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Analysis of the effect of paper sludge on the properties, microstructure and frost resistance of clay bricks

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HIGHLIGHTS

- Paper sludge (PS) mainly consists of cellulose and calcite.
- Cellulose fibre (CF) reduce the shrinkage of clay bricks.
- CF and $CaCO_3$ decomposition products change the microstructure of the clay body.
- The optimal amount of PS additive in a clay brick is up to 15%.

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ABSTRACT

The paper analyses the effect of waste sludge from paper industry (paper sludge) on physical and mechanical properties of clay bricks, their microstructure and resistance to freezing and thawing. Paper sludge is not hazardous industrial waste mainly consisting of cellulose and calcite. Clay bodies were made from the mix containing from 5% to 20% of paper sludge and fired at 900 °C and 1000 °C temperatures. Open pore structure in the clay body is developed as a result of burnt cellulose fibres and calcite decarbonization. Physical and mechanical properties of clay bodies change depending on the content of added PS: shrinkage, density and compressive strength reduce, water absorption and effective porosity increase. It is recommended to add 5% of paper sludge to the clay body and fire it at 900 °C temperature, or alternatively add 5–15% of paper sludge and fire the clay body at 1000 °C temperature. In terms of resistance to freezing and thawing, such products are regarded as frost resistant in moderately aggressive environment, i.e. in structures protected from direct environmental effects.

1. Introduction

Directive 2008/98/EC on waste provides to avoid waste generation and to use waste as a resource. Attempts should be made to recycle, re-use and utilize waste. Paper manufacturing is a complex industry involving multiple processes where different products are produced and large quantities of waste of primary, biological or de-inking origin are generated, waste water treatment sludge, primary sludge, and secondary sludge among them [1]. The primary sludge is generated in the largest quantities [2]. The production of 1 ton of paper generates about 30 kg of primary sludge, as it is mentioned elsewhere [3]. Nowadays this sludge is used for energy generation through incineration [4–7]. Sludge incineration process is regulated in the EU and typically uses fluidised bed combustion from 850 °C to 1100 °C. The incineration process, however, generates ash, which must be utilised as well. Researchers [8,9] found that paper sludge ash can be utilised in concrete manufacturing, where part of cement is replaced by hydrophobic paper sludge ash additive. It is stated that 12% of paper sludge ash added to the concrete mix significantly reduces water absorption of concrete without compromising on either density or compressive strength [8].

Authors [10–18] argue that paper sludge fired at 700–750 °C temperature acquires pozzolanic properties. Such substance can be utilised in cement manufacturing because under high temperature conditions kaolin present in paper sludge turns into metakaolin, which has the properties similar to those of commercial metakaolin [14]. Besides, the resulting ash retains more regular (chemical composition) properties compared to the bottom ash from the incineration of municipal waste.

It is also known that paper sludge can be used in concrete and cement brick manufacturing [19,20] as a substitute for certain natural additives such as lightweight aggregates [21].

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Other authors suggest producing wood-paper sludge boards where a certain amount of wood chips is replaced with paper sludge waste. Unfortunately, such boards have a much lower compressive strength due to weak bonding between wood chip and paper sludge particles. The optimal content of paper sludge in such boards is up to 10% [22].

Paper sludge can be also used in clay brick manufacturing [21,23–28]. On the one hand, paper sludge additive reduces the density and thermal conductivity of construction products; on the other hand it impairs their mechanical properties. Authors [28,29] propose to modify clay and paper sludge mix with glass scraps. Such modification improves mechanical properties of construction products, reduces the number of pores, intensifies the sintering, and changes the mineral composition of the final products.

In spite of numerous papers analysing this issue, there is no consensus on the appropriate amounts of paper sludge additive and firing temperatures. Besides, no information on the effect of this additive on the durability of clay bricks in terms of frost resistance was found in research literature.

Lithuanian paper mills generate about 12–13 thousand tons of primary paper sludge per year. The average humidity of this sludge is approx. 60%. The sludge contains 40–50% of organic matter (paper fibre) and 50–60% of mineral matter. Paper sludge properties and composition remain constant. At present paper sludge is not utilised in Lithuania. Instead, it is accumulated in the territory of the paper mill and afterwards removed to the landfill. Researchers have to find new and cheap utilisation techniques in order to solve the problem of waste accumulation in large quantities and to meet environmental requirements. Paper sludge utilisation in ceramic manufacturing industry could be a prospective and economically viable solution. This technique would protect the environment and support the production of ecological ceramic products. The main objective of this study is to examine the effects of paper sludge (primary sludge) additive on physical and mechanical properties, porosity, microstructure, and frost resistance of fired clay bricks. This is the first study in Lithuania on the utilisation of paper sludge in fired clay bricks.

2. Materials and methods

Clay, sand and paper were used in this experimental research. The clay was dried at 105 ± 5 °C temperature, crushed and sieved through 0.63 mm sieve. The sand was dried and sieved through 2.5 mm sieve. The humidity of paper sludge was 50–60%. Paper sludge was dried at 75 ± 5 °C temperature, ground and sieved through 2.5 mm sieve (Fig. 1). Paper sludge contains 40–50% of organic paper fibre and 50–60% of inorganic matter. The pH of the paper sludge was 8 ± 0.2, bulk density was 410 kg/m^3.

Fig. 1. Dried paper sludge (left) sieved through 2.5 mm sieve (right).

Initially, the components were mixed under dry conditions; then, the mixture was watered until the humidity level suitable for brick forming (e.g., 22–29%). The tests revealed that higher content of paper sludge additive increased water demand required to obtain the moulding compound of adequate plasticity. The reason is high water absorption of organic cellulose fibres. The plasticity index of the moulding compound reduces with the addition of paper mill sludge (Table 1). The plasticity of the clay was determined by the standard Vasiliev cone testing method. 60 × 30 × 18 mm, 30 × 54 mm and 70 × 70 × 70 specimens were formed from the moulding compound. 70 × 70 × 70 mm size specimens are used in frost resistance tests, 50 × 50 × 50 mm size specimens are used to test the mechanical characteristics, and 60 × 30 × 18 mm size specimens are used to test the physical characteristics. The compositions of the moulding compounds are shown in Table 1. The samples were dried at 105 ± 5 °C until stable masses were reached. The dried samples were then fired at 900 °C and 1000 °C for 1 h.

Classical chemical analysis was used to determine the chemical composition of primary silicate raw materials used for the tests by means of Si(Li) detector INCA PentaFET × 3 from Oxford Instruments. Physical and mechanical properties of the fired specimens were determined by following the standard testing methodologies: density was determined in accordance with the standard LST EN 772-13:2003 [30]; water absorption was determined in accordance with the standard LST EN 772-21:2011 [31]; the compressive strength was determined in accordance with the standard LST EN 772-1:2011 [32]; loss on ignition was determined in accordance with the standard LST EN 15935:2012 [33], and bulk density was determined in accordance with the standard LST EN 13041:2012 [34]. The durability of the specimens was evaluated as freeze and thaw resistance in accordance with the standard LST 1985:2006 [35].

The drying shrinkage and firing shrinkage was calculated from equations 1 and 2 below.

$$L = \frac{L_0 - L_1}{L_0} * 100\% \tag{1}$$

$$L_B = \frac{L_0 - L_2}{L_0} * 100\% \tag{2}$$

where L_0 is the distance between two reference marks in the wet specimen, mm; L_1 is the distance between two reference marks in the dried specimen, mm; L_2 is the distance between two reference marks in the fired specimen, mm.

The granulometric composition was determined by means of liquid analyser Cilas 1090. A dilatometer Linseis L76 was used for dilatometric analysis at 4 °C/min. A dilatometer Linseis PT-1600 was used for dilatometric analysis at 10 °C/min. X-ray analysis was performed using a diffractometer DRON-7 X-ray (Co anode and Fe filter). The microstructure was analysed by means of SEM Quanta 250 with SE detector. Differential thermal analysis was done by means of derivatograph Q 1500D. The content of heavy metals was determined by Atomic Absorption Spectroscopy technique using the spectrophotometer Buck Scientific 2010 VGP with air-acetylene flame.

The two-sided confidence intervals, with the confidence level of 0.95, of means of the measured quantities (porosities, shrinkage, density, water absorption, strength) were calculated by assuming that the porosities, shrinkage, density, water absorption and strength are normally distributed random variables. The Student distribution was applied to calculate the confidence intervals $(m - td,(1-\alpha/2)\cdot s/\sqrt{n}) \leq \mu \leq (m + td,(1-\alpha/2)\cdot s/\sqrt{n})$ of the means, where m and s are estimations of the mean and standard deviation of the random variables, n = 6 is the sample size, $td,(1-\alpha/2) \approx 2.57$ is $(1-\alpha/2) = 0.975$ quantile of the Student distribution, where