(wileyonlinelibrary.com) DOI 10.1002/jctb.7174

Paper industry slag for the production of building ceramics

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Abstract

Background: Re-use of wastes as a part of sustainable development is one of the most important aspects of a healthy human life and environmental protection. This study examines the viability of using paper industry slag for the production of building ceramics.

Results: Paper industry slag was used in the production of ceramic bricks as a burnable additive in amounts of 5, 10 and 15 wt%. The slag was introduced without and after three different preparatory operations: dried, dried and ground, granulated and dried. Clay was used as a raw material and quartz sand as a lean additive. The samples were made by plastic molding. Drying was carried out at a temperature of 100 °C. To determine the optimal heat treatment temperature, firing was carried out at 900, 950, 1000 and 1050 °C with holding at the maximum temperature for 30 min, followed by determination of the properties of the samples.

Conclusions: It has been established that the samples obtained using the slag have good physical and mechanical properties and comply with current standards. At the same time, the use of slag in the production of ceramic bricks makes it possible to solve the problem of waste management, as well as to obtain a secondary material resource from the slag – a burnable additive, which leads to savings in raw materials and energy resources in the production of ceramic bricks.

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Keywords: ceramic brick; paper mill slag; compressive strength; water absorption; waste recycling

INTRODUCTION

Currently, all over the world, there is an acute problem of waste management. Production and consumption wastes are serious sources of environmental pollution. Many wastes are not used in industrial production, although they have the properties of secondary material resources. They are sent to landfills for disposal, which leads to the alienation of land, and creates a risk of pollutants entering the environment. Waste recycling is one of the most important challenges for green chemistry, circular economies and sustainable develop-ment.^{[1,2](#page-7-0)} Wastes are often used in the production of materials for various purposes: modified materials for water treatment,³⁻⁵ wastewater treatment, 6.7 recovery of metals, 8 pigments 9.10 as well as building materials, such as gypsum, $11-13$ anhydrite, $14,15$ binders, 16 bricks, $17,18$ composites.¹⁹⁻²² etc.

One of the production wastes that is generated at cardboard and paper enterprises is osprey (slag), which is sewage sludge formed at wastewater treatment facilities. Industrial wastewater at cardboard and paper enterprises is formed at the stages of forming and pressing the paper canvas. Then it is collected in recycled water collectors, where part of the water is returned to the technological process, and the excess is fed to treatment, which results in the formation of osprey (slag). The scheme of wastewater disposal of cardboard and paper production is considered using the example of one existing cardboard and paper plant and is shown in Fig. [1.](#page-1-0) Excess wastewater enters into flotators, where the fibers are captured, which, after the pulper, returns

to the technological process. The clarified water is sent to the collector and is partially used for spraying screen cylinders. Excess clarified water from the clarified water pool, as well as wastewater generated during emergency shutdowns of machines, scheduled preventive repairs and washing of technological equipment are mixed at the pumping station, after which they enter the radial settling tanks, where cleaning of suspended particles takes place. In the process of water purification, sewage sludge (osprey) is formed, which is dehydrated in a filter press.

Osprey (slag) samples from several enterprises of the cardboard and paper industry were taken for research. Despite the fact that

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Figure 1. Scheme of wastewater disposal of a cardboard and paper plant.

all enterprises dehydrate the osprey, its final humidity is high, ranging from 63% to 75%. Dehydration is usually carried out in several stages: at the first stage, sludge thickeners are used, which allow, at the lowest cost, a reduction in the volume of sludge by several times. For deeper dehydration, centrifuges, vacuum filters, filter presses and other equipment is used, which makes it possible to obtain a product with a final moisture content of 60–80%. To further reduce humidity, thermal methods can be used, carried out in drying plants of various designs: drum dryers, spray dryers, fluidized bed dryers, etc. However, thermal methods are rarely used in practice due to their high capital and operating costs and the complexity of organizing the technological process. Based on the composition and properties of the slag and literature data, the possibility of its disposal in various areas has been established, like value-added biomaterials, 23 building materials, 24 cements²⁵ and materials for wastewater purification.^{[26](#page-8-0)} The most preferred direction is to return the slag to the process. However, the amount of slag introduced is limited to 10 wt%, because with a higher content, a deterioration in product quality is observed and breaks in the grid of paper machines are possible. One of the common methods of handling slag is heat treatment as incineration, 27 gasification 28 and pyrolysis,²⁹ including production of pellets.³⁰ The disadvantage of this method is the secondary pollution of the environment mainly due to emissions into the atmosphere. Shirvani and Noor $zad³¹$ proposed to use a slag ash for the production of clayey soil. Another option for handling slag is digestion,^{[32-34](#page-8-0)} separate hydrolysis and fermentation. 35 Slag biocomposting can be carried out by means of vermicomposting and mixing with other wastes, for example, mixed with bark and wood waste, peat and sawdust.

However, the slag composting process is lengthy and laborious due to the presence of poorly decomposable fibers in this type of waste. When composting, it is necessary to constantly maintain temperature, pH, humidity and additional nutrients, since violation of the composting conditions can lead to the death of the population of worms involved in vermicomposting. The process also requires large areas. As a sorbent, the slag can be used to collect spilled oils of vegetable and mineral origin from hard surfaces and water.³⁶ However, a problem is the regeneration of this sorbent and its final disposal. Burning the sorbent will lead to significant secondary air pollution. Lu et al.^{[37](#page-8-0)} proposed preparing a mixture as an auxiliary cementing material to prepare backfill material for mining. Hu et al.^{[38](#page-8-0)} proposed a lowered temperature recycling paper slug into artificial lightweight aggregates. Haile et al.^{[23](#page-8-0)} proposed utilization of slag for high-value-added biomaterials. An analysis of the literature and patent study show that the slag is most often used in the building materials industry, in particular in the production of waste-based cement 25 wall products, blocks and expanded clay. This research is of interest nowadays.³⁹ The idea of the paper has complied with the United Nations Sustainable Development Goal 12 which is about responsible consumption and production, prioritizing waste recycling and waste reduction via sustainable innovation processes. The technology for the production of wall panels is much simpler in comparison with the technologies for the production of expanded clay. It does not require high energy and economic costs for the production of the product. However, in this case, the issue of environmental safety of the resulting products arises due to the absence of a heat treatment stage. The most promising is the use of slag as a burnable additive in the

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The objectives of the study were: (i) to investigate the effect of various amounts and methods of introducing the slag on the properties of ceramic bricks and (ii) to determine the optimal firing temperature for ceramic bricks obtained using the slag.

MATERIALS AND METHODS

Materials and reagents

The slag was taken at various enterprises of the pulp and paper industry (the enterprises are located in central Europe). In all cases, the slag was taken after the treatment of industrial wastewater after its dehydration (Fig. [1](#page-1-0)). For research, samples were taken from three different enterprises in the same mass ratios (1:1:1). The composition of the used slag is presented in Table 1.

To obtain samples of ceramic bricks, clay, quartz sand as a lean additive and slag as a burnable additive were used.

Clay is characterized by the following composition (in terms of oxides, wt%): SiO₂, 55.70; Al₂O₃, 15.80; TiO₂, 1.07; Fe₂O₃, 6.58; CaO, 7.10; MgO, 3.20; K₂O, 3.50; Na₂O, 1.70; loss on ignition, 8.90.

Coarse-grained quartz sand was used with fineness of 0.15–1.50 mm and without carbonate inclusions. The content of clay impurities was not more than 3.5 wt%. The bulk density was 1380 kg m⁻³. .

Sample synthesis

The slag was introduced into the mixture in quantities of 5, 10 and 15 wt% without any preliminary preparation (wet samples W), or after preliminary preparation, which was carried out in three different ways: (i) drying at a temperature of 100 °C(samplesD);(ii) granulation toa diameter of 3–4 mm and drying at 100 °C (samples G); and (iii) drying at 100 °C and grinding to a size of 0.5–2 mm (samples DG). The compositions of the studied samples are presented in Table 2.

The samples were prepared by the plastic molding method. Moisture content of the molding mass was corrected taking into account the introduced additives. The bricks were dried in an oven at a

temperature of 100 °C. Then the dried samples were fired in an electric furnace. The brick firing mode was chosen in accordance with the heat treatment mode at a local ceramic brick factory: slowly raising the temperature to 580 °C while holding for 30 min at this temperature to remove moisture of crystallization from the clay (dehydration of clay minerals occurs at this temperature). Yaras^{[40](#page-8-0)} noted that at about 350 °C the combustion of slag occurs. The rate of temperature rise was 1 °C min−¹ . The temperature was further increased at a rate of 2 °C min−¹ to a temperature of 1000 °C with an exposure for 30 min to form a crystalline structure of the ceramic material. In the course of the work, analysis was carried out for at least three parallel samples.

Sample analysis

The elemental composition and morphology of the slag and obtained samples were investigated using a JSM-5610 LV scanning electron microscopy (SEM) instrument with an EDXJED-2201 chemical analysis system (JEOL, Japan).

X-ray phase analysis of the obtained samples was carried out with a D8 Advance AXS X-ray diffractometer (Bruker, Germany). The recording was carried out in the range $2\theta = 10-80^\circ$ with a step of 0.1–0.2° and the accumulation of pulses for 2 s. The obtained X-ray diffraction (XRD) patterns were identified using HighScore Plus software and the ICDD PDF-2 database.

For the obtained samples of ceramic bricks, shrinkage, apparent density, water absorption and compressive strength were determined.

The linear shrinkage of the samples was determined by measuring specially applied marks (4 cm long) before and after heat treatment with a vernier caliper with an accuracy of ± 0.01 mm in accordance with ASTM C326-03. Linear shrinkage (LS) was determined using the following equation:

$$
LS (%) = \frac{(LO - L) \cdot 100}{LO}
$$
 (1)

where L_0 is the label initial size (mm) and L the final label size (mm).

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Apparent density is the ratio of body mass to the entire volume occupied by the body, including pores. Apparent density was calculated using Eqn (2) according to ASTM C373:

$$
\rho = \frac{m}{V} \tag{2}
$$

where ρ is the apparent density (g cm⁻³), m the mass of fired bricks (g) and V the fired brick volume (cm³).

The weight of the samples was determined after drying them to constant weight. The volume of the samples was determined from their geometric dimensions, measured with an error of no more than 0.1 mm. To determine each linear dimension, a sample was measured in three places: along both edges and in the middle of the face. The final result is the arithmetic mean of three measurements.

To determine the water absorption, the brick samples were subjected to pre-drying to constant weight. Then they were placed in one row in height with gaps between them of at least 2 cm on a grate in a vessel with water at a temperature of 20 °C so that the water level was about 5 cm above the top of the samples. The samples were kept in water for 48 h. Samples saturated with water were removed from the water and weighed. Water intake was determined using Eqn (3) according to UNE-EN 772-11:

$$
B = \frac{m - m_1}{m} \cdot 100\% \tag{3}
$$

The compressive strength is the maximum compressive stress that a sample of a certain shape can withstand before its structure is broken. The compressive strength of bricks was determined in accordance with UNE-EN 772-1 using Eqn (4):

$$
R = P/F \tag{4}
$$

where P is the maximum load established during the test of the sample (H) and F the cross-sectional area of the sample, calculated as the arithmetic mean of the areas of its upper and lower surfaces (mm²).

When testing samples for compression, vertical axial lines were applied to the side surfaces of the samples. Each sample was

installed in the center of the press plate, aligning the geometric axes of sample and plate, and pressed by the upper plate of the press. The load on the sample must increase continuously and uniformly at a rate that ensures its destruction no earlier than 60 s after the start of the test.

RESULTS AND DISCUSSION

Slag composition analysis

The composition of the slag depends on the raw materials used in the production of paper and board.During the processing of waste paper, part of the short fibers of the feedstock passes into wastewater, and the components contained in the paper can then fall into the slag: (i) soluble (rosin glue, aluminium and iron sulfate, sodium aluminate, urea, starch, soda, hypochlorites, various resins, etc.) and (ii) insoluble (kaolin, chalk, talc, gypsum, wax, paraffin, etc.). Differences in the composition of the osprey can be associated with the use of other brands of waste paper as raw materials, as well as various additives introduced into the composition of the paper. In all cases, the content of the organic component was high, and was represented by randomly arranged fibers of small sizes (Figs $2(a)$ and (b)).

The powder part of the slag was very diverse, its main components being Si, Al and Ca. XRD analysis indicates that Si and Al were present in the slag in the form of quartz with a hexagonal structure with space group P3221 and characteristic peak (101) at 2 θ of 26.64° and kaolinite (Al₂Si₂O₉ phase) with a monoclinic structure with space group C1c1 and characteristic peaks (002) and (004) at 2 θ of 12.37° and 24.88° (Fig. 2(c)). Ca was found predominantly as calcite with a rhombohedral structure with space group R-3c and characteristic peak (104) at 2⊔ of 29.41°. Peak positions and parameters of crystal cells were in good agreement with appropriate cards and indicated a high degree of crystallinity. The peak at 2 θ of 23.11° corresponds to cellulose (002).

Analysis of samples of ceramic bricks

It was noted that the samples in which the amount of slag reached 15 wt% had the poorest molding properties and required the introduction of more water. The best molding properties were

Figure 2. (a, b) SEM images and (c) XRD pattern of slag.

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Figure 3. General optical images of obtained samples.

Figure 4. (a) Air shrinkage, (b) total shrinkage, (c) apparent density, (d) water absorption and (e) compressive strength of the obtained samples as functions of the content of slag.

characterized by samples with the addition of wet and granular slag. When using a dried slag, the forming properties deteriorate. Bricks obtained using a granulated slag have a more even shape, less deformed than samples obtained using a dried and ground slag; however, in comparison with samples with a ground wet and dried granular ground slag, they are more curved.

Deformation of samples can be associated with an incorrectly selected firing mode.

The slag also affects the color characteristics of the obtained samples (Fig. 3). As the slag content increases, the bricks acquire a lighter shade, which can be used for decorative purposes.

Figure 5. (a) Compressive strength and (b) sample density at various firing temperatures.

Results for shrinkage, apparent density, water absorption and compressive strength are presented in Fig. [4.](#page-4-0) All these results were obtained at a firing temperature of 1000 °C.

Air and total shrinkage decrease with increasing slag content and were in the range 5.5–10% and 7.2–12%, respectively, and correlate with previous results.^{[41](#page-8-0)} This is due to the fact that with an increase in the mass of the slag, the number of pores in the brick samples increases.

The apparent density of the obtained samples was in the range 1.29–1.88 $q \text{ cm}^{-3}$ and decreased with an increase in the amount of slag, comparable with previous results. 40

The water absorption of the obtained samples varies from 13.71 to 26.11 wt%. Moreover, with an increase in the content of the slag, water absorption also increases. This is due to the presence of pores formed from the combustion of the slag fibers. According to UNE-EN 772-11, the water absorption of ordinary products must be at least 6.0 wt%; front products not less than 6.0 wt% and not more than 14.0 wt%. All brick samples obtained comply with the requirements of UNE-EN 772-1 and can be used as ordinary products. However, none of the products obtained meet the

requirements for facing bricks, except for the sample obtained without the use of a slag. Obtained results are almost 1.5-fold higher compared to previous similar research 40 without sand in the molding compounds and comparable with the results for samples based on slag and laterite and alluvial soils in the mold-ing compounds.^{[39](#page-8-0)}

The compressive strength varies from 11.4 to 18.1 MPa. The strength values decrease with an increase in the slag content, which indicates the need to select a composition with a lower concentration. According to UNE-EN 772-1, the lowest allowable tensile strength for solid bricks is 7.5 MPa and the highest is 25 MPa. All obtained samples meet the requirements of the standard for strength. Obtained results for 15 wt% were almost 1.5-fold higher compared to similar previous research^{[40](#page-8-0)} without sand in the batch mixture; and 1.7- and 3.5-fold higher compared to laterite and alluvial soils in the batch mixture^{[39](#page-8-0)} with a slag content of 10 and 15 wt%, respectively.

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Based on all of the above, it can be concluded that the samples obtained using a dried ground slag exhibited the best values of the indicators, and the worst with the use of a granular slag. Thus,

Figure 6. SEM images of fired samples.

Figure 7. XRD patterns of fired samples.

when using a dried ground slag, the highest values of compressive strength of 14.8–17.5 MPa were observed, while with the introduction of a granulated slag, the values of compressive strength were the lowest (11.4–15.1 MPa). Also, samples obtained using a granulated slag were characterized by high values of air shrinkage (7.2–11.5%), while for samples using a dried ground slag, the total shrinkage was in the range 8.5–7.8%. However, the use of dried ground slag in production is a very energyintensive and economically unprofitable option for processing slag. The use of this method of preparing the slag requires a drying stage, which will be associated with emissions of flue gases into the atmospheric air, as well as an increase in the costs of the drying process. Next, the stage of grinding the dried slag is needed. During the experiment, it was found that grinding the slag was difficult, and also leads to heating of the grinding equipment; therefore, the stage of granulating the slag with its subsequent grinding is also necessary. But this will increase the cost of the granulation process. At the same time, at the subsequent stage of crushing the slag, the resulting dust can be released into the atmospheric air, thereby requiring the installation of cleaning equipment. Therefore, such a long, laborintensive and energy-consuming technology for preparing the slag is inappropriate.

Samples obtained using a wet slag differ little in their characteristics from samples obtained using a dried and ground slag. Thus, when using dried ground and wet slag, the highest values of compressive strength were observed to be 14.8–17.5 MPa and 15.8– 17.1 MPa, respectively. The values of the total shrinkage of these samples were also close, being in the range 7.3–9.8% for a wet slag and 8.5–9.8% for a dried, crushed slag. In addition, no preliminary preparation of the slag is required, which reduces energy costs, which is why this method was chosen as the most promising option for processing the slag. As mentioned above, the introduction of slag in an amount of 15 wt% significantly impairs the molding properties of the plastic mass, and with an increase in the slag content, a decrease in compressive strength was observed, so this concentration cannot be used for the production of ceramic bricks.

When the slag content was 5 wt%, higher values of total shrinkage (9.8%) were observed than when the slag was introduced in the amount of 10 wt% (8%). However, when using 5 wt% slag, higher values of compressive strength (17.1%) were observed, while when introducing 10 wt% slag, the compressive strength was 16.4 MPa. But since there were no large discrepancies between these values, and the introduction of a slag in the amount of 10 wt% will increase the amount of slag used in the technological process of producing ceramic bricks with obtaining marketable products, it is advisable to choose this value as optimal. Thus, the most promising is the introduction of a wet slag with a concentration of 10 wt% into the molding mass.

After molding, the samples were dried and fired. To determine the firing temperature, the samples were subjected to heat

treatment at temperatures of 900, 950, 1000 and 1050 °C and their compressive strength was determined (Fig. [5](#page-5-0)). For comparison, studies were carried out in parallel with brick samples obtained without the addition of slag.

Based on the experimental data, the optimal firing temperature for samples containing 10 wt% slag was chosen as 950 °C, while the firing temperature of brick without slag was 1000 °C. Thus, the use of the slag in the production of ceramic bricks will allow one to address the problem of waste management, turning the slag into secondary raw materials. At the same time, the use of slag will lead to a decrease in the brick firing temperature by 50 °C, which will ensure a reduction in natural gas consumption.

Brick samples containing 10 wt% slag and fired at a temperature of 950 °C were characterized by a density of 1.85 g cm⁻³, a compressive strength of 17.8 MPa and, according to GOST 530, corresponded to the M175 brick grade.

According to the images presented in Fig. [6,](#page-5-0) it can be said that when using a ground dried slag, the most uniform structure with small pores was formed; when using a wet one, a structure was formed with linear pores in the form of fibers (which burned out during firing). Samples W-10, D-10 and DG-10 were characterized by the predominant formation of pores with sizes of several tens of nanometers.

The diffraction patterns of the fired samples, for the examples of the control sample and sample W-10, were identical, and show the predominant content of quartz with a hexagonal structure with space group P3221 and characteristic peak (101) at $2\theta = 26.64^{\circ}$; anorthoclase (Na,K)(Si₃Al)O₈ phase with an anorthic structure with space group P-1 and characteristic peaks at $2\theta = 21.85^{\circ}$, 23.65°, 24.25°, 27.54°, 26.65°, 27.68° and 27.96°; and annite Si_{5.1}Al_{6.9}Fe₆₋ $K_{1.98}$ Na_{0.02}O₂₄ phase with a monoclinic structure with space group C12/m1 and characteristic peak (001) at $2\theta = 8.73^{\circ}$ (Fig. [7\)](#page-6-0).

Table [3](#page-6-0) presents a comparative analysis of the obtained results with similar research done before.

The samples obtained in this research can be classified as lightweight and medium weight (1680–2000 kg m $^{-3}$) in line with ASTM C90. According to ASTM C62, water absorption of the obtained samples is below the 22% limit (all W-x and G-x samples). But it should be mentioned that in some countries this parameter has no limitations. According to TS-EN 771-1 standard, the compressive strength of bricks should be at least 7 MPa. In line with the data obtained, samples may be used as building materials. According to ASTM C62 standard, the compressive strength of clay-based bricks is required to be 20.7, 17.2 and 10.3 MPa for severe, moderate and negligible weathering, respectively. This means that the obtained samples are suitable for moderate and negligible weathering conditions.

Thus, the use of slag in the production of ceramic bricks will make it possible to turn waste into a secondary material resource, save raw materials and also reduce the firing temperature by 50 °C.

CONCLUSIONS

- An increase in the content of slag in ceramic bricks leads to a decrease in linear shrinkage, apparent density and compressive strength and an increase in water absorption.
- The best properties were exhibited by samples obtained using a pre-dried and ground slag, but this leads to additional costs. When using the slag after dehydration without additional processing, the properties of the samples deteriorate slightly, and

the absence of additional costs for waste preparation makes this option preferable.

- The properties of brick samples containing 10 wt% slag correspond to the interstate standard GOST 530 grade M175.
- The introduction of slag in an amount of 10 wt% allows not only a saving of natural raw materials in the production of ceramic bricks, but also a reduction in energy costs due to a decrease in firing temperature by 50 °C (from 1000 to 950 °C).

AUTHOR CONTRIBUTIONS

Volha Zalyhina: Conceptualization. Formal analysis. Investigation. Methodology. Resources. Supervision. Data curation. Validation. Writing - original draft. Writing - review & editing. Victoria Cheprasova: Formal analysis. Investigation. Data curation. Validation. Valentin Romanovski: Formal analysis. Data curation. Validation. Visualization. Writing – original draft. Writing – review & editing.

ETHICS APPROVAL AND CONSENT TO **PARTICIPATE**

Not applicable.

CONSENT FOR PUBLICATION

Not applicable.

COMPETING INTEREST

The authors declare no competing interest with any previous work.

DATA AVAILABILITY

All data employed in support of the outcomes in the study are included in this article.

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