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PREPARATION OF GEOPOLYMER FOAMS USING AUTOCLAVE CURING

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Geopolymers are potential substitute materials of traditional building materials. Geopolymer foams are also perspective materials for different foamed construction materials, like autoclave aerated concretes. In this research geopolymer foams (GPF) were prepared using fly ash, sodium-hydroxide alkaline activator and aluminum paste as foaming agent. Different curing conditions (curing in furnace and curing in autoclave) were applied on the samples. Density and compressive strength were determined, SEM and FTIR tests were done on GPFs, as well. The results show that compressive strength is highly affected by the pore forming mechanisms, which influencing the porosity of GPF, however the autoclave curing has less impact on the strength.

Keywords: autoclave, compressive strength, foam, FTIR, geopolymer

INTRODUCTION

Geopolymers are artificial materials, which can be an alternative for substituting conventional concrete [1]. These materials are inorganic polymer-structured binders. As source materials different aluminosilicate materials are used. For the geopolymerization reaction alkali activator is required, which is mixed together with raw materials. During the mixing process exothermic reaction is occur, then a so-called "artificial stone" is formed [2], [3]. As solid source different waste materials, by-products (e.g. cuttings, brick powder, fly ash, red mud, concrete powder, blast furnace slag) and natural sources (e.g. metakaolin) can be used, as well [4], [5], [6]. There are beneficial properties of these materials: fire and heat resistance, excellent mechanical strength, low shrinkage, excellent immobilising capability of heavy metals [1].

Like concrete, geopolymers can be foamed, as well. In order to prepare low density, high porosity foamed materials, aluminum powder (or paste), or hydrogen-peroxide are generally used [7], [8], [9], [10], [11], [12], [13], [14], [15].

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Researches are mainly focusing on the usability of different secondary raw materials and industrial wastes (for example incinerator bottom ash, ground granulated blast furnace slag, recycled aluminum foil) as geopolymer raw materials [16], [17], [18], [19], [20] and curing conditions. However, there are only a few researches dealt with autoclave cured foamed geopolymers. *Table 1* summarizes the main results of these researches.

Table 1

Curing method, conditions and some results of autoclaved geopolymers

Ref.	Curing method	Curing conditions	Result of curing
[21]	Dry autoclave curing	Day 1: T = 80 °C, p = 0.2 MPa Day 2: T = 80 °C, p = 0.2 MPa Day 3: drying at 80 °C	bulk density decreased; water absorption increased; compressive strength decreased
[22]	Autoclave curing	T = 150 °C; p = 2 MPa; curing time: 3 hours	compressive strength increased
[23]	Autoclave curing	T = 80–120 °C; p = 0.1 MPa; curing time: 4–48 hours	density of geopolymer can be achieve: 1,000–1,700 kg/m^3
[24]	Autoclave curing	T = 150 °C; curing time: 2 hours	increase in compressive strength
[25]	Climatic chamber or furnace or autoclave can be used for harden the geopolymer at higher temperatures.	T_{min} = 40 °C	curing time of geopolymer is shorter
[26]	Heating chamber or hot air oven or autoclave	T = 80 °C; p = 2.05 MPa; curing time: 1–24 hours	increase in compressive strength; decrease in thermal conductivity

This study focuses on the autoclave curing in order to improve the properties of geopolymer foams.

1. MATERIALS AND METHODS

1.1. Sample preparation

Geopolymer foams (GPF) were prepared using Class F type fly ash and sodium-hydroxide (NaOH) alkaline activator. Fly ash was first dried before mixing after that it was mixed with alkaline activator. Finally, aluminum paste (as a foaming agent) was added to the mix. Standard mixing time was used until homogenous mortar was obtained. After mixing, samples were poured into cylindrical plastic moulds with a diameter of 55 mm and 100 mm of height. The samples were demoulded in the next day, then were placed 1) into furnace for 6 hours at 60 °C (reference samples) and 2) into autoclave for 6 hours at different temperatures and pressures.

1.2. Curing conditions

Geopolymer foam samples were cured using the following conditions according to *Table 2*.

Table 2

Curing conditions of geopolymer foams

Curing method	Curing conditions		
Curing in furnace	60 °C		
Curing in autoclave	2 bar/60 °C	2 bar/75 °C	2 bar/90 °C
	4 bar/60 °C	4 bar/75 °C	4 bar/90 °C
	7 bar/60 °C	7 bar/75 °C	7 bar/90 °C

In order to compare the influence of curing in furnace with autoclave, curing time in furnace and in autoclave was set to 6 hours. The required temperature was reached in the autoclave in 2 hours after that 4 hours of dwell time was applied. At the end of curing passive cooling was used to reach room temperature.

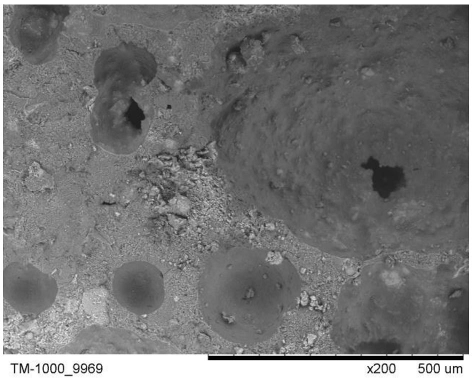
1.3. Testing methods

Fourier-transformation infrared spectroscopy (FTIR) was applied to determine organic components within the samples. FTIR analysis is an appropriate method to study the structural evolution of amorphous alumino-silicates. Infrared absorption bands enable the identification of structures and specific molecular components, as well. Bruker Tensor 27 equipped with ATR cell was used to measure the absorbance of powdered GPF samples. Background spectrum was measured at every single run. The resolution was 4 cm^{-1}, sample scan time was set to 64 in the measuring range of 4000 cm^{-1} – 500 cm^{-1}. To observe the microstructure and cell structure of GPF samples scanning electronmicroscopy was used (Hitachi TM-1000). Electronmicrographs were taken in different magnifications. Uniaxial compressive strength tests were done on GPF samples. Instron 5566 type universal testing machine were used for mechanical tests. Cylindrical samples were tested using 5 mm/min rate compression.

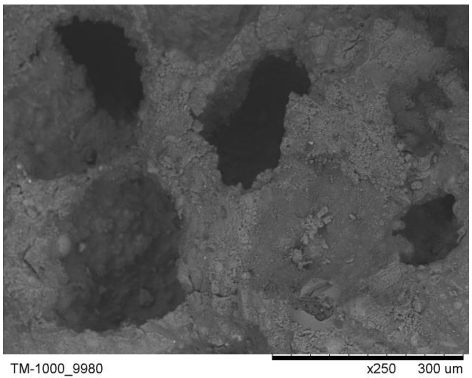
2. Results and discussion

2.1. Microstructure of geopolymer foams

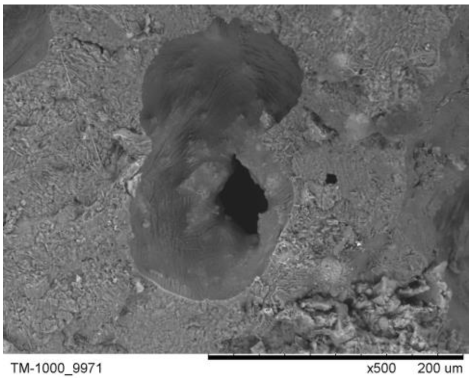
Figure 1 shows the microstructure of geopolymer foams cured at different methods. The SEM micrographs show the surface characteristics of extremas (reference samples cured in furnace; other samples cured in autoclave at 7 bars, 90 °C). In both cases large cavities/cells can be shown, which were generated by the evolved H_2 gas from the reaction of Al paste and water. The sizes of cells are varied from 50 μm to 1 mm and the distribution of cells are inhomogeneous. Observing the microstructure at higher magnification, a matrix can be seen, which attributes to the geopolymer matrix. As it is shown, the structure of this matrix was changed by applying high pressure and temperature. This matrix is more coherent, in which not dissolved fly ash particles are taken place. It refers to a strengthened geopolymer structure.



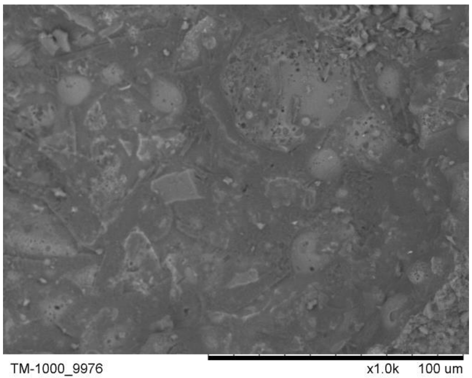
Cured in furnace at 60 °C (M = 200X)



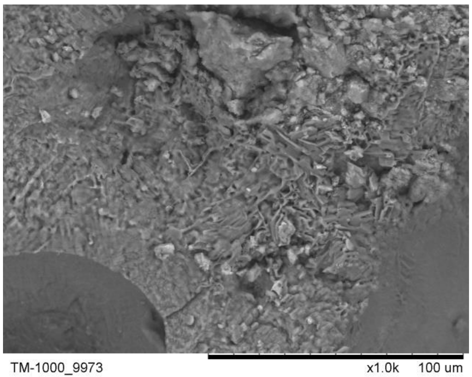
Cured in autoclave at 90 °C, 7 bar (M = 250X)



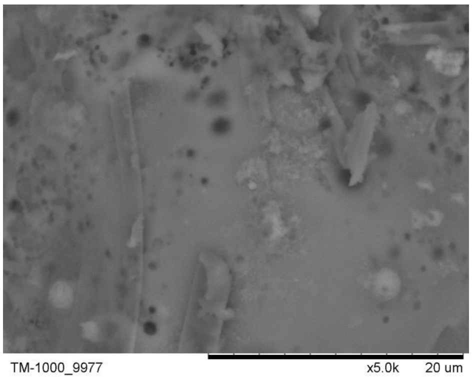
Cured in furnace at 60 °C (M = 500X)



Cured in autoclave at 90 °C, 7 bar (M = 1000X)



Cured in furnace at 60 °C (M = 1000X)



Cured in autoclave at 90 °C, 7 bar (M = 5000X)

Figure 1

SEM micrographs of GPF samples cured at different conditions

2.2. Fourier transformation infrared spectroscopy

As an example *Figure 2* shows the FTIR spectras of geopolymer foams cured in furnace and in autoclave at 7 bars. *Table 3* contains the characteristic FTIR bands of the prepared geopolymer foams.

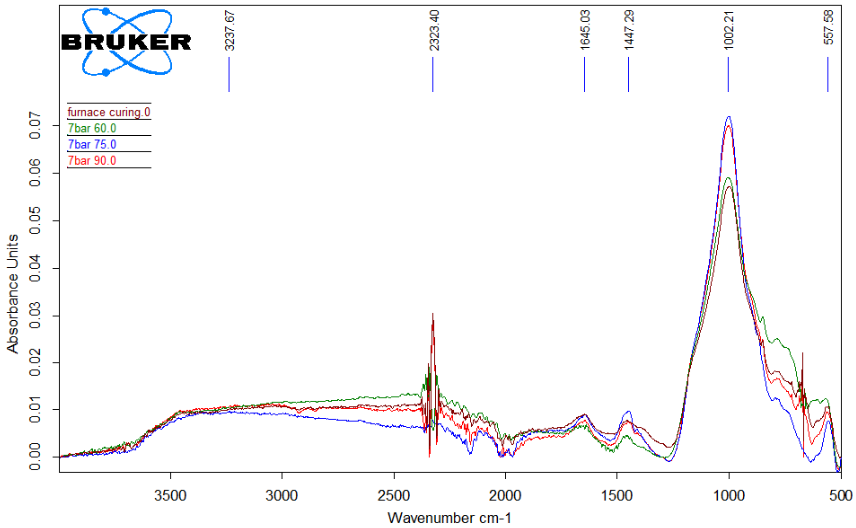


Figure 2

FTIR spectras of GPF samples cured at different temperatures and 7 bar pressure

Table 3

Characteristic FTIR bands of geopolymer foams

Peak position [cm^{-1}]	**Assignment**
3,050–3,240	Stretching vibration (–OH, H–OH)
1,640–1,645	Asymmetric bending vibration (H–OH)
1,445–1,460	Stretching vibration (O–C–O)
995–1,005	Asymmetric stretching vibration (T–O–Si, T=Si or Al)
850	Si-O stretching, OH bending (Si–OH)
775–790	Symmetric stretching vibration (Si–O–Si)
664–677	Symmetric stretching vibration (Si–O–Si and Al–O–Al)
557–566	Symmetric stretching vibration (Si–O–Si and Al–O–Al)

According to *Table 3* the following conclusion can be made. The peaks in the region of 3,050–3,240 cm^{-1} belong to the stretching vibrations of –OH and H–OH of bound water molecules, which are absorbed on the surface or entrapped in the large cells of the foam structure. The peaks in the region of 995–1,005 cm^{-1} are also confirm the formation of geopolymer material. The main peak on the spectras are attributed to the Si–O stretching and OH bending vibrations and the asymmetric stretching vibrations of T–O–Si (whereas T means tetrahedrally bonded Si or Al). In the lowest region (557–790 cm^{-1}) the symmetric stretching vibrations of Si–O–Si and Al–O–Al are represented. In the range of 1,445–1,460 cm^{-1} the peaks attributed to the stretching vibrations of O–C–O. These peaks pointed out the presence of $NaHCO_3$, which can be formed during the carbonation of NaOH solution in air.

The effect of curing temperature and pressure on the structure of geopolymer foams can also observable. 2 bars and 4 bars of autoclaving pressure have not changed the structure of geopolymer foams. In these cases, the same peaks were detected with a little peak shifts, however, 7 bars of autoclaving pressure effected on the structure. In the range of 557–1,002 cm^{-1} no peaks were detected, which shows, that there were no symmetric stretching vibrations of Si–O–Si and Al–O–Al. Si–O stretching and OH bending vibrations were not detected, as well. Generally speaking, the higher the curing temperature the higher the intensity of peaks.

2.3. Density changes of geopolymers foams

As a foamed structure, density of geopolymer foams is an important property. As foamed materials are mainly used as a thermal insulator, it is important to reach a low density during production.

During the sample preparation the density of geopolymer foams were set to an average density of 0.5 g/cm^3. After curing in autoclave the dimensions and weight were measured on the samples in order to determine their density. The average values are shown in *Figure 3*.

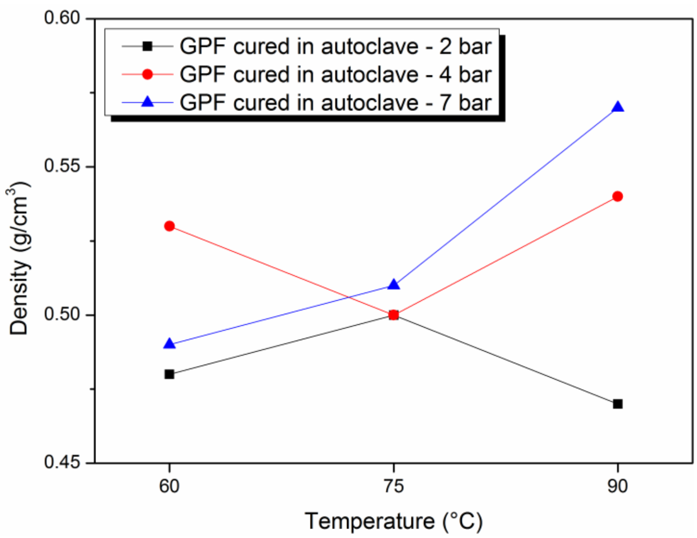


Figure 3

Density changes of geopolymer foams cured in autoclave

It can be concluded, that curing pressure and temperature have an effect on the density of GPFs. Using 2 bars of pressure the density curve has a local maximum point at T = 75 °C. Inversely, with the increase of pressure up to 4 bars a local minimum value can be shown at T = 75 °C, as well. 7 bars of autoclaving pressure unambiguously increasing the density. According to this, there was some kind of change within the GPF structure.

2.4. Compressive strength tests of geopolymer foams

As density, compressive strength is also an important material property of GPFs. *Figure 4* shows the results of uniaxial compressive strength tests.

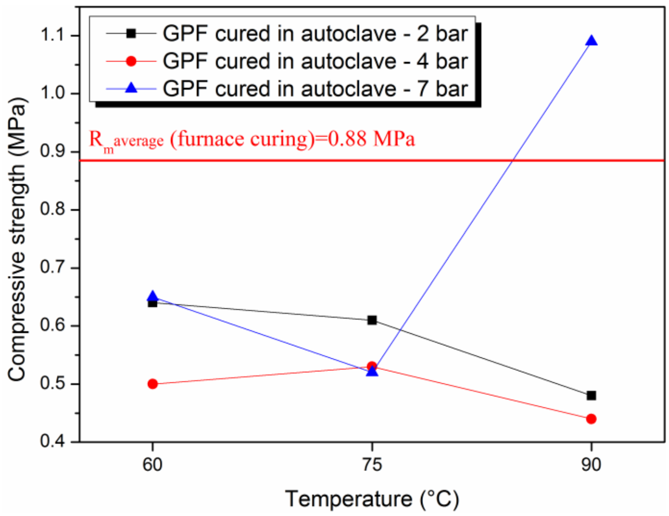


Figure 4

Compressive strength of geopolymer foams cured in autoclave

As it is shown in *Figure 4* increasing the curing temperature up to 75 °C only the samples cured at 4 bar were slightly increased. The highest curing temperature resulted more decrease in the compressive strength, however at 7 bars of pressure compressive strength was increased ($R_{m,\,7\,bar,\,90\,°C}$ = 1.09 MPa).

It can be concluded, that there is no clear correlation between compressive strength and density of samples cured in autoclave. Only at 90 °C and 7 bars of pressure in the autoclave shows some correlation. The reason of this could be the coherent geopolymer matrix, which were above mentioned.

CONCLUSION

The aim of this research was to study the effect of autoclaving curing on the properties of geopolymer foams. According to the results the following conclusion can be made:

- The structure of the geopolymer matrix was changed by applying high pressure (7 bars) and temperature (90 °C). These curing conditions resulted a strengthened geopolymer structure. These changes cannot be observed at lower temperatures and pressures.
- 7 bars of autoclaving pressure effected on the structure of geopolymer foams. FTIR test results confirm, that in the range of 557–1,002 cm^{-1} there were no peaks detected, therefore structural changes occurred.
- The highest compressive strength was reached at 7 bar at 90 °C, however autoclaving effects less on the compressive strength.
- Overall it can be said, that using autoclave for curing geopolymer foams, structural changes can be reach only at high pressure and high temperature.

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