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# Production and characterization of lightweight vermiculite/ geopolymer-based panels



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#### ABSTRACT

The production and the properties of lightweight composite panels, with expanded vermiculite as lightweight aggregate and geopolymer as binder, were investigated. Different compositions of the geopolymer binders (metakaolin or alumina-based) and two sizes of expanded vermiculite were tested. The produced composites were subjected to microstructural analyses, as well as to thermal and mechanical tests. Densities ranged between 700 and 900 kg/m³, while the average strength and thermal conductivity were about 2 MPa and 0.2 W/mK, respectively. Results show that lightweight composites can be produced with satisfactory density and mechanical and thermal properties compared with other materials used in building sector, such as plasterboard or cellular concrete

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#### 1. Introduction

In the last few years, numerous studies were made on cellular or lightweight concrete materials [1–14], with thermal conductivity in the range 0.1–1 W/mK. In accordance with the sustainable development policies in buildings and constructions [15–17], the use of lightweight materials allows to reduce both the weight of the structure (i.e., the dead load [12]) diminishing the use of raw materials and wastes, and heat transfer preserving operational energy [18] and fostering better indoor thermo-hygrometric comfort conditions.

The use of lightweight (expanded) aggregates, namely vermiculite, perlite, pumice, etc. with different kinds of inorganic binders, such as cement, gypsum or geopolymers, allows the production of composites masonry blocks, walls and panels with reduced apparent density, good mechanical performances and improved thermal properties (insulation, refractoriness and fire resistance) [6–14].

In particular, vermiculite is a mineral of the group of hydromicas, whose chemical composition consists of a complex hydrated aluminum and magnesium silicate. It can expand to 8–20 times its original thickness (exfoliation) upon heating to above 300 °C. Expanded vermiculite aggregate is formed by thin plates separated by air gaps, becoming a highly effective heat-insulating material. Expanded vermiculite can be used as filler for high temperature-resistant insulating materials thanks to high thermal stability, owing to its ability to relax temperature stress during heating [6,19].

Among possible binders, geopolymers are a class of environmentally friendly and sustainable inorganic aluminosilicate polymers, firstly introduced by Davidovits [20]. Geopolymers are produced by reacting an aluminosilicate powder (metakaolin, fly ashes, slags or any source of silica and alumina) with a highly concentrated alkali solution [20]. Geopolymer nanoprecipitates [21–23] act as a binder for fillers and aggregates producing composite materials that may meet many of the ideal characteristics of lightweight materials for building sector [5,21], such as non-flammability and high temperature resistance without changing physical properties or releasing smoke, corrosion resistance to organic solvents, acids and alkalis, durability, safety for human health and low carbon foot print. All these features are considered a fundamental improvement with respect to traditional cement and concrete technology [3,15–17,20,21].

The aim of the present research is to study geopolymer-based composites with expanded vermiculite as lightweight aggregate to produce precast panels for fire and thermal insulation. Due to the completely inorganic nature [5], expanded vermiculite–geopolymer composites can be included in the Fire Class A1, as totally noncombustible materials in accordance with the European standard EN 13501-1. Contrary to the hydraulic cement, both geopolymer binder and expanded vermiculite do not contain water in their framework, thus preventing degradation at high temperature (e.g., spalling concrete) due to conversion of the structural water into steam [24]. Zuda et al. [13] suggested vermiculite–geopolymer-composite as fire-protecting layers for concrete that overcome the intrinsic limit of concrete structure exposed at fire and thermo-mechanical stress scenarios.

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Table 1
Compositions and characteristics of the raw materials (chemical composition and mean grain size D50 from technical data sheets provided by the suppliers). \*Average values are subject to error due to the great chemical–morphological–dimensional variability of the expanded vermiculite aggregates.

Material	Grade	Supplier	Chemical composition, %							Crystalline phases	D50	
			Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	CaO	MgO		
Metakaolin	M1200S	AGS Minéraux [24]	39.71	53.55	1.50	1.40	0.92	-	0.09	0.15	Quartz Muscovite (traces)	1.7 μm
Alumina	CT 3000 SG	Almatis [26]	99.8	0.015	0.015	-	-	0.03	0.015	0.040	Corundum Bauxite (traces)	0.5 μm
*Expanded vermiculite	Type 2 Type 4	Pull Rhenen [27]	6.5–10.0	35.0-41.0	6.0-9.5	0.6–1.5	4.0-7.0	-	2.0-5.0	20.0-24.0	Micas/phlogopite Micas/phlogopite Hydrated vermiculite	3 mm 10 mm

Different compositions of the geopolymer binders (metakaolin or alumina-based) and two sizes of expanded vermiculite were tested, in order to change the final density and verify the processability of the composite materials. Macro and microstructure, porosity, mechanical strength, thermal behavior and thermal conductivity were also studied and discussed. The knowledge acquired about lightweight geopolymervermiculite composites would be beneficial for future applications (walls, partitions, protecting layers) in buildings and constructions.

#### 2. Experimental procedure

#### 2.1. Raw materials

The geopolymer binders were produced by using ultrafine powders of metakaolin (grade M1200S, AGS Minéraux, Clérac, France [25], specific surface area 19 m²/g, more details are reported in ref. [26]) or alumina (Alumina Almatis CT 3000 SG [27], specific surface area 7.8 m²/g). The composition and characteristics of the raw powders are reported in Table 1. A commercial potassium poly-silicate solution was employed as activator (KSil 35-35, S.r.l. Ingessil Industria Silicati, 33.80 wt.% of potassium silicate, molar ratio SiO<sub>2</sub>/K<sub>2</sub>O = 3.22, pH = 11).

Expanded vermiculite was used as lightweight aggregate. The characteristics and composition of the selected sizes (type 2 and 4, Pull Rhenen [28], The Netherlands, with a density of 95 and 85  $\pm$  20% kg·m $^{-3}$ , respectively) are displayed in Table 1.

#### 2.2. Preparation of geopolymer composite materials

The composition of the mixtures (Table 2) and the processing conditions were initially set up by a trial and error approach. Metakaolin or alumina was mechanically mixed with the potassium poly-silicate aqueous solution for about 10 min. Expanded vermiculite and water (if needed) were subsequently added and thoroughly mixed until uniform mixtures were obtained. Expanded vermiculite was used as received. It is known that vermiculite is generally stable and it doesn't exhibit any potential alkali silica reaction [29] The resulting slurries were cast in silicon rubber molds. A customized curing method was set up for each different mixture in order to achieve gradual water removal and, hence, avoiding excessive shrinkages, planar deformations and cracks formation. In detail, materials prepared with metakaolin were cured for 24 h at RT in closed molds, then for 24 h at 80 °C in closed molds and finally for 48 h in open molds. Materials prepared with

**Table 2**Compositions of the starting mixtures for the production of the composite panels.

Sample	Raw powder wt.%		K-silicate wt.%	H <sub>2</sub> O wt.%	Vermiculite	
	Metakaolin	Alumina			Туре	wt.%
V2-Mk	24	_	47	9	2	20
V4-Mk	26	_	53	-	4	21
V4-Al	-	26	53	-	4	21

alumina powder were cured for 72 h at RT in closed molds, then for 48 h at 80 °C in closed molds and finally 24 h at 80 °C in open molds. At least 4 panels of 55 cm  $\times$  47 cm  $\times$  3 cm were produced for each composition.

#### 2.3. Characterization and analytical techniques

The morphological and micro-structural features of the produced panels were examined by an environmental scanning electron microscope (FEI Quanta200 ESEM™) and by high-resolution photos (scanner Sharp IX330, Japan).

Total open porosity in the range 0.0058–100 µm was determined by Hg intrusion porosimetry (ThermoFinnigan 240).

The stability in distilled water of the samples was checked by complete immersion of cubic specimens (10 mm side) in distilled water at 25 °C for 11 days. Samples were preventively dried in a heater at 100 °C and, after cooling, their mass was measured. Samples were held by thin supports in order to avoid any contact with the bottom of the closed vessel. The mass of wet specimens was measured to calculate the maximum percentage of absorbed water (WS) reached after water saturation, while the weight loss percentage was calculated on the mass of the tested specimens after drying at 100 °C.

The mineralogical composition was evaluated through X-ray diffraction (XRD) (Bruker D8 Advance diffractometer with Cu Ka radiation,  $k=0.15406\,$  nm) before and after thermal treatments at 600 °C, 800 °C, 1000 °C and 1200 °C in an electrical furnace in static air.

The mineralogical composition was evaluated through X-ray powder diffraction (XRD) (Bruker D8 Advance in theta–theta configuration; scanning: 4–80 20; Cu-K $\alpha$  rad.,  $\lambda$  = 0.15406 nm; 40 kV; 40 mA) before



**Fig. 1.** Example of geopolymer–vermiculite panels of 55 cm  $\times$  47 cm  $\times$  3 cm.

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