Phase Composition of Sewage Sludge Ash Ceramics Modified by Drinking Water Treatment Sludge Filtrate

S. A. Shakhov and N. Yu. Nikolaev

Abstract Industrial wastes are widely involved in the building ceramic production. Sewage sludge ashes (SSA) are promising secondary sources for building ceramics production. According to modern concepts, formation processes of ceramic structure can be controlled by adjusting ceramic mass compositions by additives of nanosized particles. In this study, the filtrate of drinking water treatment sludge obtained at the water treatment facilities of Novosibirsk, which is a silicate sol with nanodispersed particles, was used as an additive. Purpose of the study was to assess the effect of silicate sol from drinking water treatment sludge on sewage sludge ash ceramics phase composition and microstructure. Using the thermal analysis, X-ray phase analysis and electron microscopy, the drinking water treatment sludge filtrate additive effect on SSA-clay samples phase composition and microstructure was established. It was found that ceramic obtained by sintering SSA with drinking water treatment sludge filtrate additive is characterized by a matrix microstructure.

Keywords Clay · Sewage sludge ash · Phase composition · Building ceramics · Anorthite · Matrix microstructure

1 Introduction

More and more industrial wastes are involved in the building ceramic production. Sewage sludge ashes (SSA) are promising secondary sources for building ceramics production.

SSA properties were studied in a number of works [1–3], but the results were not widely used in building ceramic industry, because SSA ceramic products not always have the necessary strength characteristics. It is due to the SSA chemical composition, which affects to the phase composition formation of ceramic.

Ceramics phase composition can be controlled by adjusting the ceramic mass composition using special additives. The most suitable modifiers for clay minerals

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structures are nanosized oxides of silicon and aluminum, which are realized in hydrosols [4].

Earlier in [5], it was shown that the drinking water treatment sludge filtrate, obtained after high-speed filters in the Novosibirsk water utility filtering station, is a sol of a silicate composition with a nanoscale particle size.

The purpose of study was to assess the drinking water treatment sludge filtrate effect on the SSA-clay ceramics phase composition and structure formation.

2 Materials and Methods of Research

Kamenskoye deposit (Novosibirsk region) montmorillonite-hydromica clay was used in this study.

Sewage sludge was obtained from Novosibirsk wastewater treatment plant (map No. 39). The sludge was burned in laboratory furnace at temperatures of 850 °C. Drinking water treatment sludge filtrate, obtained after high-speed filters in the Novosibirsk water utility filtering station and separated from the coarse fraction, was used as a modifying additive in the study.

Ceramic mixtures compositions are presented in Table 1.

Weighed portions of the mixtures with different additives amounts were mixed with water or drinking water treatment sludge filtrate. Samples were dried at 110 °C. Then the SSA-clay samples were sintered at 1100 °C with holding at a maximum temperature for 1 h.

Differential thermal analysis (DTA) was performed to identify the phase formation features of the SSA-clay samples. X-ray phase analysis (XPA), which was performed on a Bruker D8 Advance diffractometer using Cu-Kα radiation, was used to determine the phase composition of SSA-clay ceramic samples. The PDF2 database with the Search-Match shell was used to identify the obtained diffraction patterns.

Element maps and microstructure images of SSA-clay ceramic samples were obtained using Hitachi TM 3000 electron microscope with an energy dispersive analyzer.

Table 1 Ceramic mixtures compositions

Compositions №	Content of components (%)		
	SSA (%)	Clay (%)	Drinking water treatment sludge filtrate (%) (in terms of dry matter; over 100%)
1	50	50	0
2	50	50	0.05
3	50	50	0.1
4	50	50	0.25

3 Results and Discussion

The endothermic effect at 50–150 °C temperature range is due to the sorbed water loss by clay minerals. In this case, there is a loss of mass—4.5%. The weight loss occurs at a much lower rate with further mixture heating. Heat absorption is caused by the release of chemically bound water by clay minerals in the 300–500 °C range. Kaolinite is initially dehydrate, turning into metakaolinite, and then decompose into oxides at 750–900 °C [6] (Fig. 1).

The endothermic effect at 700 °C is associated with the montmorillonite crystal structure destruction. According [7], calcium carbonate dissociates in the 770–1010 °C temperature range.

Exothermic effects on the heating curve are due to new crystalline phases formation. The total weight loss for the test sample was 10.8%.

According to the XPA data (Fig. 2), for all SSA-clay samples after sintering at 1100 °C, the main identified phases are anorthite (3.10; 4.05 A0), quartz (3.35 A0) and hematite (2.7 A0).

The anorthite ($CaO \cdot Al_2O_3 \cdot SiO_2$) formation is due to solid-phase reaction between amorphous γAl_2O_3 formed in SSA from aluminum oxychloride and calcium silicate.

Another possible anorthite formation mechanism may be associated with the reaction between amorphous silica and calcium oxide. According study [8], kaolinite dehydration calcium carbonate dissociation are promoted by the anorthite crystallization in clay. The reaction equation has the form (Eq. 1):

$$Al_2O_3 \cdot 2SiO_2 + CaCO_3 = CaO \cdot Al_2O_3 \cdot 2SiO_2 + CO_2 \quad (1)$$

The phase composition results (Table 2) indicate to increase anorthite content with adding drinking water treatment sludge filtrate.

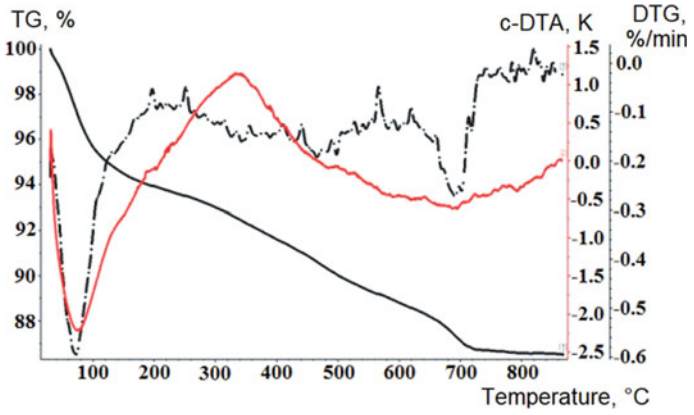

Fig. 1 DTA results (composition № 4)

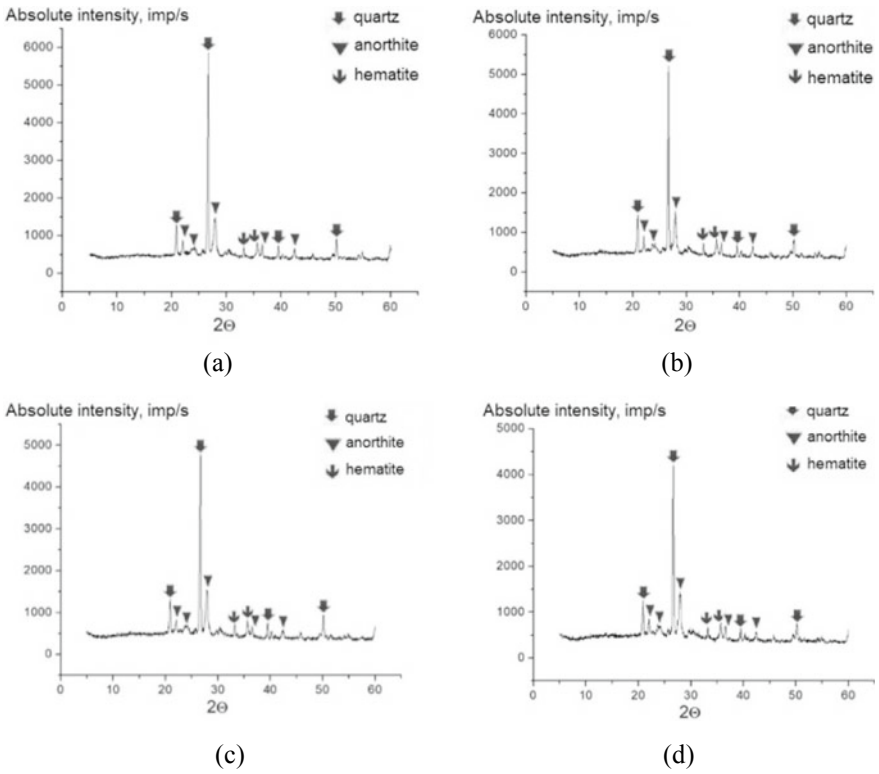

Fig. 2 XPA diffraction patterns of ceramic samples: **a**—50% SSA: 50% clay: 0% drinking water treatment sludge filtrate; **b**—50% SSA: 50% clay: 0.05% drinking water treatment sludge filtrate; **c**—50% SSA: 50% clay: 0.1% drinking water treatment sludge filtrate; **d**—50% SSA: 50% clay: 0.25% drinking water treatment sludge filtrate

Table 2 SSA-clay ceramic samples quantitative phase composition

Sample	Content of components (%)
1	64% anorthite $CaAl_2Si_2O_8$ + 34% quartz SiO_2 + 2% hematite Fe_2O_3
2	67% anorthite $CaAl_2Si_2O_8$ + 31% quartz SiO_2 + 2% hematite Fe_2O_3
3	69% anorthite $CaAl_2Si_2O_8$ + 29% quartz SiO_2 + 2% hematite Fe_2O_3
4	68% anorthite $CaAl_2Si_2O_8$ + 29% quartz SiO_2 + 3% hematite Fe_2O_3

The microstructure of all samples is characterized by uniform pores and crystalline quartz compounds distribution. At the same time, SSA-clay samples with drinking water treatment sludge filtrate addition are still noticeably different in relative position of crystalline and amorphous phases.

The SSA-clay with drinking water treatment sludge filtrate additive samples microstructure feature is matrix microstructure. The spatially arranged matrix

microstructure has phase composition differences between filler and binder, which confirmed by the electron microscopy data (Fig. 3d, f). Matrix formed from clay particles is a "binder". "Filler" formed from quartz grains contained in sewage sludge ash. It is important to note that matrix include amorphous and crystalline anorthite phase. Anorthite crystals reinforce matrix SSA-clay samples microstructure, which provides higher strength and frost resistance.

SSA-clay samples with drinking water treatment sludge filtrate additive are characterized by increased density from 2.15 to 2.21 g/cm^3 and compressive strength from 27.60 to 37.15 MPa (Table 3), which may be due to their matrix microstructure and increased anorthite phase content (from 64 to 68%).

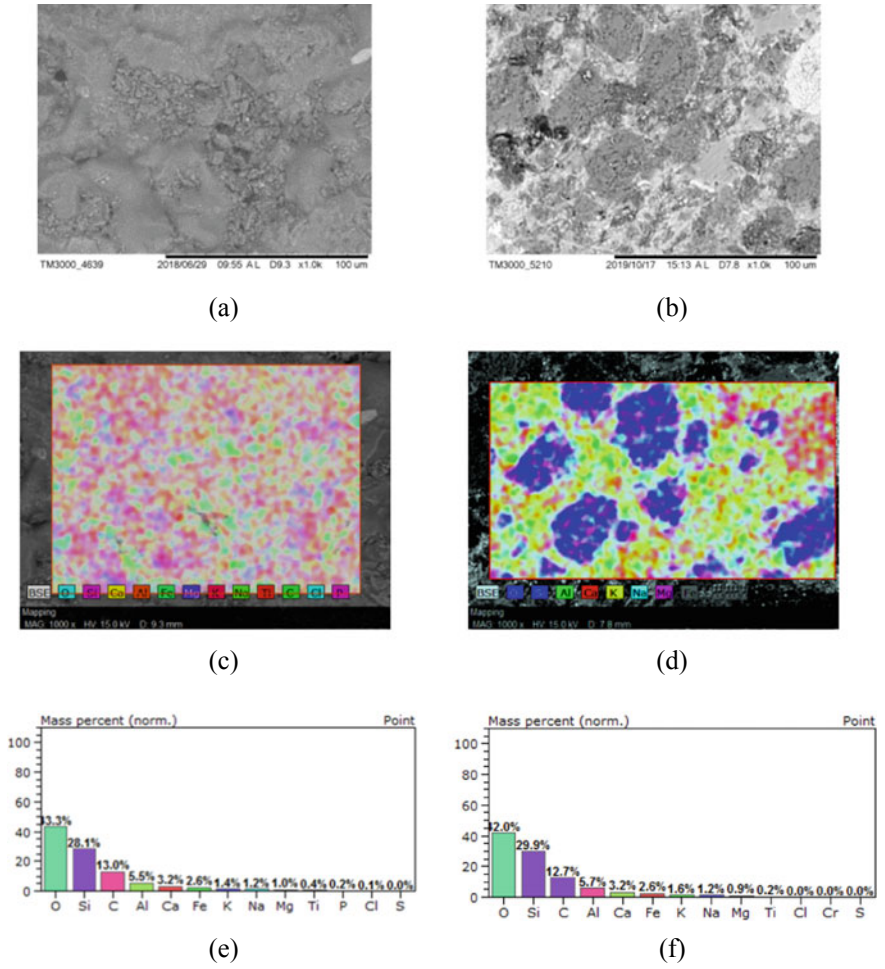

Fig. 3 Microstructure and elemental composition of SSA-clay ceramic samples: **a**, **c**, **e**—composition 1, **b**, **d**, **f**—composition 4

Table 3 SSA-clay ceramic samples water absorption (W, %), density (ρ, g/cm^3), compressive strength (R, MPa)

Compositions №	Water absorption (%)	Density (g/cm^3)	R (MPa)
1	7.79 ± 0.25	2.15 ± 0.05	27.60 ± 3.68
4	7.04 ± 0.05	2.21 ± 0.01	37.15 ± 4.86

4 Conclusions

DTA, XPA and electron microscopy results allow us to conclude that:

1. SSA-clay samples phase composition formation is determined by calcium ions diffusion into the metakaolinite structure and anorthite crystallization from melt enriched with aluminum, calcium and silicon oxides.
2. SSA-clay samples with drinking water treatment sludge filtrate additive are characterized by increased density and compressive strength, which may be due to their matrix microstructure and increased anorthite phase content.

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