Rheological properties of sewage sludge ash ceramic masses

S A Shakhov and N Yu Nikolaev

1Siberian Transport University, Dusi Kovalchuk St., 191, Novosibirsk, 630049, Russia

E-mail: nikolaevh13@mail.ru

Abstract. Industrial wastes are widely involved in the building ceramic production. Sewage sludge ashes are promising secondary sources for building ceramics production. However, sewage sludge ash application for building ceramic materials production is limited by unsatisfactory molding properties. According to modern concepts, coagulation structure formation processes can be controlled by adjusting ceramic mass compositions by highly dispersed modifying additives. In this study, the drinking water treatment sludge filtrate generated in pumping and filtering stations was used as a highly dispersed additive. The purpose of study was to assess the drinking water treatment sludge filtrate effect on rheological properties of ash-clay molding mixtures. Using the photo sedimentation analysis, X-ray phase analysis, calorimeter method and parallel-shifting plate plastometry the drinking water treatment sludge filtrate additive effect on rheological properties of ash-clay mixtures was established. It was found that ash-clay mixture modification with the drinking water treatment sludge filtrate promotes a slow elastic deformations percentage increase from 3-17% to 7-34%. Apparently, this is due to the drinking water treatment sludge filtrate highly dispersed particles adsorption on coarse ash and clay particles that promotes the growth of their hydrophilic properties. Ash-clay mixtures molding properties improvement makes it possible to increase the ceramic blanks density.

1. Introduction

Sewage sludge ash (SSA) raw materials application for building ceramic materials production is limited by unsatisfactory molding properties. Consequently, SSA is usually considered as a filler or partial substitute for clay, rather than main ceramic raw material [1].

Due to the poor cohesive and plasticity properties of SSA, water or lubricants additions was usually used to improve formability. Particularly, compressive force required to achieve the desired compaction level was higher for dry SSA mixes (60 MPa) compared to wet mixtures (40 MPa) [2, 3].

Effective control of dispersed systems rheology is possible not only by adding water or lubricants, but also by selecting mineral additives [4]. In particular, highly dispersed modifying additives with high surface energy can have a significant effect on the coagulation structure formation processes [5].

The drinking water treatment sludge filtrate generated in pumping and filtering stations can also be classified as highly dispersed modifying additive [6].

The purpose of study was to assess the drinking water treatment sludge filtrate effect on rheological properties of SSA-clay molding mixtures.
2. Materials and methods

Granulometric compositions of SSA and drinking water treatment sludge filtrate were determined by the photo sedimentation analysis method.

The infrared absorption spectrum for drinking water treatment sludge filtrate was obtained on IRAffinity-1S spectrometer.

Based on the rheological curves in the axes «absolute deformation (ε, mm) - shear time (τ, sec)» set, obtained on plastometer with a parallel-shifting plate, the absolute values of fast elastic, slow elastic and plastic deformations were determined [7].

The structural and mechanical types for SSA-clay mixtures were determined according to the fast elastic, slow elastic and plastic deformations ratio [8].

X-ray phase analysis of SSA-clay ceramic samples was performed on a Shimadzu XRD-7000 diffractometer equipped with a copper X-ray tube (radiation with a wavelength λ = 1.54178). SSA-clay ceramic samples were grind in agate mortar using heptane. Suspension samples were applied to a quartz cuvette.

Montmorillonite-hydromica clay from Kamenskoye deposit (Novosibirsk region) and SSA obtained by firing sewage sludge from Novosibirsk wastewater treatment plant at a temperature of 800 °C were the main components of the ceramic mixture. Ash is mainly represented by dusty fraction particles (5-50 µm). The SSA crystalline component includes quartz, calcite, and hematite phases (Figure 1).

![Figure 1](image_url)

**Figure 1.** Micrograph (a), granulometric composition (b) and X-ray phase analysis (c) of SSA.
The drinking water treatment sludge is close in chemical composition to clay (Table 1) and its crystalline component is represented by quartz and halloysite phases (Figure 2).

**Table 1.** Chemical composition of SSA, clay and drinking water treatment sludge.

| Component                        | Chemical composition, wt.% |
|----------------------------------|-----------------------------|
|                                 | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO |
| SSA                             | 53.2 | 20.1  | 11.1  | 8.8 | 3.2 |
| Clay                            | 67.6 | 15.3  | 4.7   | 5.8 | 1.3 |
| Drinking water treatment sludge | 59.8 | 4.6   | 17.8  | 8.1 | 13.1|

**Figure 2.** X-ray phase analysis of drinking water treatment sludge.

3. Results and discussions

The drinking water treatment sludge filtrate was obtained by passing through a filter separating the coarse particles (with diameter more than 50 μm). The filtrate photo sedimentation analysis results indicate the presence of particles with a diameter less than 1 μm (Figure 3, a). Apparently, the filtrate particles have a silicate nature, which indicated by the filtrate particles IR-spectrum. The Si-O bonds are characterized by bands with a 1200 cm⁻¹ and 1090 cm⁻¹ vibration frequency (Figure 3, b).

**Figure 3.** Granulometric composition (a) and IR-spectrum (b) of drinking water treatment sludge filtrate.
SSA-clay mixture modification with the drinking water treatment sludge filtrate promotes a slow elastic deformations percentage increase from 3-17% to 7-34% (Figure 4).

Figure 4. Slow elastic deformations percentage for SSA-clay molding mixtures without (a) and with the addition of 0.1% drinking water treatment sludge filtrate in dry matter (b).
Slow elastic deformations percentage increase contributes to the obtaining of SSA-clay mixtures falling into the fast elastic, slow elastic and plastic deformations optimal ratio zone (Figure 5).

**Figure 5.** Threefold deformations diagram for different composition of SSA-clay molding mixtures: 11 - 20% SSA : 80% clay; 12 - 20% SSA : 80% clay : 0.1% filtrate (in dry matter); 21 - 50% SSA : 50% clay; 22 - 50% SSA : 50% clay : 0.1% filtrate (in dry matter); 31 - 80% SSA : 20% clay; 32 - 80% SSA : 20% clay : 0.1% filtrate (in dry matter).

SSA-clay mixtures modification promotes the growth of their hydrophilic properties (Table 2). Apparently, this is due to the drinking water treatment sludge filtrate highly dispersed particles adsorption on coarse SSA and clay particles.

**Table 2.** Integral wetting heats ratio in polar (water) and non-polar (benzene) dispersion medium for SSA and clay particles.

| Dispersed phase | Dispersion medium | Integral wetting heats $Q_{\text{int}}, \text{J/g}$ | Integral wetting heats ratio in polar (water) and non-polar (benzene) dispersion medium |
|-----------------|-------------------|---------------------------------|--------------------------------------------------------------------------------------|
| Clay            | Water             | 9.62                            | 1.76                                                                                 |
| Clay            | Benzene           | 5.47                            |                                                                                     |
| Clay + 0.1% filtrate (in dry matter) | Water          | 21                              | 2.81                                                                                 |
| Clay + 0.1% filtrate (in dry matter) | Benzene    | 7.48                            |                                                                                     |
| SSA             | Water             | 6.98                            | 2.92                                                                                 |
| SSA             | Benzene           | 2.39                            |                                                                                     |
| SSA + 0.1% filtrate (in dry matter) | Water          | 1.32                            | 6                                                                                    |
| SSA + 0.1% filtrate (in dry matter) | Benzene    | 0.22                            |                                                                                     |
Wetting heats ratio growth for modified SSA and clay particles contributes to SSA-clay mixtures cohesive properties improvement, which makes it possible to increase the ceramic blanks density. In particular, at a compaction pressure of 15 MPa, SSA-clay mixture modification makes it possible to increase ceramic blanks density from 2.04 to 2.09 g/cm$^3$.

Figure 6. Ceramic blanks density for the compositions «50% SSA : 50% clay» (a) and «50% SSA : 50% clay : 0.1% filtrate (in dry matter)» (b).

SSA-clay mixtures molding properties improvement made it possible to obtain ceramic products (Table 3) with a reduced water absorption (from 7.79 to 7.04 g/cm$^3$), increased density (from 2.15 to 2.21 g/cm$^3$) and compressive strength (from 27.60 MPa to 37.15 MPa).

Table 3. Physical and mechanical properties of SSA-clay ceramic samples obtained by sintering at temperature 1100 °C for the compositions «50% SSA : 50% clay» (a) and «50% SSA : 50% clay : 0.1% filtrate (in dry matter)» (b).

| Composition | Water absorption, % | Density, g/cm$^3$ | Compressive strength, MPa |
|-------------|---------------------|-------------------|--------------------------|
| (a)         | 7.79±0.25           | 2.15±0.05         | 27.60±3.68               |
| (b)         | 7.04±0.05           | 2.21±0.001        | 37.15±4.86               |

4. Summary
It was found that SSA-clay mixture modification with the drinking water treatment sludge filtrate promotes a slow elastic deformations percentage increase from 3-17% to 7-34%. Apparently, this is due to the drinking water treatment sludge filtrate highly dispersed particles adsorption on coarse ash and clay particles that promotes the growth of their hydrophilic properties. SSA-clay mixtures molding properties improvement makes it possible to increase the ceramic blanks density, which to some extent may cause improvement of SSA-clay ceramic samples physical and mechanical properties.

5. References
[1] Ravindra K, Dhir OBE, Ciaran J L, Gurmel S G 2016 Sustainable Construction Materials: Sewage Sludge Ash (Woodhead Publishing Ltd) p 288
[2] Merino I, Arévalo L F, Romero F 2005 Characterization and possible uses of ashes form wastewater treatment plants Waste Management 25 1046–1054
[3] Merino I, Arévalo L F, Romero F 2007 Preparation and characterization of ceramic products by thermal treatment of sewage sludge ashes mixed with different additives Waste Management 27 1829–1844
[4] Shakhov S A, Klyuchnikova N S, Kozhemyachenko A S 2014 Composition and technological properties of sewage sludge and sewage sludge ashes University News. Construction 11(671) 103-113
[5] Zhenzhurist I A, Zaripova V M, Mubarakhina L F, Hozin V G 2010 The effect of silicon and aluminum oxides hydrosols nanosized particles on the structure formation of clay minerals in an aqueous medium Glass and Ceramics 7 28-32
[6] Shahov S A, Nikolaev N Yu, Rudaya T L 2018 Potential of drinking water treatment sludge as a raw material component of building ceramics Siberian Transport University Bulletin 1 61–67
[7] Kruglitsky N N 1977 Physicochemical mechanics foundations. Part 3 (High school) p 136
[8] Ilyin A P, Prokofiev V Yu 2004 Physicochemical mechanics in the catalysts and sorbents technology. Monograph (Ivanovo state University of Chemical Technology) p 316

Acknowledgments
The reported study was funded by RFBR, project number 20-33-90197.