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2017 IOP Conf. Ser.: Mater. Sci. Eng. 175 012044

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# Ceramic-like open-celled geopolymer foam as a porous substrate for water treatment catalyst

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**Abstract.** This paper presents results from experimental study on microstructural and mechanical properties of geopolymer-based foam filters. The process for making porous ceramic-like geopolymer body was experimentally established, consists of (a) geopolymer paste synthesis, (b) ceramic filler incorporation, (c) coating of open-celled polyurethane foam with geopolymer mixture, (d) rapid setting procedure, (e) thermal treatment. Geopolymer paste was based on potassium silicate solution  $n(\text{SiO}_2)/n(\text{K}_2\text{O})=1.6$  and powder mixture of calcined kaolin and precipitated silica. Various types of ceramic granular filler (alumina, calcined schistous clay and cordierite) were tested in relation to aggregate gradation design and particle size distribution. The small amplitude oscillatory rheometry in strain controlled regime 0.01% with angular frequency 10 rad/s was applied for determination of rheology behavior of prepared mixtures. Thermal treatment conditions were applied in the temperature range 1100 – 1300 °C.

The developed porous ceramic-like foam effectively served as a substrate for highly active nanoparticles of selected  $\text{Fe}^{+2}$  spinels. Such new-type of nanocomposite was tested as a heterogeneous catalyst for technological process of advanced oxidative degradation of resistive antibiotics occurring in waste waters.

## 1. Introduction

Geopolymers are inorganic materials that form long-range, covalently bonded, predominantly amorphous networks [1]. Geopolymers are a class of materials synthesized in alkaline medium with aluminosilicates sourced from calcined clays, natural minerals, industrial wastes etc. and/or from mixtures of these [2]. The reactivity of raw aluminosilicates mainly depends on their chemical properties, surface functionalization, size/morphology of particles and structural composition [3]. In a strong alkaline medium the disaggregation process of Si–O–Si, Al–O–Al and Al–O–Si bonds on the surface of solid particles takes place. Disaggregation/dissolution of aluminates-silicates produces aluminate and silicate species and formation of hydroxylated complexes such as  $\text{Si}(\text{OH})_4^-$ ,  $\text{Al}(\text{OH})_4^-$  or  $\text{Al}(\text{OH})_3^-$  [4]. Through the polycondensation reaction a free tetrahedral unit  $\text{SiO}_4$  and  $\text{AlO}_4$  are linked in an alternating manner to yield a three-dimensional amorphous matrix [5].

Geopolymers display excellent mechanical strength and resistance to attack by aggressive environment, their hardening kinetic is easily adjustable and ceramic-like structure and high melting point indicate structural stability at elevated temperatures [6].

Recently, study on geopolymers as a novel photocatalyst for degradation of dye from wastewater showed a synergetic excellent performance in cleaning process [7]. Therefore, porous geopolymer substrates having high specific area making them ideal candidates for catalyst supports. Many methods



have been explored to synthesize reproducible porous substrates or foams with homogenous pore characteristics. Various processes were applied such as, gaseous method[8], suspension and solidification method through foaming agents[9] and freeze-casting technique [10]. Furthermore, little work has been devoted to the feasibility of making geopolymer-synthesized foam through polymeric sponge method. In this study, we investigated the optimal manufacturing process of the potassium based geopolymer open-cell porous substrate by the polyurethane sponge impregnation.

## 2. Experimental

The geopolymer paste was synthesized as a two component system consists of alkaline potassium silicate solution with silica modulus 1.6 and powder mixture of calcined kaolin clay and precipitated silica as a main source of solid silica and alumina as presented in Table 1 with particle size distribution as follows:  $d_{50}$  at  $3.31\mu\text{m}$ ,  $d_{90}$  at  $6.15\mu\text{m}$ . The mixture was formulated by molar ratios:  $\text{SiO}_2/\text{Al}_2\text{O}_3 = 5$ ,  $\text{SiO}_2/\text{K}_2\text{O} = 3$ ,  $\text{Al}_2\text{O}_3/\text{K}_2\text{O} = 0.6$ ,  $\text{H}_2\text{O}/\text{K}_2\text{O} = 19.5$ . The alumina, schistous clay and cordierite ceramic powders were used as granular filler with maximum grain size  $100\mu\text{m}$  and particle size distribution presented in Table 2.

**Table 1.** The chemical composition of solid components (wt.%)

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	TiO <sub>2</sub>	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>
Powder mixture	58.00	38.10	0.12	0.15	1.45	0.62	1.00

**Table 2.** Mean size and volume average particle distribution

Size distribution	$d_{50}$ [ $\mu\text{m}$ ]	Dv10 [ $\mu\text{m}$ ]	Dv90 [ $\mu\text{m}$ ]
Alumina	28.0	3.9	82.8
Schistous clay	39.9	7.3	82.1
Cordierite	53.0	13.8	99.4

The materials were proportioned by weight ratio and mixed in a laboratory automatic blender for 6 min (1000 rpm). After that, the portion of ceramic filler was added and mixtures were blended for an additional 5 min. The weight portion of different ceramic filler varied in relation to optimal workability of the mixtures. In virtue of this, dynamic rheological measurements were performed using a rotary rheometer (Ares G2, TA Instruments) operated in oscillation mode with small amplitude. Time dependent experiments were performed at an angular frequency 10 rad/sec (1.59 Hz) and strain rate 0.01% in plain-plain geometry. Consequently, polyurethane (PU) porous sponge was immersed into the geopolymer mixture and taken back several times. After that, it was heated in closed container at  $65^\circ\text{C}$  to accelerate rapid hardening mechanism. As a solid substrate the geopolymer/PU sponge was dried at room temperature for the next 24 hours and heat-treated in a kanthal furnace. The temperature was raised up to  $600^\circ\text{C}$  at  $1^\circ\text{C}/\text{min}$  in order to burn out the organic substrate entirely. Then the geopolymer substrate was sintered for 4 hours at various temperatures from 1100 to  $1300^\circ\text{C}$ .

## 3. Results and Discussion

### 3.1. Viscosity of the fresh mixtures

Our investigation of fresh mixtures used in this study indicated the interval of workability corresponding to a proportion of filler added and blended into the geopolymer paste. Based on trial test the maximum weight proportion of filler was defined for each mixture taking into account particle size distribution of ceramic powders. It was determined that geopolymer paste with alumina powder, labeled as a mixture A, consisted of 105 wt.% of ceramic filler. The weight of ceramic filler is expressed as weight ratio to powder part, where 100 wt.% was powder part of geopolymer binder.

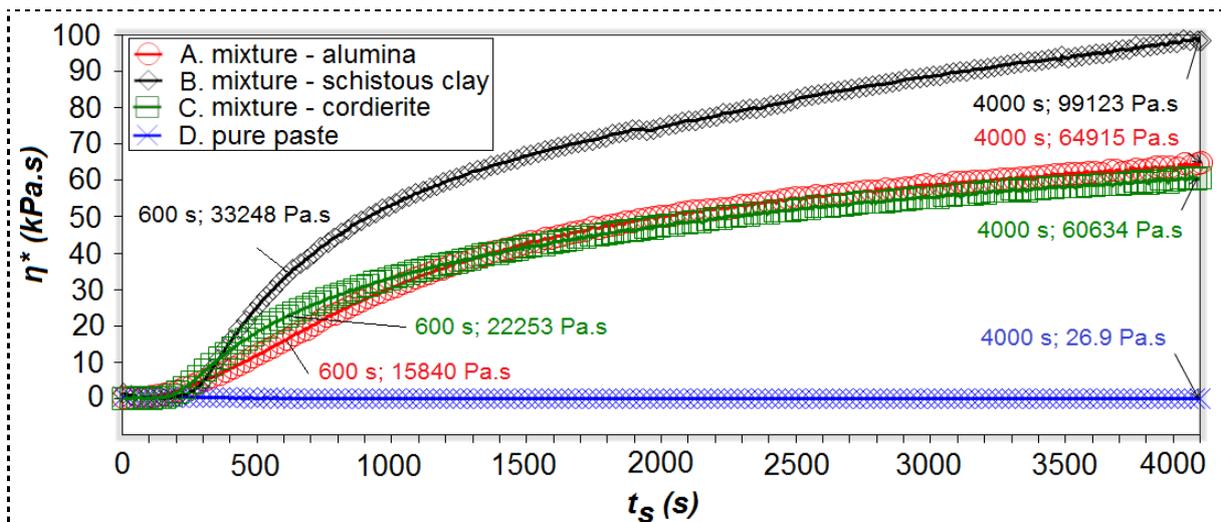
Based on these, geopolymer paste with schistous clay consisted of 210 wt.% (mixture B) and paste with cordierite powder consisted of 120 wt.% (mixture C).

Time dependent measurements confirmed the large interval of workability of potassium based geopolymer mixtures. It should be noted that, the time zero is taken as a + 10 min in virtue of preparation of the mixture for each measurement at  $30\text{ }^{\circ}\text{C} \pm 0.1\text{ }^{\circ}\text{C}$ . The pure paste in interval from 5 to 60 min presented steady state plateau in viscosity ( $\eta$ ) between 18.9 Pa.s and 30.5 Pa.s as presented in Figure 1 (D curve).

The addition of ceramic filler increased viscosity rapidly in comparison with pure paste and emphasized the thixotropic properties of the system paste-filler, even before structural reorganization into three-dimensional aluminasilicate network. The mixtures A, B, C showed concurrent time dependent increase in viscosity after  $\sim 3$  min. in measuring cell, which can be interpreted as a residual stresses relaxed prior to each rheological measurement.

The mixture B showed more increase in viscosity after 10 min of measurement with values over 33 kPa.s. The mixtures A and C revealed similar trend, after 10 min their viscosity ranging between 15  $\sim$  23 kPa.s and gradually raised up to 60  $\sim$  65 kPa.s after 60 min.

As showed, the proportion of ceramic filler markedly affected ultimate viscosity values including viscosity rate in time dependent experiment. Mixture B showed an intensive increase in viscosity with respect to highest proportion of granular filler (210 wt.%) in final mixture. The viscosity of mixture with schistous clay powder reached  $\sim 99$  kPa.s after 60 min.



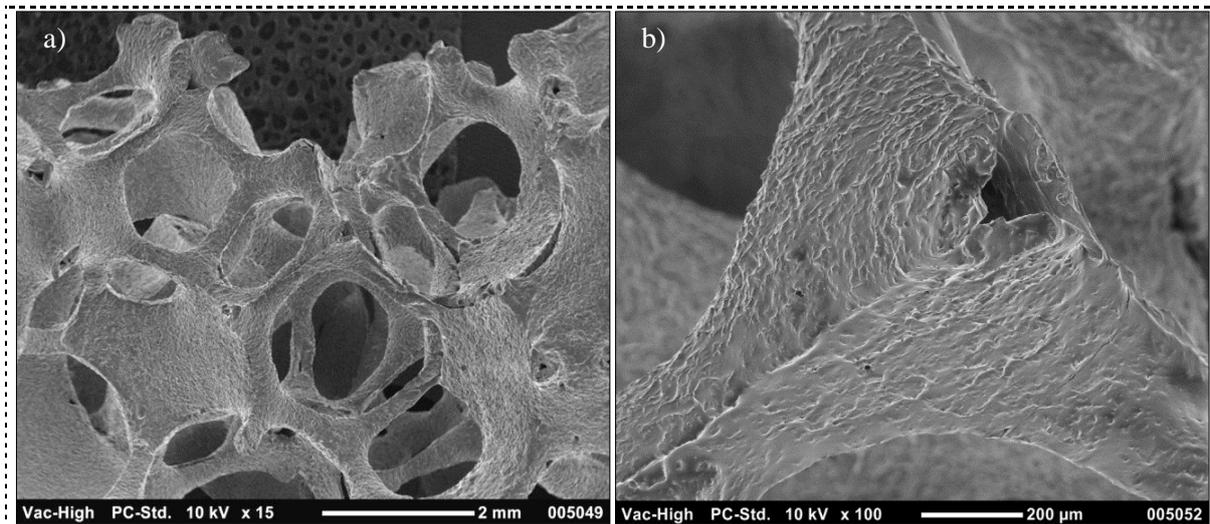
**Figure 1.** Variation of viscosity ( $\eta$ ) as a function of time till  $\sim 60$  min. for pure paste (D), mixture with alumina powder (A), mixture with schistous clay (B) and mixture with cordierite powder (C)

### 3.2. Microstructural and mechanical properties

It can be concluded that by comparing of three types of ceramic filler in geopolymer paste, after thermal treatment at  $1300^{\circ}\text{C}$ , microstructural properties vary in relation to the surface quality and structural integrity. Mixture A with very fine particles of alumina powder ( $D_{v10} = 3.9\mu\text{m}$ ) partially coagulated during blending process and even at lower viscosity (see Figure 1) non-homogenously covered PU sponge during impregnation. Geopolymer substrate after thermal treatment become fragile structure with partially connected struts in solid body of substrate. Compression strength ranging between

0.15 – 0.30 MPa in relation to thermal treatment at 1100 up to  $1300^{\circ}\text{C}$ . A similar results were obtained through mixture C where cordierite powder became fractional sediment after blending process and non-homogenously covered PU substrate due to sharp-edge geometry of the particles.

The homogenous-thick coating of the slurry on the surface of PU sponge revealed mixture B with 210 wt.% of schistous clay powder. Figure 2. shows heat-treated substrate at 1300°C for 4h, partially sintered grains and fully covered amorphous phase on the surface of sponge body with open pore size 0.5~2 mm (a) and hollow strut of geopolymer sponge in detail (b). Compression strength values, on experimental specimens 20x20x20 mm, reached 0.75 MPa after thermal treatment at 1300°C.



**Figure 2.** SEM photos of geopolymer sponge foam, based on mixture B, heat-treated at 1300°C.

#### 4. Conclusion

In this investigation, the mixture B showed an intensive increase in viscosity with respect to highest proportion of granular filler in final mixture and revealed broad interval for workability of the geopolymer slurry. The best conditions for homogenous-thick coating was mixture B with schistous clay filler resulted in compression strength 0.75 MPa after thermal treatment at 1300°C and uniform open pore size 0.5 ~ 2 mm.

#### Acknowledgements

The result was developed within the CENTEM project, reg. no. CZ.1.05/2.1.00/03.0088, cofounded by the ERDF as part of the Ministry of Education, Youth and Sports OP RDI programme and, in the follow-up sustainability stage, supported through CENTEM PLUS (LO1402) by financial means from the Ministry of Education, Youth and Sports under the National Sustainability Programme I.

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