



## Closed cycle of recycling of waste activated sludge



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### ABSTRACT

The recycling of waste activated sludge (WAS) formed in the process of biological purification of sewage is an urgent ecological problem. In the present work, two ways of recycling of WAS containing from 8 to 30% free water, namely, the synthesis of a carbon-containing component and synthesis of porous building ceramics (bricks) with the use of WAS and waste carbonizate, have been considered. For the preparation of a carbon adsorbent, the carbonization of WAS has been carried out in an argon atmosphere. For the synthesis of ceramics, clay-cullet-tezontle-WAS mixtures with different contents of the components have been used. Sintering has been performed in air.

It has been established that, in treatment of WAS at 600 °C for 30 min, better adsorption properties are obtained due to the presence of free carbon bonds. The efficiency of water purification from dyes (methylene blue) depends on the standard conditions: the methylene blue concentration, carbonizate-to-solution ratio, and exposure time of the carbonizate in solution. The use of wet WAS makes it possible to exclude the addition of water from the traditional scheme of preparation of a plastic semiproduct, i.e., realize a water-saving technology. The introduction of low-melting cullet, basalt, and WAS powders into red clay makes enables us to reduce substantially the sintering time of porous bricks (down to 8 h) and vary their strength properties.

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## 1. Introduction

At present, the biological method with the use of activated sludge is extensively employed for the final purification of sewage, as indicated in (Asano et al., 2007; van Haandel and van der Lubbe, 2007; Chan et al., 2009; Tilley, 2011; Activated Sludge Process, 2012). The biopurification process is based on the ability of microorganisms to utilize organic and inorganic substances for nutrition in the process of their vital activity (organic substances are carbon sources for microorganisms). Contacting with them, microorganisms partially destruct them into ecologically neutral compounds, transforming aromatic and aliphatic hydrocarbons into innocuous carbon dioxide, water, nitrite ions, sulfate ions, etc. The other part of the substance is consumed on the formation of biomass. Activated sludge consists of suspended particles that were not arrested in primary settling tanks and adsorbable colloid substances with microorganisms reproduced on them. The composition of activated sludge depends on the composition of sewage,

contents of salts and oxygen in it, temperature, pH, etc. In terms of a dry substance, activated sludge contains 70–90% organic substances and 10–30% inorganic substances. The treatment and recycling of waste activated sludge (WAS) removed from the system, as a rule, turn to be a much more laborious problem than the sewage purification process because these waste have different composition, high moisture, and large volume and contain a number of dangerous elements. This is why the recycling of such waste is an important environmental conservation problem of the industry.

There exist a number of methods of recycling of WAS. A promising method of recycling of waste activated sludge that includes neutralization of heavy metals is obtaining pyrolysis products and activated coal on the base of them, as described in (Tilley, 2011; Activated Sludge Process, 2012; Ma et al., 2017; Ruiz-Gómez et al., 2017, Maroušek et al., 2017). Depending on the pyrolysis regime, adsorbents that remove pollutants of different type from water are obtained. As a rule, the solid residue (carbonizate) is activated by different methods to increase the porosity (of the surface) of the material, as was shown in (Fasoli and Genon, 1976; Jeyaseelan and Lu, 1999; Ahmedna et al., 2000; Rozada et al.,

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2003, 2005; Al-Qodah and Shawabkah, 2009; Wu et al., 2015; Chen et al., 2015; Sheha et al., 2013). A manufacturing technology of inexpensive adsorbents proposed in (Grassi et al., 2012) arouses particular interest because activation processes of carbonizates lead to high additional financial expenditures. Furthermore, various technological schemes for the preparation of activated carbon are laborious and energy-intensive and introduce additional contaminants into the environment. Note that low-temperature and medium-temperature pyrolysis in an argon atmosphere (or under conditions of oxygen deficiency) in the range 450–800 °C are characterized by the largest yield of the solid residue. This feature of pyrolysis formed the basis for investigations of properties of the low-temperature carbonizate obtained from WAS. On the other hand, in view of the principle of preparation of porous ceramics, it was interesting to consider the synthesis of porous aluminum silicate ceramics due to the burnup of WAS.

The objective of the present work is to investigate the possibilities of reprocessing of WAS only after partial elimination of free water into two types of materials, namely, the low-temperature carbonizate (adsorbent) without the activation stage of the carbon component, and into porous brick with use of WAS and waste carbonizate.

## 2. Experimental

To investigate properties of the carbonizates obtained from WAS containing 8–12 wt.% free water, balls ~20 mm in diameter were formed and placed into a vacuum chamber. In the chamber, a pressure of  $10^{-2}$  mm Hg was produced, after which it was blown by argon for 10 min. In the chamber, specimens were placed into air-tight steel sleeves, and then the sleeves were relocated into a muffle furnace. The temperature treatment of specimens was carried out in the temperature range 200–700 °C for 10–60 min. For comparison, a part of specimens was burnt in air. After investigation of the adsorption properties, the waste carbonizate was used as a pore-forming component in sintering of brick products.

For the preparation of ceramics (bricks), five compositions were used for the preparation of billets. These are

- (1) 50 wt.% red clay–50 wt.% glass–water for plastification;
- (2) 50 wt.% red clay–50 wt.% glass–40 g of WAS containing ~ (20–30) wt.% free water for plastification;
- (3) 50 wt.% red clay–50 wt.% glass–10 wt.% damp/dry carbonizate–water for plastification;
- (4) 50 wt.% red clay–50 wt.% glass–10 wt.% damp/dry carbonizate–WAS in an amount providing forming of billets;
- (5) 50 wt.% clay–40 wt.% milled ceramics obtained from the composition 4–10 wt.% wet/dry/carbonizate–WAS in an amount providing forming of billets.

Similar mixtures (6, 7) were prepared with the use of milled ceramics obtained in the previous stage.

Specimens were sintered in air at 1000 °C for 8 h. Plastically formed laboratory briquettes had sizes  $12 \times 20 \times 60$  mm.

Along with the indicated series, specimens were synthesized from different mixtures plasticized by 40 g of WAS containing 30 wt.% free water, namely,

- (1) red clay–milled glass –WAS;
- (2) red clay–tezontle–WAS;
- (3) red clay–milled glass–tezontle–WAS.

The choice of the components of the mixtures was governed by the necessity of recycling large volumes of cullet, products of

stone-working production, and WAS, which contaminate the environment.

Specimens were sintered in air at 1000 °C for 8 and 12 h. The plastically formed briquettes had sizes  $120 \times 50 \times 240$  mm.

The obtained material was investigated by the X-ray diffraction (XRD) method in Cu  $K_{\alpha}$  radiation (Siemens D-500 diffractometer). An electron microscopy study and electron-probe microanalysis were performed with a LEO 1450 VP type and SU 5000 Hitachi scanning electron microscopes. An electron paramagnetic resonance spectroscopy (EPR) investigation was carried out on an EPR spectrometer (Jeol JES TE-300, Tokyo, Japan) operated in the X-band mode at a modulation frequency of 100 kHz at room temperature under a pressure of  $10^{-2}$  mm Hg. An IR study was performed with a Bruker ALPHA-P FT-IR spectrometer. The contents of oxides and carbon in specimens were determined with use of an X-ray Fluorescence S8 TIGER spectrometer (Bruker). Adsorption properties were studied by the UV–vis method with the use of an USB4000-XR1 Ocean Optics spectrometer. For the determination of the adsorption properties of the carbonizate, a 50 ppm aqueous solution of MB was used. The MB content was evaluated from changes in the intensity of the band of UV–vis spectra with  $\lambda \sim 665$  nm and preliminarily prepared calibration graphs:  $C = f(I)$ , where  $C$  is the dye concentration,  $I$  is the intensity of the band of UV–vis spectra. The mechanical properties of the ceramic were investigated by standard techniques.

## 3. Results and discussion

### 3.1. Characterization of the raw material

The composition of WAS is presented in Table 1. Biowaste contain predominantly carbon, smaller amount of oxygen,

**Table 1**

The content of composition of the active sludge waste, carbonizate and waste carbonizate after adsorption of waste water of textile factory.

| Formula                        | Initial WAS | Carbonizate | Waste carbonizate |
|--------------------------------|-------------|-------------|-------------------|
| C                              | 99.10%      | 98.10%      | 66.90%            |
| SiO <sub>2</sub>               | 0.26%       | 0.53%       | 20.37%            |
| Na <sub>2</sub> O              |             |             | 3.51%             |
| CaO                            | 0.12%       | 0.26%       | 3.07%             |
| Al <sub>2</sub> O <sub>3</sub> | 0.1%        | 0.18%       | 1.41%             |
| Fe <sub>2</sub> O <sub>3</sub> | 0.11%       | 0.23%       | 1.40%             |
| SO <sub>3</sub>                | 0.17%       | 0.39%       | 1.15%             |
| P <sub>2</sub> O <sub>5</sub>  | 0.06%       | 0.12%       | 0.63%             |
| MgO                            | 80 PPM      | 0.02%       | 0.36%             |
| ZnO                            | 0.03%       | 0.05%       | 0.33%             |
| K <sub>2</sub> O               | 0.01%       | 0.02%       | 0.23%             |
| TiO <sub>2</sub>               | 0.02%       | 0.03%       | 0.15%             |
| Cl                             | 81 PPM      | 97 PPM      | 0.15%             |
| NiO                            | 58 PPM      | 0.01%       | 0.07%             |
| MnO                            | 28 PPM      | 62 PPM      | 0.03%             |
| SnO <sub>2</sub>               | 47 PPM      | 75 PPM      | 0.03%             |
| CuO                            | 0.04%       | 40 PPM      | 0.03%             |
| Cr <sub>2</sub> O <sub>3</sub> | 8 PPM       | 16 PPM      | 0.03%             |
| CeO <sub>2</sub>               | 23 PPM      | 41 PPM      | 0.02%             |
| Bi <sub>2</sub> O <sub>3</sub> | 17 PPM      | 28 PPM      | 0.02%             |
| ZrO <sub>2</sub>               | 12 PPM      | 16 PPM      | 0.02%             |
| BaO                            | 19 PPM      | 21 PPM      | 0.02%             |
| Y <sub>2</sub> O <sub>3</sub>  |             | 16 PPM      | 87 PPM            |
| SrO                            | 4 PPM       | 7 PPM       | 73 PPM            |
| PbO                            |             | 5 PPM       | 59 PPM            |
| Br                             | 8 PPM       | 8 PPM       | 17 PPM            |
| As <sub>2</sub> O <sub>3</sub> |             |             | 7 PPM             |
| Nb <sub>2</sub> O <sub>5</sub> |             | 2 PPM       | 7 PPM             |
| Rb <sub>2</sub> O              |             |             | 5 PPM             |
| MoO <sub>3</sub>               | 3 PPM       |             |                   |

Note: used the 7 g of material; carbonizate obtained at  $T_{tr} = 600$  °C,  $t_{tr} = 30$  min.

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