Utilisation of drinking water treatment sludge for the manufacturing of ceramic products

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Abstract. The influence of the additive of drinking water treatment sludge on the physical and mechanical properties, structural parameters, microstructure of the ceramic products is analysed in the research. Drinking water treatment sludge is renewable, environmentally-friendly, economical additive saving expensive natural raw materials when introduced into the ceramic products. The main drinking water treatment sludge component is amorphous Fe₂O₃ (70%). Formation masses are prepared by incorporating from 5 % to 60 % of drinking water treatment additive and by burning out at the temperature 1000 °C. Investigation showed that the physical and mechanical properties, microstructure of the ceramic bodies vary depending on the amount of drinking water treatment additive incorporated. In addition, drinking water treatment additive affects the ceramic body as a pigment that dyes the ceramic body in darker red colour.

Keywords: drinking water treatment sludge, amorphous Fe₂O₃, ceramic body, density, strength, water absorption.

1. Introduction

The great worldwide attention is paid to management, utilization and reuse of waste. The utilization of waste for the production preserves traditional raw materials. Hereby, less energy is consumed, and the environment is less polluted by hardly degradable materials. However, global and regional management of waste is different, it depends on the economic development level of countries, urbanization level of regions as well as production capacity.

Currently, the greatest part of Lithuanian industrial and household waste is landfilled. However, such waste may be recycled and reused for the production of products. Many technogenic waste can be used for the production of building materials and products. The application process of secondary waste from economic and technological prospects requires additional and specific scientific researches. The use of such waste may be related to its peculiarity and introduction to forming masses specificity.

Different authors have investigated the incorporation of different types of waste in the production of traditional ceramic materials [1-5]. Although the industry of building materials is highly promising for the final disposal of solid waste, there is a lack of information about recycling of drinking water treatment sludge in clay ceramics.

The characteristics of drinking water treatment sludge depend on the origin of the drinking water treatment method. The main chemical components of drinking water treatment sludge are SiO₂, Fe₂O₃, CaO, MgO. Authors [6-9] have analysed the possibility of water treatment sludge to be utilised for the production of ceramic products. It is as well-known that iron can be used as a decent color pigment [10].

The main objective of the present study is to investigate the feasibility of drinking water treatment sludge to be used for the production of clay bricks. The impact of the amount of drinking water treatment sludge in the clay mixture is discussed in terms of physical–mechanical properties, porosity and microstructure.
2. Materials and methods

Mixture of the clay, sand and drinking water treatment sludge is used in the research. Raw materials and additive are dried at 100-105 °C temperature, ground and sieved out through 0.63 mm sieve. Initially, dry mixture of the components is mixed, then mixture is watered till the humidity reached the level suitable for the formation. The amount of water required for formation masses varies from 20% to 40%. The higher amount of drinking water treatment sludge is introduced into formation masses, the higher amount of water is required. The mixing of forming masses is performed manually. This formation mass is kept for three days at (95–5)% humidity, in order to evenly distribute humidity in the formation mass. After three days of hardening, 70×70×70 mm samples are formed from formation masses.Specimens which have been exposed at normal conditions, are dried in the drying oven: 60±5°C temperature during the first day, next day at 105±5°C temperature. Burning of the samples is carried out at 1000°C temperature, overall burning period is 34 h, by keeping at the highest burning temperature for 4 hours. Composition of the formation masses is presented in Table 1.

| Raw materials                          | Composition of formation masses (% of the mass) |
|----------------------------------------|-----------------------------------------------|
| Clay + Sand                            | 100  95  90  80  60  40  60                    |
| Drinking water treatment sludge        | –     5  10  20  40  60                       |

Chemical analysis of brick raw materials is made by using X-Ray Fluorescence Spectrometer (XRF). Physical and mechanical characteristics of the ceramic products are determined through the implementation of the standard methodologies: density - according to LST EN 772-13:2003, water absorption - according to LST EN 771-21:2011, compressive strength - according to LST EN 772-1:2011 [11–13]. Linear shrinkage is measured using a calliper after drying and burning steps. Total open porosity is determined in accordance with the methodology [14]. Shrinkage is calculated by employing 1 equation.

\[ L = \frac{L_0 - L_1}{L_0} \cdot 100\% \]

where: \(L_0\) – distance between indentations on the formed sample, mm; \(L_1\) – distance between indentations on the dry or burnt sample, mm.

3. Results and discussion

The mixture of clay and sand consists of (60–65)% of clay and (35–40)% of sand. The clay is illite with small content of carbonates. Considering the chemical composition, clay+sand mixture belongs to the group of the acid and semi-acid carbonaceous mixtures because its contaminated with the coarse carbonaceous insertions (≤3.0)%, \(\text{Al}_2\text{O}_3\) in the mixture reaches 17.09%. This mixture belongs to the group of the mixtures with low amount of dyeing oxides because it contains 6.56% of \(\text{Fe}_2\text{O}_3\).

Chemical analysis of drinking water treatment sludge shows that its main component is \(\text{Fe}_2\text{O}_3\) with small amounts of \(\text{SiO}_2, \text{P}_2\text{O}_5, \text{CaO}\) and other oxides which do not exceed 1.5% (Table 2). Microscopic investigations of drinking water treatment sludge show that sludge consists of various sizes spherical shaped particles which are coupled into conglomerates. Density of these conglomerates is not high,
there is a large amount of hollow areas among the coupled particles (Figure 1 a) and b)). Continuous conglomerates are crushed during the grinding of drinking water treatment sludge, their particles form structures with the size of 138 µm and smaller. Surface of these structures is grained and formed from a lot of external, open, fine, interconnected pores. pH of drinking water treatment sludge additive is identified to be 7.81, it forms slightly alkaline medium. It is determined that the bulk density of DWTS particles is 547 kg/m³, particles' density – 2270 kg/m³, specific surface area – 9657 cm²/g.

After the differential thermal analysis of drinking water treatment sludge additive, it is determined that the first endothermic effect occurs at temperature range of (40–200)°C. Maximum value of this effect is found at 107°C temperature and it is related to the extraction of free hygroscopic water from drinking water treatment sludge. Mass losses during this dehydration process reach 8%. Further endothermic effect is followed by exothermic effect. Exothermic effect occurs at the temperature range of (200–463)°C, the maximum of this effect is observed at 290 °C temperature. During this effect, the mass of the sample decreases evenly. It is assumed that this exothermic effect is related to the burning out of the organic materials (iron bacterium) from drinking water treatment sludge. In addition, the crystallisation process occurs. i.e. amorphous phase (ferrihydrite) is transformed into crystal phase – hematite. Mass losses at 463°C temperature reach 20.5%. When the temperature is further increased up to 1000°C, mass losses increase up to 22.5%.

### Table 2. Chemical composition of raw materials

| Raw materials                              | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | K₂O | SO₃ | P₂O₅ |
|--------------------------------------------|------|-------|-------|-----|-----|-----|-----|------|
| Clay + Sand                                | 67.52| 17.09 | 6.56  | 2.14| 1.88| 4.23| 0.58| –    |
| Drinking water treatment sludge            | 10.90| 1.34  | 68.65 | 8.23| 0.61| –   | 0.88| 9.39 |

**Figure 1.** Microstructure of the drinking water treatment sludge

It is known from the analysis of the drying processes of ceramic semimanufactures that water mechanically admixed to the formed semimanufacture forms a net of branchy capillaries with the open ends reaching the surface. During the drying of the semimanufacture, moisture starts to evaporate from the surface, and concentration of humidity in the inner layers increases Therefore, humidity constantly moves from the inner layers towards the surface.

The volume decreases of the analysed samples containing drinking water treatment sludge additive and after the drying it varies from 7.2% to 10.0%. The decrease is related to the dried colloidal
particles of the clay, and especially the ones of porous drinking water treatment sludge which absorb water from the atmosphere and during the formation, thus increasing the volume. After the drying, when water evaporates it decreases. Total volume decrease after the drying and burning at 1000°C temperature varies (depending on the composition of the formation mass) from 11.5% to 15.2%. The change of volume after the burning is related to the fact that, during the fragmentation of the components of clay and drinking water treatment sludge additive, organic burning materials are removed from the formation masses.

![Figure 2. The linear shrinking after drying and burning](image)

The density and water absorption of the bricks produced with drinking water treatment sludge are shown in Figure 3. The density for the clay brick made with drinking water treatment sludge is within the range of 1140-1807 kg/m³. The density of the ceramic body without drinking water treatment sludge is 1714 kg/m³. When 5 % of drinking water treatment sludge additive is added to the formation mass and it is burned at the selected burning temperatures, density increases and reaches 1807 kg/m³.

When up to 10 % of drinking water treatment sludge additive is added to the formation masses, water absorption is lower than the one of the ceramic bodies without drinking water treatment sludge additive. The amount of absorbed water is usually determined by pores’ volume of ceramic products, dimensions and arrangement of the pores. When more than 10 % of drinking water treatment sludge additive is added, the ratio of water absorption increases.

Compressive strength is a very important parameter which is used to meet engineering quality in construction material applications. Therefore, compressive strength and total open porosity of all brick samples are determined and mean values are given in Figure 4. During the burning, light, porous drinking water treatment sludge additive and its burning out components form additional pores in the ceramic body. Due to these pores, total open porosity as well as water absorption of the ceramic bodies increase. When the maximum amount of 60 % of drinking water treatment sludge additive is utilised, compressive strength and total open porosity after the burning at 1000°C temperature reaches 2.5 MPa and 62 %, respectively. Even though the compressive strength of ceramic bodies is low, it is close to the compressive strength of aerated concrete products, and it is 1.5 MPa.

The results show that drinking water treatment sludge is light, sufficiently fine burning additive (mass loss after burning at 1000°C temperature is 22.5%). 5% of this additive evenly distribute through the whole ceramic body, partially fill open pores, therefore, the content and volume of open pores reduce. The amount of organic matter existent in ceramic body due to 5% of an additive is low. It is assumed that it might be the reason of the increase in density and compressive strength, reduction in
water absorption and open porosity. The addition of 10% and more, increases the amount of burning organic matter, therefore, fine particles of drinking water treatment sludge agglomerate, thus reducing the mentioned physical-mechanical properties and increasing porosity.

![Figure 3. Density and water absorption of the brick samples](image)

**Figure 3.** Density and water absorption of the brick samples

![Figure 4. Compressive strength and total open porosity of the brick samples](image)

**Figure 4.** Compressive strength and total open porosity of the brick samples

SEM investigations are conducted in order to get a better understanding of the morphology of the microstructure. Microstructure of the ceramic body without additive (Figure 5 a and c) burned at 1000°C temperature shows that the microstructure is sufficiently dense. There are few small spherical and oblonged pores. Most of them have closed structure, and the porosity of such ceramic body is low. Microstructure of the ceramic body with 60% of additive (Figure 5 b and d) burned at 1000°C is different. The microstructure is distinguished by the porous structure. A characteristic feature of the ceramic body is high density of pores. Pores are hollow, interconnected, having different size and shapes.
It is well known that iron from drinking water treatment sludge is a decent colour pigment. The colour characteristics of drinking water treatment sludge are extensively exploited in clay brick manufacturing. The colours of the clay bricks produced with drinking water treatment sludge are shown in Figure 6. It is identified that when not less than 5% of water treatment sludge is introduced into the formation mass, and it is burned at 1000°C temperature and kept at the highest burning temperature for 4 h, the colour of the ceramic body becomes darker.
Figure 6. Colour of the ceramic bodies: a) ceramic body without drinking water treatment sludge additive; b) ceramic body with 5% of drinking water treatment sludge additive.

Conclusions

In this study, the properties of clay bricks with drinking water treatment sludge are investigated. It is found that the additive of drinking water treatment sludge is effective additive which influences physical and mechanical properties as well as structural parameters of the ceramic body:

- after the addition of 5% of drinking water treatment sludge additive into clay mixture and after burning samples at 1000°C temperature, the ceramic body with the following parameters is obtained: density – 1807 kg/m³, compressive strength – 48.5 MPa, linear shrinkage – 11.5% (after burning), water absorption 11.0%, total open porosity 27.6%.;
- after the addition of 60% of drinking water treatment sludge additive into clay mixture and after burning samples at 1000°C, the ceramic body with the following parameters is obtained: density – 1140 kg/m³, compressive strength – 2.5 MPa, linear shrinkage – 15.2% (after burning), water absorption – 28%, total open porosity – 62%.

Investigations confirm that water treatment sludge containing dyeing Fe₂O₃ oxides is an intensive dyeing additive for ceramic body.

References

[1] Uslu T, Arol A I 2004 Waste Manage 24(2) 217–220
[2] Dondi M, Ercolani G, Guarini G, Raimondo M 2002 J. Eur Ceram Soc 22 1729–1735
[3] Barbieri L, Andreola F, Lancellotti I, Taurino 2013 Waste Manage 33(11) 2307–2315
[4] Cusidó J A, Cremaides L V, Soriano C, Devant M 2015 Applied Clay Science 108 191–198
[5] Dondi M, Marsigli M, Fabbri B 1997 Tile & Brick Int 13 218–315
[6] Anderson M, Skerratt R G 2003 Brit Ceram Trans 102(3) 109–113
[7] Ferreira J M F, Olhero S M, 2002 J Eur Ceram Soc 22 2243–2249
[8] Monteiro S N, Alexandre J, Margem J I, Sanchez R, Vieira C M F 2008 Constr Build Mater 22(6) 1281–1287
[9] Onaka T 2000 Water Science Techn 41 93–98
[10] Wayne E Brownell 1957 J Amer Ceram Soc 40(6) 179–187
[11] LST EN 772–13:2003 Methods of test for masonry units - Part 13: Determination of net and gross dry density of masonry units (except for natural stone)
[12] EN 772–21:2011 Methods of test for masonry units. Determination of water absorption of clay and calcium silicate masonry units by cold water absorption
[13] LST EN 772–1:2003 Methods of test for masonry units – Part 1: Determination of compressive strength
[14] Kizinievič O, Balkevičius V, Pranckevičienė J, Kizinievič V 2015 Ceram Inter 41 (9) 11234-11241