Study on the effect of emulsifiers on the pore structures of geopolymer prepared by emulsion templating

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Abstract
The metakaolin-based porous geopolymer were prepared by emulsion templating method with sunflower oil as the template agent. The effects of the type of emulsifiers with different ionic properties, including sodium stearate (SS), polyacrylamide (PAM), and cetyltrimethylammonium bromide (CTAB), which are respectively belonging to anionic, nonionic and cationic emulsifiers, on the paste viscosity, mechanical strength and pore structure of geopolymer were studied. Fourier transform infrared spectroscopy (FTIR) was used to characterize the influence of emulsion template on the geopolymerization. Scanning electron microscope (SEM) and Image-Pro Plus (IPP) software were used to examine the pore structure of geopolymers, including pore size distribution, average size and roundness of pores. Results indicate that geopolymerization is not significantly affected by the incorporation of emulsion template, while the average size, shape and connectivity of pores in geopolymer were significantly influenced by the type of emulsifiers. With the addition of SS, PAM, and CTAB, the average pore size were 18.1, 27.4, and 11.7 μm, respectively, while the control is 18.9 μm. Both the shape regularity and open porosity of pores were increased with SS and PAM, while no significant changes with CTAB. It is promising to make geopolymer with controllable macro pore structure by adjusting the types of emulsifiers in emulsion templating method by adjusting the oil-water interfacial tension and the particle dispersion.

1. Introduction

Geopolymer refers to a novel cementitious material formed by the depolymerization-polycondensation reaction of aluminosilicate materials (fly ash, metakaolin, slag, etc) under strongly alkaline conditions [1]. Due to its unique zeolite-like three-dimensional network structure, geopolymer possesses some properties similar to glass and ceramic materials, such as excellent strength, fire and corrosion resistance, while giving a low-carbon footprint in the preparation process [2–4]. In addition to be an alternate to traditional Portland cement as structural material, various porous geopolymers have been studied increasingly in recent years for functional applications, such as thermal insulation, adsorbent, filtration membrane and catalyst carrier etc [5–7]. Since the pore structure play a critical role in these applications, it is therefore crucial to explore suitable pore-forming method for preparing a porous geopolymer with controllable pore structures [8, 9].

Emulsion templating method is a strategy that the dispersed phase droplets of emulsion are used to create pores in a solid material by polymerization of the continuous phase [10, 11]. It has been used for the preparation of porous polymers and inorganic ceramic materials for a wide variety of applications [12–14]. One significant advantage of this approach is that the pore structure, e.g. pore size and distribution, can be effectively regulated by changing the types of template and emulsifier [10, 15, 16]. In recent years, emulsion templating method was introduced into the preparation of porous geopolymers [17–20]. Template droplets are homogeneously dispersed and stabilized by the aluminosilicate particles with certain hydrophilicity in the geopolymer paste, and
a porous geopolymer is obtained with the removal of emulsion droplets from hardened paste by thermal treatment or solvent leaching process. Porous geopolymer made with this method gives a multi-scale gradient pore structure, i.e. a combination of micro-sized pores from template droplet and the nano-sized gel pores from geopolymer itself [17, 20]. Different templates, such as canola oil, soybean oil, and hexadecane oil, have been successfully used to prepare porous geopolymers [17, 18, 20]. However, little attention has been paid to the influence of the emulsifiers in the development of porous geopolymer by emulsion templating method. Actually, in addition to the type of template, the emulsifiers play an important role in affecting paste viscosity, pore forming process and the resulting pore structures of porous polymers and inorganic ceramic materials [13, 14]. In the preparation of porous geopolymer by emulsion templating method, some researchers found that the small amount of surfactant (soap molecule) produced by the saponification of template oil may play a key role in pore structure of geopolymer [17]. However, the saponification of triglyceride oil usually requires high temperature and participation of reaction assistants, the reaction degree of saponification is difficult to control and relatively low at room temperature [21], it is difficult to controlling the pore structure of geopolymer rely on the saponification products. Therefore, it is necessary to introduce additional emulsifier into geopolymer paste to prepare geopolymer with controllable pore structure.

In the present study, we aimed to better understand the interactions between emulsifiers and geopolymer matrix and their effect on the pore structure of porous geopolymer. The porous geopolymers are prepared with three types of emulsifier by emulsion templating method. The effects of type of emulsifier on the paste viscosity, mechanical strength and pore structure of geopolymers are investigated.

2. Experimental

2.1. Materials

Commercial metakaolin (MK) produced by Taojinfeng New Materials Co., Ltd (Fujian province, China) and water glass were used to prepare the geopolymer matrix. The chemical composition of metakaolin was determined by x-ray fluorescence (XRF) as 48.43 wt% SiO2, 38.68 wt% Al2O3 and 1.69 wt% CaO. The median particle size (d50) was 3.94 μm. The modulus (SiO2/Na2O molar ratio) of the commercial water glass is 3.18 and the solid content is 34.7%. Deionized water and solid sodium hydroxide (AR, Xilong Scientific Co., Ltd) were used to adjust the modulus of water glass.

Sunflower oil (Mighty, Standard Foods, China) was employed as the template agent, saponification value (SV) of 134. Three different type of surfactants, sodium stearate (SS), polyacrylamide (PAM) and cetyltrimethylammonium bromide (CTAB) were used as emulsifiers. SS is a chemical purity agent from Sinopharm Chemical Reagent Co., Ltd PAM is from Aladdin, with a molecular weight of 200 000, CTAB is an AR agent from Sinopharm Chemical Reagent Co., Ltd SS, PAM and CTAB are belonging to anionic, nonionic and cationic, respectively.

2.2. Sample preparation

The geopolymer was prepared at a theoretical molar ratio of SiO2/Al2O3 = 4, H2O/Na2O = 15. The process of preparing porous geopolymers was shown in figure 1. The modified water glass, with SiO2/Na2O ratio of 1.4 and a solid content of 37%, was prepared by dissolving solid sodium hydroxide in deionized water and then into commercial water glass. It needs to be stored for 24 h. Various emulsifier was firstly added into the modified water glass at a constant 5 wt% (based on modified water glass), and kept mixing for 5 min with a magnetic agitator. Sunflower oil, the template agent, was then added at 20 wt% (based on the total mass of the alkaline solution and metakaolin), and kept mixing at 20 000 rpm for 120 s by a high-speed shear mixer (ULTRA TURRAX ® T18 basic, IKA ®, Germany) to form an alkaline emulsion. Geopolymer paste was then prepared by mechanical mixing the alkaline emulsion and metakaolin in a laboratory mixer for 5 min. Finally, the geopolymer paste was immediately poured into plastic molds of 20 × 20 × 20 mm³ and cured in a standard curing box at 60 ± 2 °C with humidity of 90 ± 1% for 24 h.

The obtained geopolymer blocks were immersed in deionized water at 80 °C in order to remove the template, and the deionized water was replaced every 2 h until it remained clear.

2.3. Methods

Bulk density (ρb) and compressive strength tests were performed according to GB/T 5486-2008. The bulk density of geopolymer block was determined by the ratio of the weight to volume. The specimens were placed in the blast drying oven and raise the temperature gradually to 110 °C, drying for 12 h to constant weight and then cooled down to room temperature. The weight and volume of specimens were measured by electronic balance and Vernier caliper, respectively. The compressive of geopolymer block was measured at 7 days using a universal
Testing Machine (AEC-201) at a loading rate of 2.4 KN S$^{-1}$. Both bulk density and compressive strength results were the averages of at least three specimens.

Total porosity (TP) of geopolymer block was calculated by the equation: $TP(\%) = 100\% \times (1 - \rho_1/\rho_0)$. The $\rho_0$ was defined as the true density of geopolymer and measured according to GB/T 208-1994. Open porosity (OP) was calculated using Archimedes’ Principle with deionized water [22–24].

The viscosity of geopolymer paste was measured by a digital rotary viscometer (NDJ-5S). The functional groups of geopolymer were identified by a Nexus 670 FTIR spectrometer (Nicolet, USA) using the KBr pellet method. Absorbance spectra were collected in the range of 4000–400 cm$^{-1}$ at a resolution of 2 cm$^{-1}$.

The microstructure of the specimen section was characterized by a JSM-6501 scanning electron microscope (SEM, JOEL, Japan) with an accelerating voltage of 15 kV.

The pore structure parameters of geopolymer were obtained from the SEM images using Image-Pro Plus (IPP) software (usually 300 – 500 pores were measured per sample), and the effective pore-size ($D_{sphere}$) was determined by the stereological equation $D_{sphere} = D_{circle}/0.785$ according to ASTM D3576-98 [7, 19, 25], the $D_{circle}$ was defined as the pore size which exported from the IPP software.

3. Results and discussion

3.1. Effect of emulsion template on geopolymerization

The Fourier transform infrared spectroscopy (FTIR) spectra of pure geopolymer, geopolymer before and after template removal are shown in figure 2.

The broad bands at about 3440 cm$^{-1}$ and 1640 cm$^{-1}$ are due to O–H stretching and bending vibrations of adsorbed water, respectively [26]. The broad and strong band at 1030 cm$^{-1}$ is attributed to Si–O–T (T = Si or Al) asymmetric stretching vibration, and the band at about 694 cm$^{-1}$ is attributed to bending vibration of Si–O tetrahedra. These bands indicate the successful geopolymerization in the samples [18, 27, 28].

In the spectra of sample GP/Oil, the weak bands at around 2924 cm$^{-1}$ and 2854 cm$^{-1}$ are attributed to C–H symmetric and asymmetric stretching vibration of methylene group (–CH$_2$–), respectively. The band at 1563 cm$^{-1}$ is related to anti-symmetric stretching vibration of carboxyl group (–COO$^-$), these bands revealed that the presence of the organic phase, sunflower oil, in GP/Oil sample.
The bands of methylene and carboxyl groups are disappeared in the spectra of sample GP, while the type and position of bands that belong to geopolymer are very similar to each other in all of the spectra. It suggests that the product of geopolymerization is not significantly affected by the addition and removal of emulsion template.

3.2. The viscosity and compressive strength of geopolymer

Figure 3 shows the viscosity of geopolymer paste prepared using SS, PAM and CTAB as emulsifier, respectively. It is obvious that the viscosity of geopolymer pastes varies significantly with the types of emulsifiers. Compare to the control sample, the viscosity of the paste with SS is significantly reduced, which is only about 1/6 of the control group. The viscosities of pastes with the PAM and CTAB groups are increased significantly, which are about 4.7 and 21 times higher than that of the control, respectively.

The compressive strength of geopolymer prepared with different emulsifiers is shown in figure 4. Compared to the control, the addition of SS or PAM does not show much influence on the compressive strength of geopolymers. A slight increase, about 7% with the addition of SS, while about 5% decrease with PAM. The compressive strength of the geopolymer with CTAB is 6.1 MPa, being decreased about 27%.

3.3. The pore structure of geopolymer

The pore structures of geopolymer prepared with different emulsifiers are listed in table 1. The bulk density and total porosity of geopolymer are in the range of 0.63 ~ 0.75 g cm$^{-3}$ and 56.1 ~ 63.1%, respectively.
The open porosity is proportional to the pore connectivity of geopolymers. The open porosity of geopolymers with SS, PAM and CTAB are 61.0%, 50.7% and 45.6%, increasing about 13%, 8% and 3%, respectively, when comparing with the control.

Roundness is defined as a value to describe the degree of particles or pore deviating from the sphere. The roundness of sphere is 1, and the value of roundness increases with the increases of the degree of particles or pore deviating from the sphere. The roundness of the porous geopolymer was obtained by the image analysis software Image-Pro Plus. As shown in table 1, the shapes of pores in geopolymers with SS and PAM are more regular and close to sphere, while the regularity of pores is reduced in the geopolymer with CTAB.

The addition of PAM and CTAB shows a strong influence on the average pore size in geopolymers, while a slight decrease is observed with the addition of SS. The average pore size of the control, SS, PAM, and CTAB are 18.9, 18.1, 27.4, and 11.7 μm, respectively.

Figure 5 shows the micrographs and pore size distribution of the porous geopolymer with different emulsifiers. Clearly, the spherical pores with the diameter of 5 ~ 40 μm are created by removing the template oil droplets. These pores are homogeneously dispersed in the geopolymer matrix, suggesting the well-distribution of template droplets in the geopolymer paste.

The influence of emulsifier types on porous geopolymers is mainly reflected in the morphology and diameter distribution of the pores. As figures 5(a), (b) shows, the shape of pores of the control group geopolymer is relatively regular, but the agglomerations phenomena and a few large pores are observed, the pore size distribution mainly in the range of 10 ~ 30 μm. With the addition of SS (figures 5(c), (d)), the shape of pores seems more regular and being more homogeneously distributed in the geopolymer matrix is observed. The average pore size is decreased, and the fraction at the range of 5 ~ 15 μm is increased compared to the control group. The shape of pores in of geopolymer with PAM is the most close to round, as shown in figures 5(e), (f), but with a relatively wide pore size distribution and a larger pore size. The average pore size is about 27.4 μm and almost 1/5 of pores are over 40 μm in diameter. Figures 5(g), (h) shows the sample with CTAB gives pores with a very fine in size and a narrow pore size distribution, while a fair amount of large air pore are also observed. The average pore size is about 11.7 μm, and over 70% of the total pores are in the ranges of 5 ~ 15 μm.

Table 1. Pore structures of geopolymers prepared by different emulsifiers.

| Emulsifier | Bulk density (g cm⁻³) | Total porosity (%) | Open porosity (%) | Roundness | Average pore size (μm) |
|------------|------------------------|--------------------|-------------------|-----------|-----------------------|
| Control    | 0.75                   | 56.0               | 42.7              | 1.39      | 18.9                  |
| SS         | 0.63                   | 63.1               | 61.0              | 1.25      | 18.1                  |
| PAM        | 0.69                   | 59.7               | 50.7              | 1.22      | 27.4                  |
| CTAB       | 0.75                   | 56.1               | 45.6              | 1.42      | 11.7                  |

Figure 4. Effect of emulsifier types on the compressive strength of geopolymer.
3.4. The mechanism of the effect of different emulsifiers on the paste viscosity and pore structure

In the oil/geopolymer composites paste, the aluminosilicate particles with certain hydrophilicity are able to stabilize the emulsion droplets via absorbed at the oil-water interface. The oil-in-water (o/w) Pickering emulsion stabilized by the solid particle can be formed in the composite paste, and the excess aluminosilicate particles are distributed in the continuous phase of the emulsion [20, 29].

Figure 5. SEM images (a), (c), (e), (g) and pore size distribution (b), (d), (f), (h) of porous geopolymer prepared by different emulsifiers: (a), (b) Control; (c), (d) SS; (e), (f) PAM; (g), (h) CTAB.
In Pickering emulsions, the viscosity of paste with a low proportion of oil phase (20 wt%) is mainly determined by the interactions between solid particles [30]. According to the DLVO theory for oxide suspensions [31], two kinds of forces exist between colloidal particles, van der Waals attractive forces and electrostatic repulsive force. Any changes in each type of force will have a direct influence on the interaction of aluminosilicate particles, and therefore on the viscosity of paste. In the paste with SS, the negative value of zeta potential of metakaolin particles are further decreased by the addition of anionic surfactant SS [32]. The electrostatic repulsive forces between particles are enhanced, thus reducing the aggregation of particles in the continuous phase and the viscosity of the geopolymer paste. On the other hand, when addition of CTAB with positive charge group in paste, the negative charge of the aluminosilicate particles would be neutralized via electrostatic interactions. The decrease of electrostatic repulsion among particles results in the significant increase of viscosity of geopolymer paste. With the addition of PAM, the polymer bridge can be formed between aluminosilicate particles by adsorbing of the PAM molecular. It is favor for the aggregation of aluminosilicate particles and probably the main reason for the increase of the viscosity of geopolymer paste.

The pore structure of geopolymer is mainly determined by the size of emulsion droplets. In the pastes with SS and CTAB, the adsorption of emulsifier molecule at the oil-water interface between the aluminosilicate particles and the oil droplet surface would decrease the oil-water interfacial tension and the required energy for emulsification, which results in the decreased size of emulsion droplets and the average pore sizes in the geopolymers. It is worth noting that compared to the anionic surfactant SS, the cationic surfactant CTAB shows a stronger ability to reduce the oil-water interface tension [33], resulting in a smaller average pore size. Unlike the SS or CTAB, the addition of PAM does not bring about significant changes to the interfacial tension, the pore size of PAM is closely related to the size of solid particles. The size of aluminosilicate particle is increased by the aggregation of particles, thus the droplet diameter of Pickering emulsion, which stabilized by aluminosilicate particle, is increased, the average pore size of PAM is also increased accordingly.

This difference in the morphology and dispersion of the pores can be explained by their different viscosities. The interface between the emulsion droplets is flowable at a lower paste viscosity and does not prevent the coalescence of droplet, which is favor for the formation of interconnected pores, and resulting in a high open porosity (table 1). On the contrary, the interface between the emulsion droplets is stiff at a high paste viscosity, which the coalescence of the droplets is limited and leads to a narrow pore size distribution, but show a low connectivity of pores. Moreover, the poor workability of geopolymer paste is caused by the high viscosity, difficult to squeeze out the air bubbles in forming process, resulting in a few large pores existed in geopolymer.

4. Conclusions

In this paper, the metakaolin-based porous geopolymer with a pore size of 10 ~ 50 μm are prepared using three different emulsifiers by emulsion templating method. The effect of different emulsifiers on the paste viscosity, mechanical strength and pore structure of geopolymer were studied. Results showed that the geopolymerization is not significantly affected by the addition and removal of emulsion template. The pore structure of geopolymer can be effectively modulated by use of emulsifiers with different ionic types. The ability to control the pore structure, e.g. average pore size, pore size distribution and connectivity, makes porous geopolymer potentially attractive for function applications as adsorbent, filtration membrane and catalyst carrier.

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Author contributions

Zhuangzhuang Wang designed and performed the experiments; Zhuangzhuang Wang and Duyou Lu wrote the paper.

Conflict of interest statement

The authors state that they have no conflicts of interest in this work.
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