



Thermal insulating foamy geopolymers from perlite

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ABSTRACT

Geopolymerization is an emerging technology which utilizes solid aluminosilicate raw materials that are easily soluble in caustic solutions, in order to produce inorganic polymers with excellent physical, mechanical and thermal properties. In this paper is demonstrated the ability of geopolymerization technology for production of thermal insulating foamy inorganic polymers utilizing as solid raw material ultrafine perlite which is a by-product from comminution and sizing operations of perlite exploitation. Hydrogen peroxide is used as a chemical blowing agent for the foaming of inorganic polymers. The effect of addition of the blowing agent on the thermophysical properties of thermal insulating materials is demonstrated and these properties are compared with the ones of the commercial thermal insulating materials indicating the high potentiality for the development of this new family of inorganic polymeric materials.

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1. Introduction

The energy consumption in the sectors of households and services for the European Union of 27 member states amounted 388.46 Mtoe in 1990 and increased further to 455.18 Mtoe in 2006, which constitutes an increase of 17% (EU energy and transport, 2009). The major part of this consumption is attributed to the buildings' heating, ventilation and air-conditioning. The figures in the north European Mediterranean countries are even worse. For the same time period, the increase in energy consumption in the sectors of households and services was 101.6% in Greece, 93% in Spain, 86.5% in Portugal, 184.2% in Cyprus, 30.6% in Italy and 16.66% in Malta (EU energy and transport, 2009). In order to invert this trend in energy consumption, EU adopted specific legislation on the energy performance of buildings (Directives 2002/91/EC, 2005/32/EC and 2006/32/EC), which is a cost effective way of improving energy security and fighting against climate change. In this environment, the principles of bioclimatic design and thermal insulation in buildings (Giama and Papadopoulos, 2007) grew up and became basic design tools for the sector of building construction.

The most important thermal insulating materials for buildings (Karamanos et al., 2005) are classified in two categories; (a) organic and (b) inorganic materials. The European market of organic materials is dominated by the foamy polystyrene products extruded polystyrene (XPS) and Expanded Polystyrene (EPS). The most widely used inorganic materials are the fibrous glass wool and stone wool which belong to the same group of mineral wool.

Although both organic and inorganic materials are good insulating materials they have a number of technical limitations. Polystyrene is highly flammable or easily ignited and is classified as a B2 or B3 product according to DIN4102. When polystyrene is burned (Department of Energy 1; Department of Energy 2; Hawley et al., 1984) at high temperatures (800–900 °C) or at lower temperatures (as in case of household fireplace or in a campfire) polycyclic aromatic hydrocarbons (PAHs) can be produced creating serious health effects. Although the available fire-retardant grades are inflammable, they could possibly create health and environmental risks due to the use of brominated flame retardants such as hexabromocyclododecane (EPA, 2008). Expanded polystyrene breaks down gradually when exposed to direct sunlight or to temperatures higher than 80 °C and reacts with common solvents (Bynum, 2001; Papadopoulos, 2005; Al-Homoud, 2005). The blowing agent for the production of EPS is usually pentane which may pose a flammability hazard in manufacturing and storage of new products. Carbon dioxide or hydrochlorofluorocarbons (HCFCs) are used as propellant gases for the production of XPS having obvious effects on ozone depletion and global warming (Papadopoulos, 2005). Finally, the maximum temperature at which polystyrene products can be exposed without losing their thermophysical properties is 75–80 °C (Papadopoulos, 2005). Mineral wool insulation is cheaper than the organic ones and is non-flammable classified as an A1 or A2 product according to DIN4102 (Papadopoulos, 2005; Karamanos et al., 2005). In addition, it can withstand very high temperatures (500–750 °C) without losing its thermophysical properties (Papadopoulos, 2005). Two are the main drawbacks related to the utilization of fibrous mineral wool (Bynum, 2001; Papadopoulos, 2005; Al-Homoud, 2005): (a) it has very low

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resistance to vapour diffusion and therefore has the tendency to increasing thermal conductivity upon vapour absorption and (b) it has the potential to cause damage on humans' health. It can irritate the skin, eyes and respiratory system and therefore precautions are needed during production and handling of material. The material is reviewed by the International Agency for Research on Cancer (IARC) as an agent belonging to group 3 – not classifiable as to carcinogenicity to humans – (IARC). Therefore, there is open field for development of a new thermal insulating material which would potentially have the advantages of common organic and inorganic insulating materials without having their drawbacks.

The geopolymerization technology is a low cost, green technology (Davidovits, 2005) that can transform a variety of solid amorphous silicate and aluminosilicate raw materials (Papias et al., 2007; Maragos et al., 2008; Dimas et al., 2009; Xu and van Deventer, 2000; Palomo et al., 1999) to useful products with high added value (Papias et al., 2007; Maragos et al., 2008; Dimas et al., 2009; Palomo et al., 1999; Cheng and Chiu, 2003; Wu and Sun, 2007; Pinto and Vieira, 2005). Perlite is an amorphous aluminosilicate volcanic glass which has the potential to be utilized as a raw material in geopolymerization technology. Therefore, this paper deals with the production of perlite based geopolymers which comprise the skeleton of a new family of inorganic thermal insulating foamy materials that have excellent thermophysical properties, are non-flammable and safe for humans and environment.

2. Experimental

2.1. Materials

The raw materials used for the synthesis of foamy geopolymers were perlite, sodium hydroxide and hydrogen peroxide. The chem-

ical analysis of perlite determined by X-ray Fluorescence (XRF) is given in Table 1 while its mineralogical analysis determined by X-ray Diffraction (XRD) is presented in Fig. 1. Perlite is an amorphous volcanic glass rich in SiO₂ and Al₂O₃ which contains some crystalline phases as biotite, albite, quartz and calcite. The raw perlite used in this work was a by-product from comminution and sizing operations of perlite exploitation and therefore was extremely fine with mean particle size $d_{50} = 7.13 \mu\text{m}$ measured on a MALVERN Laser Particle Size Analyzer. The specific gravity of perlite measured according to the ASTM D854-06 standard using a water pycnometer was 2.34.

The alkaline activator used for the synthesis of geopolymers was a sodium hydroxide solution that was prepared by dissolving anhydrous sodium hydroxide pellets (Merck Chemicals, 99.5% purity) in deionised water.

A hydrogen peroxide (H₂O₂) solution 30% w/w (Merck Chemicals) was used as a chemical blowing agent for the foaming of the geopolymeric pastes. H₂O₂ is thermodynamically unstable and therefore can be easily decomposed to water and oxygen gas (reaction (1)) with the latter playing the role of the geopolymeric paste blowing agent:



2.2. Experimental procedures

The geopolymeric viscous paste was prepared by mixing mechanically perlite with sodium hydroxide solution. The prepared paste remained at ambient temperature for 1 h and then the necessary amount of the hydrogen peroxide (H₂O₂) solution was added under mechanical mixing so that the sodium hydroxide concentration in the aqueous phase to be 8 M and the solid to liquid ratio to be 1.2 g/mL. The % w/w H₂O₂ content in the prepared pastes was varied in-between 0.6% and 2.9%. The resulted paste was molded in appropriate open plastic molds and was cured at 35 °C for 2 h. During this period H₂O₂ was decomposed rapidly and the resulting oxygen blew up the paste forming the foamy materials. Then, the curing procedure was continued at 65 °C where the materials remained for 24 h. After curing, the specimens were de-molded and kept in a dry atmosphere. The foamy specimens were prepared in two different geometries; discs with 110 mm diameter and 10 mm thickness for the measurement of thermal conductivity and cubes with side 50 mm for the measurement of compressive strength.

Table 1
Chemical analysis of perlite.

Species	% w/w
SiO ₂	71.58
Al ₂ O ₃	13.23
K ₂ O	4.02
Na ₂ O	3.36
CaO	2.12
Fe ₂ O ₃	1.83
MgO	1.04
H ₂ O (chemical/combined)	2.5
Total	99.68

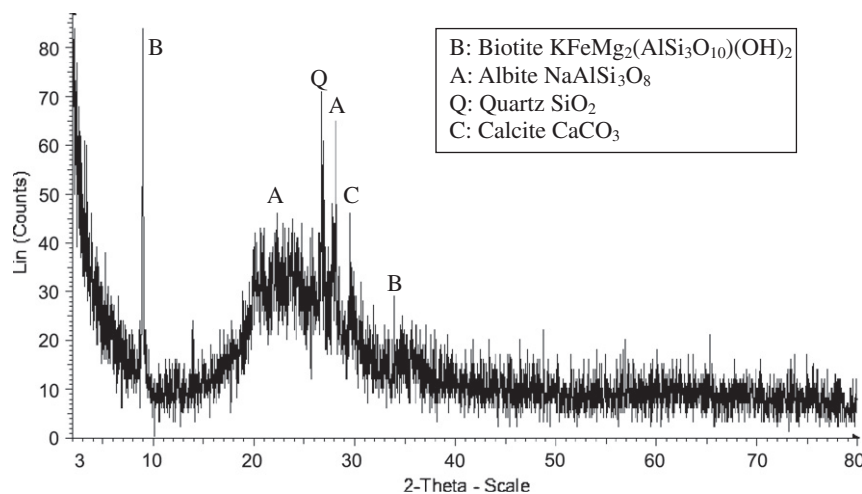


Fig. 1. X-ray diffractogram of perlite.

2.3. Analysis and tests

The apparent density (ρ) of the foamy geopolymers was determined by measuring the weight of specimens with a precision balance as well as their dimensions with a precision electronic calliper. The density (d) of the geopolymeric matrix was measured according to the ASTM D854-06 standard using a water pycnometer. The % cell volume (C) of the foamy materials was determined according to the following formula:

$$C = \frac{(1 - \rho) \cdot 100}{d} \quad (2)$$

The thermal conductivity was measured with a thermal conductivity analyser (Mathis TCi, SETARAM) utilizing the modified transient plane source technique. The compressive strength was measured on a testing machine of the Structural Behavior Engineering Laboratories Inc. (PLT-10 model). For the determination of the material properties two specimens were always tested and the average value of measurements was reported as the result. Finally, the cell structure was examined by scanning electron microscopy (SEM) on a JOEL JSM-type scanning electron microscope.

3. Results and discussion

3.1. Properties of the foamy materials

The effect of H_2O_2 content in the geopolymeric paste on the apparent density as well as % cell volume of foamy materials is shown in Fig. 2. Two regions can be seen; (a) the region I in-between 0.7% and 2% w/w H_2O_2 where the apparent density decreases sharply from 665 kg/m^3 to 335 kg/m^3 as the H_2O_2 content in the geopolymeric paste increases accompanied by a substantial increase of the cell volume from almost 74–87% and (b) the region II for H_2O_2 content higher than 2% w/w where the change of the apparent density as well as the % cell volume is practically limited and the foamy materials tend to have an apparent density a little bit higher or lower than 300 kg/m^3 and a cell volume varied in-between 87% and 89%. This behaviour can be understood in view of the foaming mechanism of the geopolymeric paste. The decomposition of hydrogen peroxide liberates oxygen creating initially very small bubbles inside the mass of the very viscous paste which behaves as a non-Newtonian plastic or pseudo-plastic fluid. The pressure exerted on the bubbles' wall plays the role of the shear stress for the viscous paste which is deformed

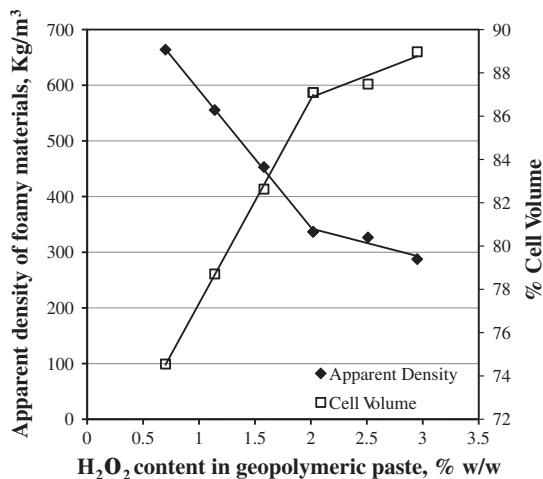


Fig. 2. Apparent density and % cell volume of foamy materials as a function of % w/w H_2O_2 content in the geopolymeric paste.

causing the bubbles' expansion and the foaming of the paste. The bubbles' expansion causes a decrease of the oxygen pressure under constant temperature eliminating gradually the shear stress and therefore stopping the bubbles' expansion and the paste's foaming. When the oxygen volume per paste volume is low enough (the case of low H_2O_2 content), the spherical bubbles are discernible and they have low population density in the geopolymeric paste. As the H_2O_2 content increases, the bubbles' population density increases as well as the amount of oxygen in each bubble due to the increased local oxygen concentration. Therefore, the bubble size increases, the thickness of the cells as well as the apparent density of foams decrease and the cell volume increases (region I – linear part with high gradient). When the oxygen volume per paste volume exceeds a threshold, the bubbles' population density and the amount of oxygen per bubble increase substantially resulting the aggregation of a number of cells creating a network of cells partially open to environment and thus causing marginal expansion and marginal increase to the cell size (region II – linear part with small gradient).

The microstructure of foamy materials is presented in Fig. 3. The cells are normally closed and almost spherical when the % content of H_2O_2 in the paste is low. Aggregation among cells takes place as the % content of H_2O_2 increases changing the cells' geometrical shape from spherical to oval, creating a network of interconnecting cells and affecting the mean cell size as is seen in Figs. 3 and 4.

The mean cell size is 700 μm at the lowest studied H_2O_2 content of 0.7% increasing almost linearly with the H_2O_2 content and reaching at the value of 1.9 mm at 2.02% H_2O_2 in the geopolymeric paste. Further increase of H_2O_2 content up to the highest studied value of 2.95% causes marginal changes on cell size, which reaches the value of 2.08 mm, explaining thus the limited increase of cell volume as well as the decrease of apparent density (Fig. 2) of foamy materials in the same range of % H_2O_2 content.

The thermal conductivity of foamy materials is shown in Fig. 5. Thermal conductivity follows identical trend with apparent density decreasing substantially from 0.053 W/m K to 0.03 W/m K as the H_2O_2 content ranging in-between 0.7% and 2.02%. Further increase of the H_2O_2 content in the geopolymeric paste to the studied value of 2.95% w/w has not a measurable effect on the thermal conductivity value. The results showed that the thermal conductivity of foamy geopolymers is correlated to their cellular structure and a substantial decrease of the thermal conductivity value can be achieved by controlling appropriately the cells type, size, shape and volume. The results are in good agreement with the state-of-the-art on thermal insulation foamed plastics where the thermal conductivity is correlated firstly to the thermal conductivity of the filling gas followed by the apparent density of foams and then the morphology of the cellular structure (Weber et al., 2005).

The compressive strength of foamy materials is shown in Fig. 6. The fracture of materials resembles the behaviour of rocks for H_2O_2 content in the geopolymeric paste higher than 1.58%. In this case the materials have almost the same compressive strength of 780 kPa. For H_2O_2 content lower than 1.58%, the materials have more plasticity due to incomplete geopolymerization because the network of small and closed cells hinders the water removal during the curing period. Therefore, the incomplete geopolymerized foamed materials have compressive strength ranging in-between 250 and 780 kPa.

3.2. Comparison with the other current state-of-the-art insulating materials

A comparison among the state-of-the-art commercially available organic and inorganic insulating materials as well as of the best foamy perlite geopolymer produced in this work (with

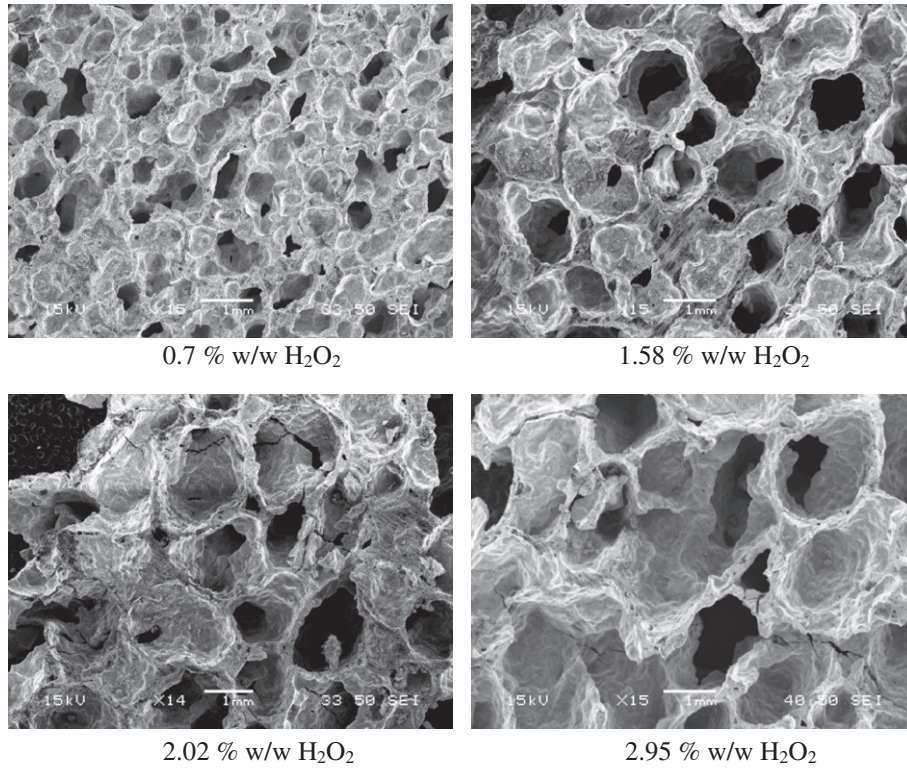


Fig. 3. SEM photos of foamy materials prepared with different % w/w H₂O₂ content in the geopolymeric paste.

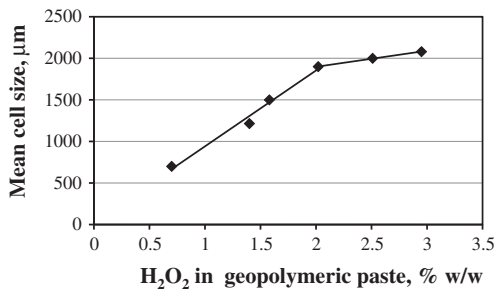


Fig. 4. Mean cell size as a function % w/w H₂O₂ content in the geopolymeric paste.

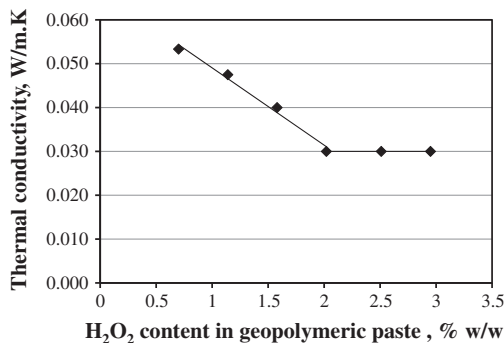


Fig. 5. Thermal conductivity of foamy materials as function % w/w H₂O₂ content in the geopolymeric paste.

2.9% w/w H₂O₂ in the geopolymeric paste) is shown in Table 2. The foamy perlite geopolymer is a very good thermal insulating material. Its thermal conductivity value of 0.03 W/m K is directly comparable with the lowest ones of the organic materials (XPS and EPS) as well as of inorganic materials (glass and mineral wool).

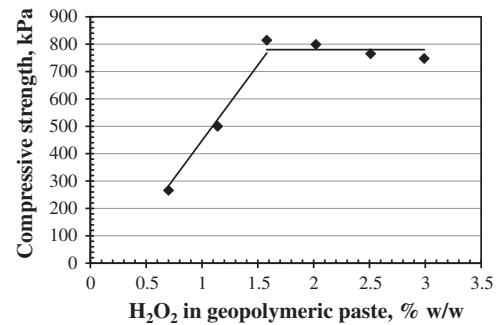


Fig. 6. Compressive strength of foamy materials as function % w/w H₂O₂ content in the geopolymeric paste.

The maximum application temperature of foamy perlite geopolymer is 700 °C which is substantially higher than the ones of organic materials, higher than the one of glass wool and directly comparable of the one of mineral wool. The foamy perlite geopolymer does not melt in the temperature region in-between 700 and 1000 °C but it softens due to the glassy nature of perlite. The perlitic water trapped in the structure of perlite particles vaporizes and therefore expand the foamy geopolymer decreasing its apparent density from 290 kg/m³ to 160 kg/m³ without affecting the value of thermal conductivity. The geometrical features (shape and dimensions) of foamy geopolymers change during the softening procedure and for this reason the temperature of 700 °C is chosen as the maximum application temperature.

The 100% inorganic nature of foamy perlite geopolymer renders it totally non-combustible categorizing it in the Fire Class A1 in accordance to the standard DIN4102. The foamy perlite geopolymer has superior fire resistant properties in comparison to the other common organic and inorganic insulating materials shown in Table 2. The commercial inorganic insulating materials such as

Table 2

Typical values of the basic thermophysical properties of common insulating materials.

Material	Glass wool	Mineral wool	Extruded polystyrene, XPS	Expanded polystyrene, EPS	Foamy perlite geopolymer
Apparent density, kg/m ³	13–100	30–180	20–80	18–50	290
Thermal conductivity, W/m K	0.03–0.045	0.033–0.045	0.025–0.035	0.029–0.041	0.03
Maximum application temperature, °C	500	750	75	80	700
Fire Class	A1–A2	A1–A2	B1–B2	B1–B2	A1
Compressive strength at 10% deformation, kPa	16 (1)	0.01–69 (1), (2), (3)	100–700 (4)	70–450 (5), (6), (7)	780 ^a

The most values have been taken from Papadopoulos (2005).

(1) National Insulation Co.

(2) Buska and Maciulaitis (2007).

(3) Industrial Insulation Group.

(4) Extruded Polystyrene Foam Association.

(5) EPS Molders Association.

(6) Desjarlais and Zarr (2002).

(7) Quality Foam.

^a The material is fractured at 2% deformation.

mineral and glass wool are often characterized in the A2 Fire Class (about 98% non-combustible) because they contain a combustible organic binder which binds the inorganic fibres creating the marketable products in the form of panels or felts.

The fractural behaviour of foamy perlite geopolymers resembles the one of rocks and is totally different from the behaviour of the other commercial inorganic and organic insulating materials shown in Table 2. The foamy geopolymer has not plasticity or elasticity and has enough high compressive strength for the practical application of buildings' insulation. In general, it has superior mechanical properties in relation to the other common insulating materials.

The relative high value of apparent density is the most important drawback of foamy perlite geopolymer in relation to the other common insulating materials. The achieved value of 290 kg/m³ is almost 1.6 and 3.6 times higher than the corresponding ones of mineral wool and extruded polystyrene which are commonly used for buildings' insulation. Improving apparent density is a target of great importance for the commercial application of foamy perlite geopolymers. This target could be achieved by the application of one of the three following actions or by the synergetic effect of all of them:

- Foaming of geopolymeric paste by blowing air under controlled pressure and volumetric flow rate (mechanical foaming) instead of blowing by chemical means as it was applied in this work. Mechanical foaming can achieve better cells' structure and % cell volume higher than 90% decreasing the apparent density of foamy materials up to 150–200 kg/m³.
- Use of expanded instead of non-expanded perlite as a raw material for synthesis of geopolymers. Taking into account that the bulk density of expanded perlite is in-between 30 and 150 kg/m³ while the bulk density of non-expanded perlite is about 1100 kg/m³, it could be assumed that foamy geopolymers based on expanded perlite would have apparent density in-between 20 and 100 kg/m³ which is a value directly comparable with the apparent densities of the common commercial insulating materials. Although this work did not study the expanded perlite as a raw material for geopolymerization, the thermal tests for determination of the maximum application temperature of foamy perlite geopolymer showed that at temperatures in-between 700 and 1000 °C the apparent density of foamy materials decreased to a value of 160 kg/m³ due to incomplete perlite expansion. Therefore, this result offer indirect evidence that the above

stated assumption is not an illusion and could be a good estimate for the apparent density of foamy expanded perlite geopolymers.

- The temperature at which the green foamy perlite geopolymers are cured can affect the apparent density of final materials. As is seen in Fig. 7, 8–10% of the weight of foamy materials is physically adsorbed water which can be easily removed at curing temperatures around 100 °C decreasing the apparent density of materials.

Finally, water absorption measured by the resistant to vapour diffusion factor and/or humidity assimilation rate is a very important physical property that can affect the thermal conductivity of insulating materials. The foamy perlite geopolymers, especially the ones that have been produced with high H₂O₂ content in the geopolymeric paste, can absorb water and have low resistant to vapour diffusion factor, because they are consisting of networks of interconnecting cells some of which are partially open to the environment, increasing their thermal conductivity value. This is the other drawback of the foamy perlite geopolymer produced in this work. To avoid this drawback, the foamy perlite geopolymer has to be provided, where necessary, with a very thin geopolymeric surface cover with very low permeability (Maragos et al., 2008).

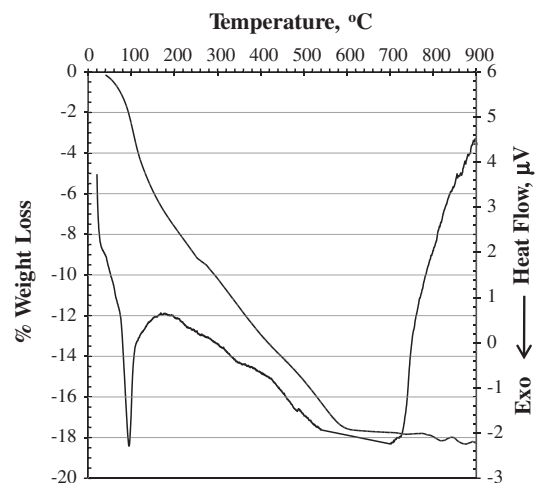


Fig. 7. Typical TG/DTA curves for foamy perlite materials which have been synthesized with 2.98% w/w H₂O₂ content in the geopolymeric paste.

4. Conclusions

Foamy geopolymers from non-expanded perlite proved to be very good thermal insulating materials. In comparison with the commercial organic (expanded and extruded polystyrene) and inorganic (glass and stone wool) thermal insulating materials, the foamy geopolymers from perlite have (a) almost similar thermal conductivity (0.03 W/m K); (b) superior compressive strength (780 kPa at 2% deformation) and a fracture behaviour resembling the one of rocks; (c) superior fire resistant properties (100% non-combustible) categorizing them in the Fire Class A1 according to DIN4102; (d) superior maximum application temperature (700 °C) only comparable with the one of mineral wool, which render the material proper not only for buildings' thermal insulation but also for industrial insulation applications where high temperatures occur.

At the current stage of development, foamy geopolymers from non-expanded perlite have the two following drawbacks: (a) relatively high apparent density (290 kg/m³) and (b) high water absorption capacity. The most promising way to decrease the apparent density at a level comparable to the ones of commercial organic and inorganic thermal insulating materials is the synthesis of foamy geopolymers utilizing (a) expanded instead of non-expanded perlite as a solid raw material and (b) mechanical foaming of geopolymeric paste by blowing air under controlled pressure and volumetric flow rate. Finally, an effective way to increase the resistant to vapour diffusion factor is to cover the surface of foamy geopolymers with a thin layer from a compact geopolymer with low permeability which is a practice always applied to materials with open or partially open cellular structure.

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