as AAC. Due to the lower temperature used in the production of geopolymers and the lack of calcination, only 10-20% of the CO_2 produced in the production of conventional Portland cement is generated (*Davidovits 2002*).

The term "geopolymer" was first used by French scientist, Joseph Davidovits in 1972. Geopolymer refers to a class of mineral substances that have a chemical composition similar to zeolite but have a mixed microstructure (from amorphous to semi-crystalline structure) (*Davidovits 1991*). He called geopolymer the three-dimensional aluminosilicates formed from natural aluminosilicates at low temperatures and pressure (*Davidovits 1988*).

Due to their energy-efficient, environmentally friendly production and their special properties (e.g. excellent mechanical properties, heat and fire resistance, low curing temperature and time, moldability etc.), geopolymers are an alternative raw material not only in many fields of construction but also for high-tech applications.

However, different applications require different material properties: e.g. high densities and low coefficient of permeability are required when embedding radioactive waste, whereas high porosity, lightweight structures are required for thermal insulating materials.

These properties can be controlled by process operations like grinding, compaction, mixing, and heat treatment.

In connection with the above summarized results, the aim of my research is to develop fly ash-based geopolymers and technologies whose physical properties (compactness, porosity and strength) can be consciously tailored by process operations.

Based on the literature and the scientific background, it can be stated:

- Various primary raw materials (e.g. kaolin), industrial by-products (e.g. fly ash, slag) and calcined clay minerals (e.g. metakaolin) are suitable for geopolymer and geopolymer foam production.
- Methods used in concrete technology can be used for compacting of geopolymer binder and geopolymer concrete, such as compaction, ramming, vibration compacting. However, increasing the compactness of the geopolymer can also be achieved by high-pressure compaction.
- The reactivity of the starting materials used in the preparation of geopolymers can be enhanced by mechanical activation, thereby increasing the strength of the resulting geopolymer products.
- For the production of geopolymer foam many foaming processes are known, such as chemical foaming (use of foaming agent, primarily metal powder or hydrogen peroxide), physical foaming (introduction of air into the geopolymer paste by mixing and use of surfactants to stabilize the foam).
- The formation of the porous structure of the geopolymer foams is influenced by the foaming process used, the amount of foaming agent used and the rheology of geopolymer paste.

Based on the results presented above, I have identified the following research gap:

- Literature research on compaction of geopolymer binder and geopolymer concrete does not investigate in details the effect of the compaction method used on the product properties.
- Furthermore, the literature does not mention monitoring changes in the mechanical properties and structure of geopolymers by changing of vibration compacting parameters.
- The available literature does not deal with the comparative study of the development of geopolymer foam based on lignite and brown coal fly ash with different origins and compositions.
- Among the factors influencing the properties of foam geopolymers, besides the amount of foaming agent, the literature deals with the liquid/solid ratio affecting the workability of the geopolymer paste. However, it does not deal with changes in the flow (rheological) properties of the geopolymer paste as a result of grinding the raw material.
- Furthermore, according to the available data, it does not monitor the changes in the structure of the fly ash based geopolymer during foam formation (gas formation). In particular, the micro and macro structure of the finished foam product is investigated.

2. The objective of my scientific work

In connection with the above, the main objective of my research is to develop fly ash based geopolymers with special properties (high density as well as foam products) whose properties (compactness, strength, porosity, etc.) can be consciously controlled by different methods (e.g. grinding of raw material, changing compaction parameters).

During my research I intend to achieve the following sub-goals:

- Investigation of the effect of the applied geopolymer compression method on the product properties.
- To investigate the applicability of lignite and brown coal fly ash of various origins, composition (lime content) for geopolymer foam production.
- Controlling the workability (flow properties) of the geopolymer paste and the formation of foam by grinding the raw material.

- To study the effect of the mechanical activation of the raw material on the geopolymer foam microstructure and mechanical properties (compressive strength, specimen density, porosity).
- Tracking the structural changes in geopolymer formed during foaming.
- To investigate the varying degree of relationship between compressive strength and specimen density of geopolymers and geopolymer foams.

3. Description of raw materials, experimental equipment, applied measurement methods

Materials

Two types of fly ash were used for the experiments, such as lignite and brown coal (F-type) fly ashes. The lignite fly ash was originated directly from the electrostatic precipitator system of the Mátra Power Plant, while the brown coal fly ash was sampled from the fly ash landfill near Tiszaújváros. The lignite fly ash had high SiO_2 and Al_2O_3 content (88.03 w/w%), the SiO_2/Al_2O_3 ratio was 2.3. The brown coal fly ash contained a smaller amount of these two components (62.52 w/w %) with SiO_2/Al_2O_3=3.34. The particle size distribution of fly ashes was also very different. The median particle size of the brown coal fly ash was x_{50}=78 µm, while the median size of the lignite fly ash was x_{50}=48.4 µm. The activator solution consisted of a mixture of NaOH solution and water glass (sodium-silicate). For the preparation of the geopolymer foam, Al powder and H_2O_2 solution were used as foaming agents.

Experimental equipment, measurement methods

During my experiments at the Institute of Raw Material Preparation and Environmental Processing, I used a laboratory ball mill with an internal diameter of 305 mm and a length of 305 mm for grinding of the fly ash samples. For determination of particle size distribution and calculated specific surface area of raw materials (raw and ground fly ashes) a Horiba LA-950 V2 laser particle size analyzer was used.

Aanton Paar Physica MCR 51 rotary rheometer was used to determine the flow properties of the mixture (geopolymer paste) of different fineness of fly ash and activator. According to its measurement principle, the liquid (or suspension) to be measured is placed between two coaxial cylindrical surfaces (Couette flow). The angular velocity of the rotated cylinder is proportional to the shear rate (γ) and the torque applied to the stationary cylinder is proportional to the shear stress (τ) in the liquid (or suspension).

The chemical (oxidic) composition of the initial materials was determined using a WD-XRF RIGAKU Supermini 200 WDXRF.

By Fourier-transform infrared spectroscopy (FT-IR) methods, the vibrations and periodic oscillations of bonds (bound atomic groups) in molecules or crystalline materials can be investigated, thus indirectly detecting chemical and local structural information (*Hegman et al. 2011*). Measurements were performed using the Jasco FT-IR 4200 in reflection mode using diamond ATR.

The composition of the starting materials and geopolymer foam samples was determined by X-ray powder diffraction, the main parameters of the apparatus were: XRD, Bruker D8 Advance, Cu-Kα radiation, 40kV and 40mA generator plant, parallel beam geometry with spherical mirrors, Vantee- 1 detector with aperture 1º, with a measurement time of 0.007° (2θ)/24 sec.

During the preparation of geopolymers, it is important to achieve a high degree of mixing of the raw materials, i.e. the homogeneous nature of the geopolymer paste. I used a lab mixer to mix the ingredients.

A laboratory vibration table was used for geopolymer compaction experiments, whereby cylindrical and other moulds could be fixed by means of a cross strap placed on integrated threaded rods.

For heat treatment of geopolymer and geopolymer foam specimens Nabertherm L(T)3 laboratory static furnace was used, while an SDL Atlas G212-D1 conditioning chamber was used to store specimens at a given temperature (23 °C) and humidity (90%) until the specimens were strength tested.

The compressive strength of the specimens was determined by a uniaxial compression test. During the experiment, on the cylindrical specimens placed between the parallel steel plates was bring about axial compression and loading by slowly approximating the plates. The load was applied until the specimen failed. The mechanical test was carried out by a SZF-1 type hydraulic compression testing machine with a maximum load of 25 kN (i.e. 2.5 tons).

4. Investigation plan

Figure 1 illustrates the complex investigation plan starting from the knowledge of the properties of the initial material to the characteristics of the geopolymer product made from it.

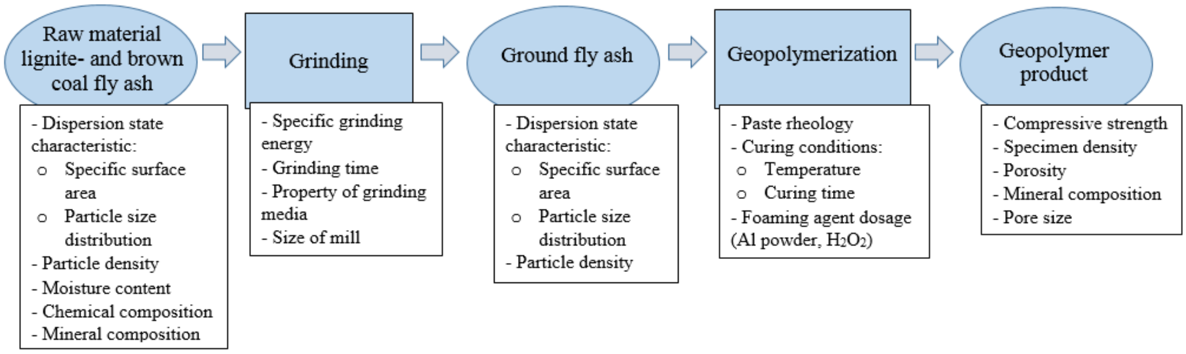


Figure 1 Complex investigation plan

The experiments started with the determination of the mean properties of the starting materials, including the particle size distribution and geometric (outer) surface area, as well as the material properties such as particle density, chemical composition and mineral composition. During grinding, only the grinding time was changed among the operating parameters. Similarly to the starting material, the ground fly ash resulted after grinding are characterized by their dispersion properties and grain density. The paste produced as a first step in geopolymerization processes is characterized by its rheological properties. The geopolymerization process is influenced by the heat treatment conditions (temperature, curing time), the optimum choice of these parameters are key for geopolymer production. In addition to the flow properties of the paste, the effect of foaming agent addition was also investigated. The products obtained at the end of the geopolymerization were characterized by physical properties (compressive strength, specimen density, porosity) and material structure properties (mineral composition).

Geopolymer and geopolymer foam manufacturing technology

The technological flowsheet for the lignite and brown coal fly ash based geopolymer and geopolymer foam production methods is illustrated in *Figure 2*.

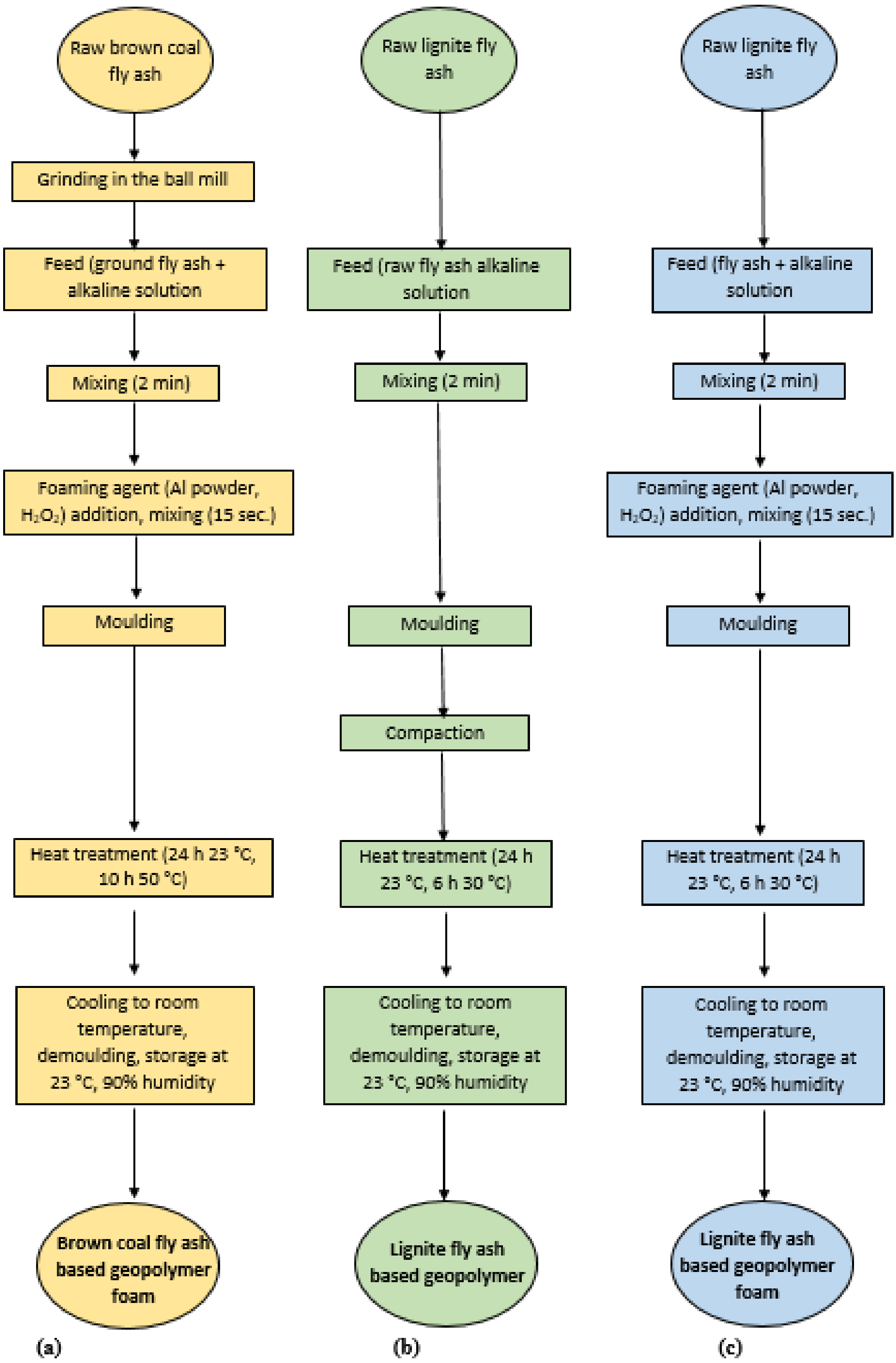


Figure 2 Technological flowsheet of the geopolymer (b) and geopolymer foam (a and c) manufacture

5. Scientific results, theses

Thesis 1.

Investigations showed that the compressive strength and specimen density of lignite fly ash based geopolymers were mainly influenced by vibration deflection amplitude and compression time in the investigated range. With optimized compression parameters, the strength of the geopolymers can reach maximum value.

1/a.

During the investigations it was found that the compressive strength of the geopolymers changed with the increase of the vibration amplitude of deflection in both x (horizontal) and y (vertical) direction. In addition, the higher directional-y deflection favored the formation of a higher strength geopolymer.

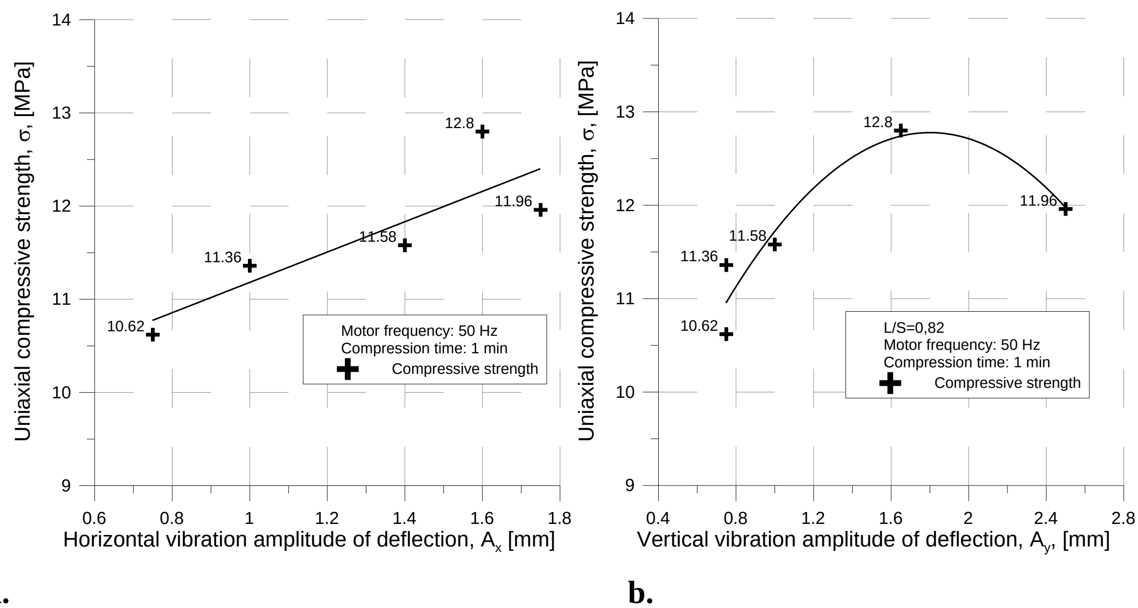


Figure 3 Effect of vibration amplitude of deflection on uniaxial compressive strength of geopolymers. a - Horizontal (x direction), b - Vertical (y direction) vibration amplitude of deflection

1/b.

Based on the experimental results, it is possible to determine an optimal compaction time, where the strength of the geopolymers takes maximum value.

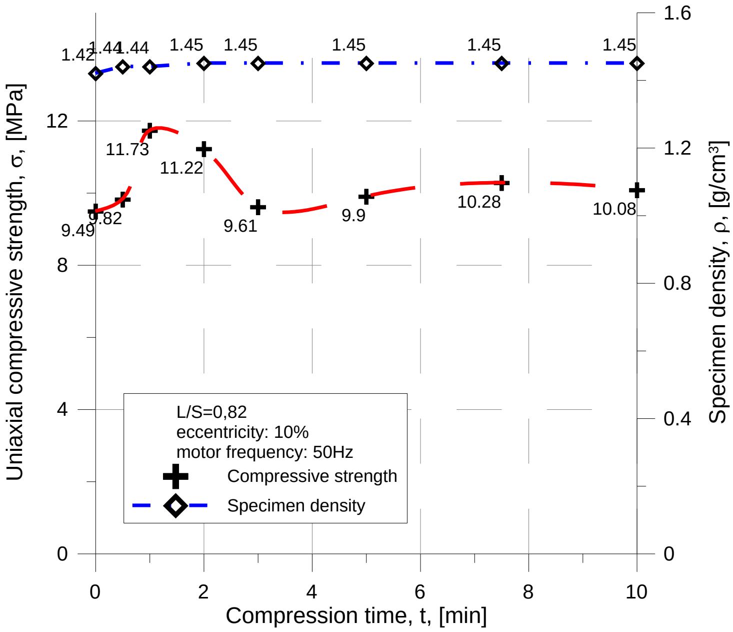
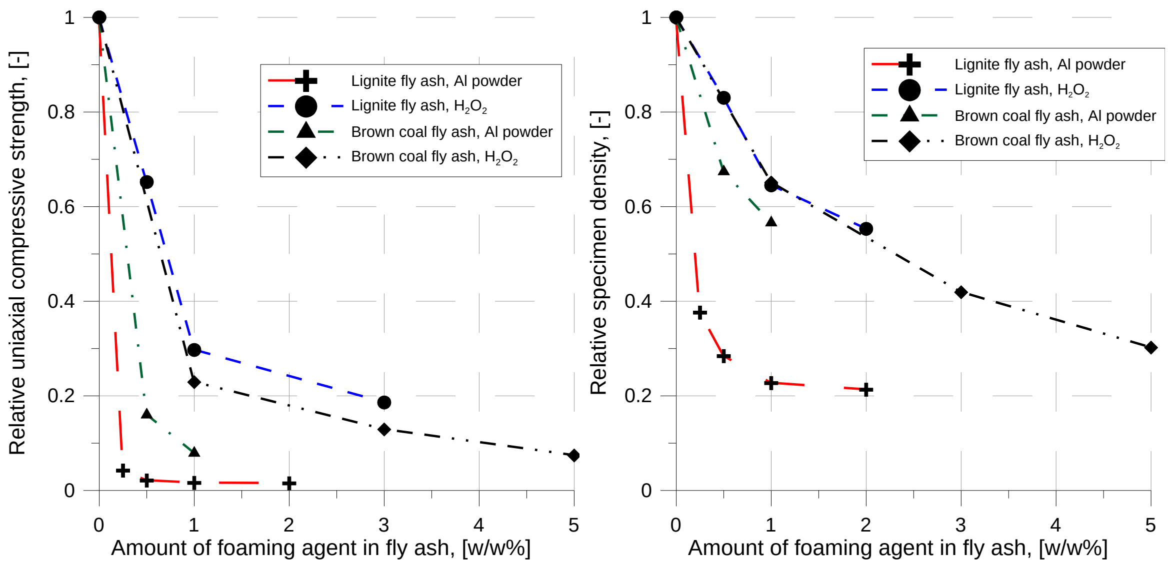


Figure 4 Effect of compression time on the compressive strength and specimen density of the geopolymer

Thesis 2.

Based on the experimental results, it was found that the changes in compressive strength and specimen density of lignite and brown coal fly ash based geopolymer foams as function of foaming agent concentration can be described typically by similar trend, but depending on the fly ash type and foaming agent type it is characterized by varying degrees of intensity.



a. **b.**

Figure 5 Effect of applied foaming agent on the uniaxial compressive strength (a) and the specimen density (b) of geopolymer foams

Thesis 3.

Based on the X-ray diffraction measurement results, I found that the foaming agents used (Al powder, H_2O_2) induced phase changes in both lignite fly ash and brown coal fly ash based geopolymer foams. In the case of lignite fly ash based geopolymer foams (LGPF), by the addition of both foaming agents it was formed thenardite (Na_2SO_4), while in case of the brown coal fly ash based geopolymer foams (BGPF) with using Al powder, it was formed gibbsite [γ-Al(OH)$_3$].

Table 1. Mineral phases of lignite fly ash based geopolymer foams using different foaming agents

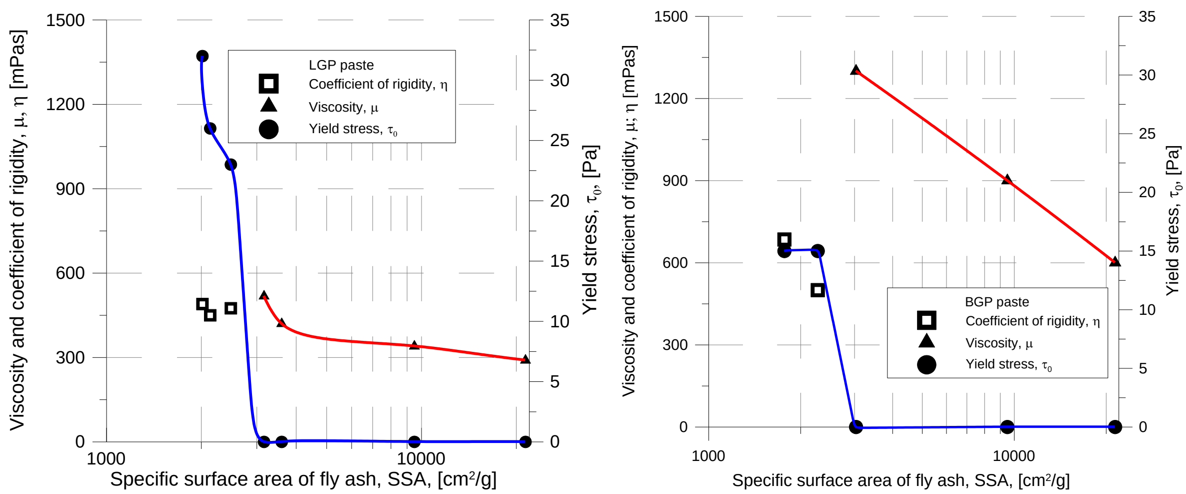
Phase	**LGP**	**LGPH 0,25% Al**	**LGPH 2% Al**	**LGPH 0,5% H_2O_2**	**LGPH 3% H_2O_2**
	w/w%				
Quarz	15,1	12,8	13,5	14,5	**11,3**
Mullite 2:1	1,1	**0,3**	**0,2**	0,9	0,8
Maghemite	4,9	4,3	3,9	5,3	3,7
Albite	1,7	2,2	2,4	**0,0**	1,5
Calcite	4,0	7,5	5,3	4,1	7,5
Akermanite	3,9	4,0	3,6	4,5	3,3
Hematite	3,7	3,7	2,8	3,4	2,8
Oligoclase An16	7,6	8,3	6,1	**11,7**	5,4
Thenardite	-	-	**3,3**	**0,6**	**1,8**
Anhydrite	-	-	0,2	-	-
Rutile	-	-	0,8	-	-
Amorphous	58,0	57,0	58,0	55,0	**62,0**
sum	100,0	100,0	100,0	100,0	100,0

Table 2. Mineral phases of brown coal fly ash based geopolymer foams using different foaming agents

Fázis neve	**BGPH**	**BGPH 0,5 % Al**	**BGPH 1% Al**	**BGPH 1% H_2O_2**	**BGPH 3% H_2O_2**
	w/w%				
Quarz	2,0	2,6	2,3	2,5	1,9
Mullite 2:1	11,2	13,1	12,4	12,6	10,5
Cristobalite low	1,0	1,0	1,0	1,1	0,5
Maghemite	0,5	0,7	0,5	0,6	0,4
Cristobalite high	0,2	0,1	0,1	0,1	0,1
Gibbsite	-	**1,4**	**2,7**	-	-
Thermonatrite	-	-	-	-	0,5
amorphous	85,0	81,0	81,0	83,0	86,0
sum	100	100	100	100	100,0

Thesis 4.

It was found that the geopolymer paste with a given fly ash content (55 w/w%), with originally non-Newtonian (Bingham-plastic) behaviour, became Newtonian liquid - regardless of the fly ash type (lignite or brown coal)- over a given specific surface area of fly ash (about 3000 cm^2/g) due to increasing the fly ash fineness.

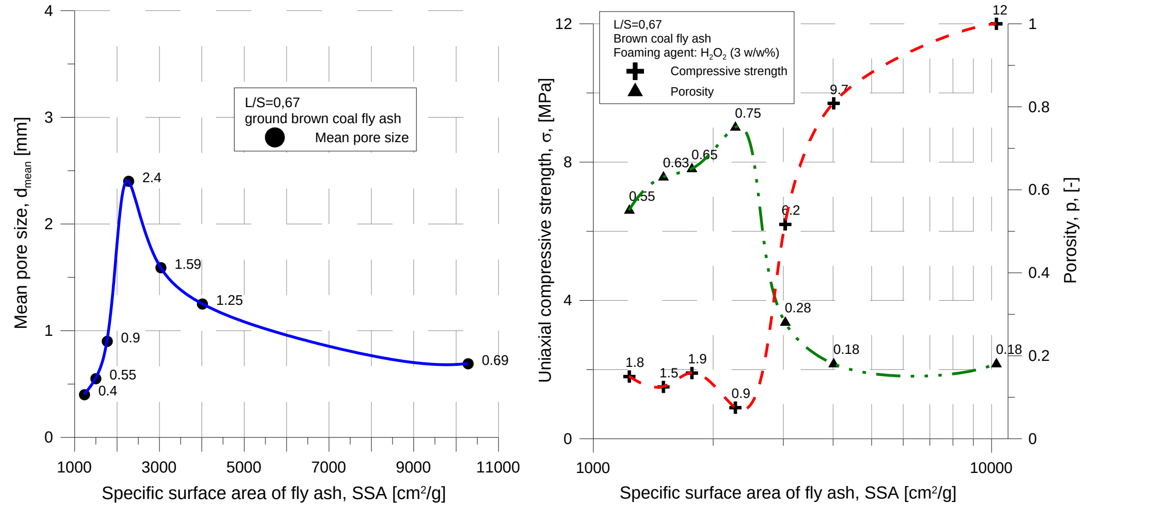


a. **b.**

Figure 6 Effect of grinding fineness on the flow behavior of lignite (a) and brown coal fly ash based geopolymer pastes

Thesis 5.

Based on the experimental results I found that the flow behavior of the geopolymer paste significantly influenced the porosity, the size and the amount of the pores, and thus the compressive strength of the brown coal fly ash based geopolymer foam made using hydrogen peroxide foaming agent.

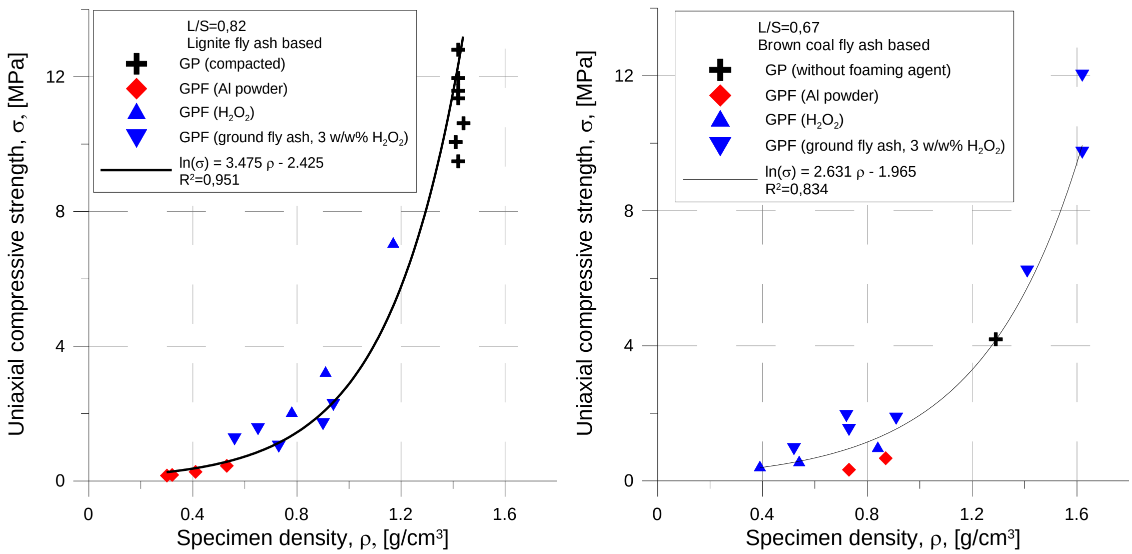


a. **b.**

Figure 7 Effect of brown coal fly ash fineness on the pore size (a) and on the compressive strength and porosity of the geopolymer foam (b)

Thesis 6.

I found that the relationship between compressive strength and specimen density of geopolymer foams and geopolymers with the same composition, but with different fly ash fineness, can be characterized by an exponential function.



a. **b.**

Figure 8 Relationship between compressive strength and specimen density of lignite fly ash based (a) and brown coal fly ash based (b) geopolymers and foams

Table 3 Relationship between compressive strength and specimen density of lignite fly ash based) and brown coal fly ash based) geopolymers and foams

Type of fly ash	**Equation**	R^2
lignite	$\ln(\sigma) = 3.475\ \rho - 2.425$	0,951
brown coal	$\ln(\sigma) = 2.631\ \rho - 1.965$	0,834

6. List of publications in the field of dissertation

International journal articles

1. Gábor Mucsi, Ádám Rácz, Zoltán Molnár, Roland Szabó, Imre Gombkötő, Ákos Debreczeni (2014): Synergetic use of lignite fly ash and metallurgical converter slag in geopolymer concrete. *Mining Science* 21 pp. 43-55.
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3. Gábor Mucsi, Roland Szabó, Ádám Rácz, Ferenc Kristály, Sanjay Kumar (2019): Combined utilization of red mud and mechanically activated fly ash in geopolymers. *Rudarsko Geolosko Naftni Zbornik - Mining Gelological Petroleum Engineering Bulletin* 44 (1) pp. 27-36.

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6. Szabó Roland (2019): Lignit pernye alapú geopolimerek mechanikai tulajdonságainak szabályozása vibrációs tömörítéssel. *Építőanyag* 71 (2), pp. 66-71.

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1. Gábor Mucsi, Ákos Debreczeni, Roland Szabó, Zoltán Molnár (2014): Development of lignite based geopolymer. In: 28th microCAD International Multidisciplinary Scientific Conference, Konferencia helye, ideje: Miskolc, Hungary, University of Miskolc, 2014. 04. 09.-10. paper A7.
2. Gábor Mucsi, János Lakatos, Zoltán Molnár, Roland Szabó (2014): Development of geopolymer using industrial waste materials. Book Series: International Conference on Environmental Engineering (ICEE), Konferencia helye, ideje: Vilnius Litvánia, 2014. 05. 22-23. paper 39.
3. Gábor Mucsi, Ádám Rácz, Zoltán Molnár, Roland Szabó, Ákos Debreczeni (2014): Effect of heat curing on lignite fly ash-based geopolymers. 18th International Conference on Waste Recycling, Konferencia helye, ideje: Miskolc, Magyarország, University of Miskolc, 2014. 10. 09. pp. 1-7.
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6. Gábor Mucsi, Roland Szabó (2015): Properties of lignite fly ash based geopolymer foam. In: XIX. Waste Recycling Conference, Abstract, Konferencia helye, ideje: Krakkó, Lengyelország, 2015. 10. 22-23.
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8. Szabó Roland, Nagy Sándor (2017): Geopolimerek mechanikai tulajdonságainak szabályozása tömörítéssel. In: Kékesi, Tamás (szerk.) MultiScience - XXXI. microCAD International Multidisciplinary Scientific Conference, Konferencia helye, ideje: Miskolc, Magyarország, Miskolci Egyetem, 2017. 04. 20.-21. pp. 1-8.
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