



## Application of Sorbent Waste Material for Porous Ceramics Production

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### ABSTRACT

The paper considers the application of waste sorption material utilization and pumpkin seed husks formed during the extraction of heavy metal ions from aqueous solutions, as a combustible additive to clay mixtures in production of the porous ceramics. In this regard, this study evaluates the effects of different amounts (2-8%) of the spent sorption material in the charge composition with changes in the physical and mechanical properties of ceramic samples obtained by firing at temperatures of 950-1050 °C. One finding is that the combustion of the organic additive is accompanied by the formation of voids and the release of gases with the formation of pores in the ceramic piece. Another finding is that all clay mixtures with a combustible additive allow the production of porous ceramics to meet the requirements for compressive strength, porosity, density, water absorption and linear shrinkage. It is recommended using 4 % of combustible additive in order to obtain optimal properties in terms of density and strength. During the testing of the developed porous ceramics for heavy metal leaching, the material does not pose an environmental hazard. Finally, the results of this study are applicable for the construction of internal partitions and household buildings.

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## 1. INTRODUCTION

Wastewater treatment sludge is one of the most widespread and large-scale wastes. Annually, around the world, millions of tons of such wastes are generated, which are partially utilized and used later in agriculture and production cycles. However, most of them are stored at industrial landfills, disposing large areas of fertile soils and having a negative impact on the environment. In addition, construction is an industry where sewage sludge can be used. This excludes some costly and energy-intensive recycling steps and the resulting construction material is often stable and safe [1-3]. Extensive research was carried out on the use of sewage sludge in the production of building materials such as roofing tile, bricks, lightweight aggregates, cement, concrete and geopolymers [4,5]. Particular attention is paid to wastewater treatment sludge containing heavy metals. The potential hazard of heavy metals depends on the physic-chemical forms of metals in the sludge. Heavy metals are considered as one of the hazardous pollutants

because they are toxic with high persistence in the environment and food chain. In addition, these metals do not decompose and have the ability to accumulate in the environment and present one of the most dangerous pollutants in the biosphere [6,7].

The existing literature sources touch upon the issue of the economic efficiency of sorbents from agricultural crops for removing heavy metal ions from wastewater [8] and the efficiency of using agricultural waste in the construction industry [9-11]. The metal-containing wastes formed after water treatment pose an environmental hazard and necessitate their disposal. The use of organic sediments for water treatment of heavy metals is a promising option in the production of ceramic products [12,13] and porous ceramics [14]. This solution makes it possible to avoid secondary pollution and also increases the value of the waste converting it into a useful material. In addition, sewage sludge acts as a fuel during the sintering process and saves up to 40% of energy for firing [15].

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During sintering the components of organic matter coming from the sewage sludge additionally emit gases, which contribute to the formation of pores and the creation of a porous structure of ceramic material, which characterizes it as a combustive additive [16].

Recent studies in this research area reveal that the heat treatment allows the fixation of heavy metals coming from the sludge of treated wastewater in a silicate structure. When the sintering temperature of ceramics is reached, heavy metals interact with basic clay minerals (for example, alumina, kaolinite, and hematite) and form a crystalline phase of aluminosilicates and silicates [17,18]. The synthesis of various compounds can depend on temperature and basic oxide content.

One of the main trends in the literature review refers that the ceramic matrix stabilizes the mobility of heavy metals, reducing the potential hazard of their release [19,20]. According to the results of the leaching test, a low limit of extraction of metals such as Pb, Cd, Zn, Cr, Ni and Cu is noted [6], this confirms their stabilization in the ceramic structure of building material [21]. The tests carried out in the work [22] confirm the harmlessness of ceramic materials obtained using wastewater treatment sludge from heavy metals during their operation.

To provide an additional step to the advancement of knowledge in this research area and in order to expand the range of methodological approaches to the disposal of wastewater treatment sludge containing heavy metals in the production of ceramic products, we studied the effect of wastewater treatment sludge with nickel ions  $Ni^{2+}$  (WTSN), where crushed pumpkin seed husk (PSH) was used as a sorbent. The main purpose of this research is to develop porous ceramics with low density, high porosity and without significant changes in mechanical strength.

## 2. MATERIALS AND METHODS

We used the Yastrebovsky deposit clay (Belgorod region, Russia). The chemical composition (Table 1) was determined by X-ray fluorescence using ARL 9900 WorkStation X-ray fluorescence spectrometer with a built-in diffraction system, cobalt anode. According to the classification of clay raw materials GOST 9169-75 (Russia), this clay in terms of content ( $Al_2O_3 < 15\%$ ) belong to acidic clay, in terms of the content of coloring oxides it belongs to a group with a high  $Fe_2O_3$  content and a low  $TiO_2$  content.

In order to study the features of phase formation and sintering of a ceramic piece, the measurements of heat and mass fluxes were carried out when clay samples were heated to  $1000\text{ }^\circ\text{C}$ . We used a STA 449 F1 Jupiter® synchronous thermal analysis by NETZSCH Proteus® software. According to the results (Figure 1) in the range from  $70$  to  $130\text{ }^\circ\text{C}$  (peak point is  $86.3\text{ }^\circ\text{C}$ ) the endothermic effect is observed associated with the removal of adsorbed water. The exo-effect is observed in the range from  $130$ - $430\text{ }^\circ\text{C}$  - organic impurities are combusted. Endoeffects characterizing the phase transitions of clay minerals occur in the ranges from  $430$  to  $550\text{ }^\circ\text{C}$  (peak point is  $469.3\text{ }^\circ\text{C}$ ). The endothermic effect characterizing the polymorphic transformations of quartz occurs in the range from  $550$  to  $650\text{ }^\circ\text{C}$  (peak point is  $577.8\text{ }^\circ\text{C}$ ). The endothermic effect of the decomposition of carbonates is observed in the range from  $650$  to  $750\text{ }^\circ\text{C}$  (peak point is  $724.6\text{ }^\circ\text{C}$ ). The determination of the phase composition of a clay sample, ground through a 008 sieve, was carried out by powder X-ray diffraction using ARL XTRA Termo Fisher Scientific diffractometer, copper Cu anode. In this regard, the results of X-ray of the clay are drawn in Figure 2.

TABLE 1. Clay chemical composition percentage

$SiO_2$	$TiO_2$	$Fe_2O_3$	$CaO$	$MgO$	$Na_2O$	$K_2O$	$Al_2O_3$	LOI	$\Sigma$
71.3	1.02	4.62	1.61	1.2	0.62	2.06	14.3	3.27	100.00

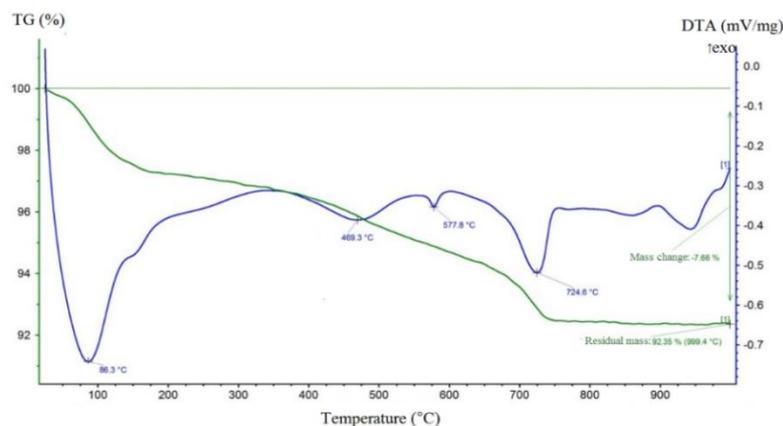


Figure 1. DTA and TG curves for the clay

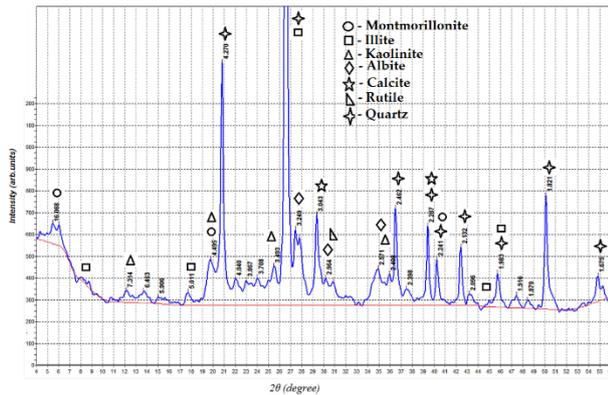


Figure 2. X-ray of the clay

In accordance with GOST 9169-75 (Russia), the following technological properties of clay were determined: plasticity; coefficient of sensitivity to drying. Based on the obtained indicators, the clay of the Yastrebovsky deposit belongs to the moderately plastic class - plasticity number  $P = 15$ ; the studied clay is insensitive to drying with a sensitivity coefficient  $K_s = 0.4$ .

In addition, WTSN was obtained treating model waste water containing nickel ions. PSH was used as a sorbent. The number of  $Ni^{2+}$  ions in the solution was 100. The sorption material had a mass of 1 g. The solution with the sorbent was stirred for 20 minutes. The moisture content of the WTSN was 30%, the content of nickel ions was 0.24%. The size of the PSH particles used for cleaning was 1-2 mm. WTSN was used as a combustible additive in the preparation of porous ceramics.

In order to prepare clay samples, raw clay was roughly crushed in a laboratory jaw crusher. The crushed clay was dried to a moisture content of 7% and then dry grinding was carried out in laboratory runners of the "LM - 2e" brand, followed by sieving through a sieve No. 063. The WTSN additive was introduced in the amount of 2, 4, 6, 8 % while reducing the proportion of clay in the mixture. WTSN was mixed with clay in a mill for 15 minutes. Then the mixture of clay with the addition of WTSN was moistened with water to a moisture content of 18%. The prepared clay mass was kept for 7 days in order to complete the formation of adsorbed hydration shells. Then, the samples - cubes with a size of  $30 \times 30 \times 30$  mm were molded from the clay mixture by plastic molding. Preliminary drying of the samples was carried out under natural conditions for 7 days and then in a drying cabinet at a temperature of  $100-110^\circ C$  to a residual moisture content of 1% (4 hours at  $T = 50-60^\circ C$  and 6 hours at  $T = 110^\circ C$ ). The samples were fired in a SNOL-1/9 muffle furnace at temperatures of 950, 1000,  $1050^\circ C$ .

After firing, the samples were tested for a number of physical and mechanical properties, such as compressive

strength, average density, total porosity and water absorption in accordance with GOST 2409-95 (ISO 5017-88) (Russia). The samples without addition of WTSN were taken as control compositions.

The study of the microstructure of finished products was carried out using the method of scanning electron microscopy (SEM) by using high-resolution microscope TESCANMIRA 3 LMU.

### 3. MEASURES AND RESULTS

#### 3. 1. Physical and Mechanical Properties of Ceramic Sample

With the addition of WTSN to the clay mixture, the number of voids and rounded pores of various morphology increases. Figure 3 shows the change in the structure of ceramic samples obtained at a firing temperature of  $1050^\circ C$ . The samples with the addition of WTSN have deep voids with broken edges with a maximum size of  $100-400 \mu m$ . Small micro-voids of  $\leq 50 \mu m$  are noted. The size of closed pores is in the range  $10-50 \mu m$ .

The presence of voids is the result of the action of WTSN as a combustible additive. The micrograph of the ceramic structure of the sample fired at a temperature of  $950^\circ C$  (Figure 4a) clearly shows a WTSN particle included in the clay matrix, which leaves fixed voids during combustion (Figure 4b). According to the literature [23-26], with the increase in the firing temperature of ceramic mixtures with organic combustible additives, the process of the formation of a

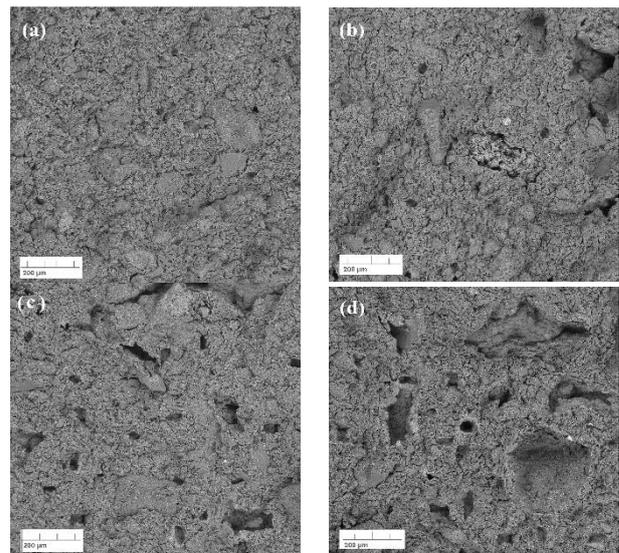
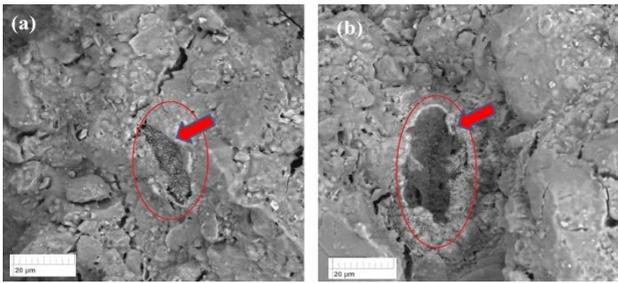


Figure 3. SEM analysis of the microstructure of ceramic samples fired at a temperature of  $1050^\circ C$ : (a) Without additive; (b) With the addition of 2% WTSN; (c) With the addition of 4% WTSN; (d) With the addition of 8% WTSN

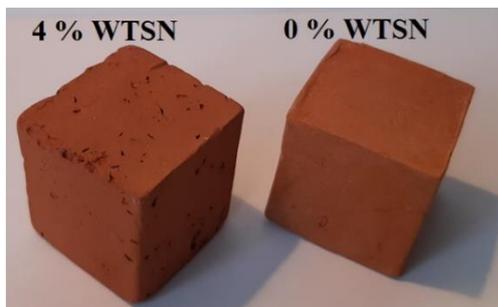


**Figure 4.** SEM analysis of the microstructure of ceramic samples: (a) With the addition of 4% WTSN, fired at a temperature of 950 °C; (b) With the addition of 4% WTSN, fired at a temperature of 1050 °C

liquid phase on the surface of the raw mixture particles intensifies. The formed water penetrates into pores and capillaries formed by gas release and participate in the processes of hydration of the cementing binder with the formation of thin films at the interface between the phases, contributing to the consolidation of pores and voids.

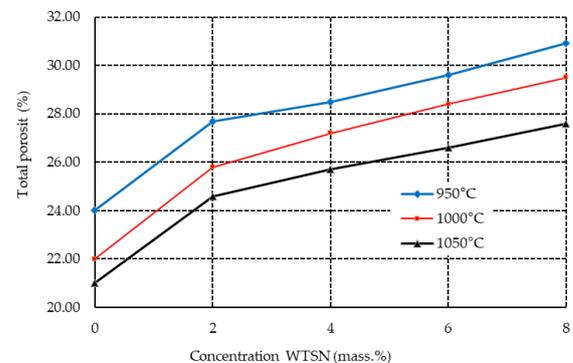
The pores in the structure of a ceramic piece are formed as a result of the participation of gaseous products of CO<sub>2</sub>, released during the combustion of organic matter [27]. The visual analysis of ceramic brick samples is shown in Figure 5; where there is no color change and increase in the number of voids with the introduction of WTSN additive.

An increase in the concentration of voids has reflected in the total porosity of ceramic samples (Figure 6). It goes without saying that voids act as a barrier for heat flow. With an increase in the firing temperature from 950 to 1050 °C, the total porosity of the product decreases. The highest porosity of 30.9% was achieved with the addition of 8% WTSN and a firing temperature of 950 °C. The lowest values of porosity were 24.6% with the addition of 2% WTSN and a firing temperature of 1050 °C. An increase in the total porosity is reflected in the decrease in the density of ceramic samples (Figure 7). In this regard, this study uses the metal in the structure of the ceramic samples that potentially enhance thermal conductivity. It is noted that the maximum density

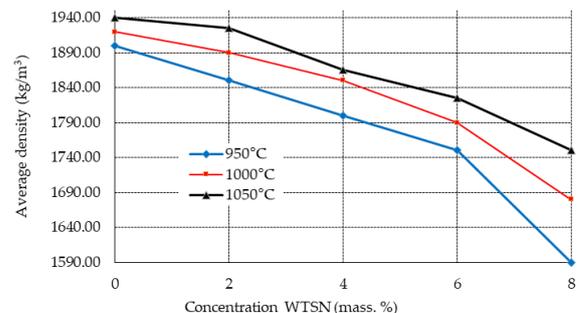


**Figure 5.** Samples of ceramic products based on 4% WTSN and 0% WTSN

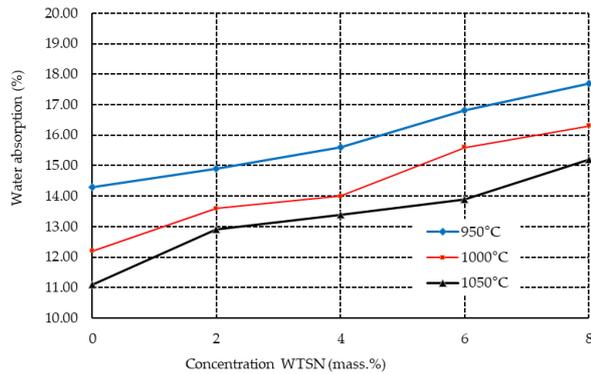
reduction of 1590 kg/m<sup>3</sup> is achieved with the introduction of an 8% additive and a firing temperature of 950 °C. The porous microstructure offers advantages for specific applications of the developed, such as thermal insulation or thermal resistance, which allows bricks to withstand sudden temperature changes through the reduction of the modulus of elasticity and shear [28]. Voids isolate the heat flow, causing the decrease in the thermal conductivity of the samples. The developed porous structure leads to an increase in the value of water absorption (Figure 8). It is known [29] that water absorption of a high-quality porous ceramic product should be within the range of 6 - 20% by mass. It is noted that in all studied cases, the water absorption indicators do not cross the permissible limit (20%). Figure 9 shows the change in the mechanical strength of clay samples. The strength of the samples decreases with an increase in the amount of additive and the increase in porosity, which is typical for porous ceramic materials [30]. The average compressive strength of the developed ceramic samples of the control composition (without additives) in the firing temperature range of 950-1050 °C was 21.5 MPa. Higher sintering temperatures increased compressive strength. With an increase in the concentration of WTSN in the ceramic matrix, it is accompanied by the decrease in the strength of the fired samples.



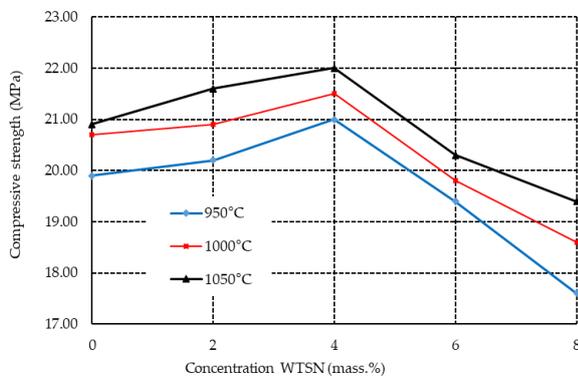
**Figure 6.** Change in the total porosity of ceramic samples from the composition of the charge and the firing temperature



**Figure 7.** Change in the average density of ceramic samples from the composition of the charge and firing temperature



**Figure 8.** Change in water absorption of ceramic samples from the composition of the charge and firing temperature



**Figure 9.** Change in the physical and mechanical properties of ceramic samples from the composition of the charge and firing temperature

According to the purpose of this study to obtain highly porous clay ceramics with sufficient strength, it is necessary to note that, in connection with the available data, the strength of porous ceramics with the use of organic combustible additives is in average 20-25 MPa [25,31-33].

We should admit that the optimal introduction of WTSN into the clay mixture as a combustible additive is up to 4 %. In this case, the compressive strength is within acceptable limits of at least 20 MPa. The migration of gases through the matrix, obtained as a result of the combustion of WTSN in an amount of more than 4 %, created a highly porous clay material, which negatively affected the mechanical strength.

In order to select the optimal firing mode and obtain ceramic objects with specified dimensions, the values of air and fire shrinkage of the dried and fired samples were determined at different firing temperatures (950-1050 °C) and different contents of the combustible additive WTSN. Shrinkage during drying is based on the amount of water in the test material. The amount of air shrinkage varies depending on the crystallinity of clay mineral. The change in the firing shrinkage based on the firing

temperature indicates the level of the caking of clay raw materials. Table 2 shows the observed shrinkage during the drying and firing cycles of clay bricks containing various concentrations of additives.

An increase in the mass fraction of WTSN in the composition of the clay mixture leads to the increase in the indicators of air and fire shrinkage. However, these changes do not lead to the deformation and deterioration in the quality of the resulting products. In accordance with GOST 530-2012 (Russia), for ceramic bricks, air shrinkage is allowed up to 8%, fire shrinkage - from 1% to 2%. All the values obtained are within the standard range. The observed relatively low values of air shrinkage of 2.27-2.79% when firing clay without additives is typical for sandy clays, such as used clay with a content of 71.3% SiO<sub>2</sub> (Table 1). An increase in the amount of introduced additive causes a natural increase in air and fire shrinkage, which is associated with the removal of residual and chemically combined water, as well as with the transformation of additives into ash. These chemical reactions during firing, along with rearrangement of particles and regulation in the crystal lattice, form a more compact solid texture in comparison with the initial state, which causes shrinkage deformations [34].

### 3. 2. Nickel Ion Leaching Testing of Porous Ceramics

The additive of waste sorption material contains compounds of heavy metals, potentially hazardous to the environment. We investigated the probability of migration into the environment of WTSN nickel ions, enclosed in the structure of a ceramic piece. Powder of porous ceramics with the addition of WTSN was ground to a particle size of less than 0.08 mm and placed in solutions of hydrochloric acid at pH = 3; 4; 5; 6, was kept for 24 hours at an aqueous medium temperature of 20 ± 0.5 °C. During the experiments, the specified pH value was maintained by acidification. Then the suspensions were filtered through a paper filter and the concentration of Ni<sup>2+</sup> ions were determined by the atomic adsorption method according to GOST R 57162-2016 (Russia). The experimental results (Table 3) indicate the resistance of ceramic products to leaching of Ni<sup>2+</sup> ions, which is estimated as the value of the concentration of Ni<sup>2+</sup> ions in an acid solution.

**TABLE 2.** The impact of the content of wastewater treatment sludge in the raw mixture on the shrinkage<sup>1</sup> of ceramic samples

Burning temperature (°C)	Additive content in the charge percentage				
	0	2	4	6	8
950	2.27/0.80	2.28/0.90	2.99/0.98	2.84/1.12	3.04/1.20
1000	2.31/0.91	2.58/1.02	3.04/1.20	3.19/1.26	3.52/1.39
1050	2.79/1.10	3.12/1.23	3.52/1.39	3.73/1.47	4.03/1.59

<sup>1</sup>linear air shrinkage value (%) / fire shrinkage value (%).

**TABLE 3.** Ion washout dynamics Ni<sup>2+</sup>

WTSN content in clay mixture (%)	Ion concentration Ni <sup>2+</sup> (mg/dm <sup>3</sup> )				
	pH=3	pH=4	pH=5	pH=6	pH=7
0	0.000	0.000	0.000	0.000	0.000
2	0.000	0.000	0.000	0.000	0.000
4	0.000	0.000	0.000	0.000	0.000
6	0.000	0.000	0.000	0.000	0.000
8	0.001	0.001	0.000	0.000	0.000
16	0.001	0.001	0.000	0.000	0.000
24	0.001	0.001	0.000	0.000	0.000

The insignificant leaching of nickel from ceramic powder occurs in the samples containing more than 8% WTSN at pH=3 and 4. At pH>4, nickel is not leached out. We should note that according to GOST R 57162-2016 (Russia), the concentration of nickel ions in solutions with pH=3 and 4 is lower than the LOC for nickel water for fishery facilities (0.01 mg/dm<sup>3</sup>). Thus, the disposal of waste sorption material in the production of ceramic bricks eliminates the risk of nickel entering the environment.

#### 4. CONCLUSION

Here, we provide the conclusion and findings of this paper as follows:

- The possibility of the use of WTSN based on pumpkin seed husks as a combustible additive to clay mixtures was investigated.
- Microstructure SEM analysis showed that the combustion of the organic additive promotes the formation of voids, and the emission of gases to the formation of pores in the ceramic piece during firing.
- The addition of WTSN and changing the firing temperature within the range of 950-1050°C is reflected in the indicators of ultimate strength in compression, water absorption, total porosity and density of ceramic samples.
- The optimal amount of WTSN as a combustible additive for the production of porous ceramics is not more than 4 %. In this case, as a result of firing at the temperature range from 950 to 1050°C, the compressive strength was 20.0-20.7 MPa, the density of the product was 1800-1860 kg/m<sup>3</sup>, and the water absorption was in the range of 15.6-13.4%. The linear shrinkage values of the resulting products were within the standard range.
- The dynamics of the washing out of heavy metal ions from the ceramic matrix began when the content of the WTSN additive was over 8% and

ended completely at pH values > 4, which eliminated the danger of their entry into the environment.

- The use of WTSN in a clay mixture allowed obtaining porous ceramics with satisfactory technical and operational characteristics, which could be used as an effective building material for the construction of internal partitions and household buildings.
- The use of optimization theory to reduce the waste materials with novel metaheuristics like red deer algorithm [35-36], water wave optimization [37-38] and social engineering optimizer [39], is another interesting idea for further research.

#### 5. ACKNOWLEDGMENTS

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### Persian Abstract

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#### چکیده

در این مقاله امکان استفاده از مواد جاذب زیاله - پوسته بذر کدو تنبل، که هنگام استخراج یون های فلزات سنگین از محلول های آبی ارائه شده است، که به عنوان یک افزودنی قابل احتراق برای مخلوط های رس در تولید سرامیک های متخلخل استفاده گردید. تأثیر مقادیر مختلف (2-8٪ جرم) مواد جذب شده صرف شده در ترکیب شارژ در تغییر در خصوصیات فیزیکی و مکانیکی نمونه های سرامیکی بدست آمده در دمای 950-1050 درجه سانتیگراد مورد بررسی قرار گرفت. مشخص شد که احتراق مواد افزودنی آلی موجب تشکیل حفره ها و انتشار گازها با تشکیل منافذ در قطعه سرامیکی همراه است. همه مخلوط های رس با افزودنی قابل احتراق امکان تولید سرامیک متخلخل را فراهم می آورد که شرایط لازم برای مقاومت فشاری، تخلخل، چگالی، جذب آب و انقباض خطی را برآورده می کند. توصیه می شود تا حداکثر 4 درصد وزنی استفاده گردید. درصد مواد افزودنی قابل احتراق به منظور به دست آوردن خواص بهینه از نظر چگالی و استحکام. در طول آزمایش سرامیک متخلخل توسعه یافته برای شستشوی فلزات سنگین، مشخص شد که این ماده خطری برای محیط زیست ایجاد نمی کند. مصالح ساختمانی حاصل برای ساخت پارتیشن های داخلی و ساختمانهای خانگی توصیه می شود.

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