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UTILIZATION OF SUGAR BEET PULP BY GETTING ACTIVATED CARBON

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Abstract. The formation and properties of sugar beet pulp have been analysed in the article. It has been proposed to use it as raw material to produce activated carbon. A better hydrophilicity of the obtained carbon in comparison with the samples of industrial production has been shown. Structural features and a porous structure of carbon made of sugar beet pulp have been analysed by X-ray diffraction and small angle scattering methods. It has been found that pores with average radii of inertia of 3.5 nm make the main contribution to the pore structure.

Key words: pulp, recycling, activated carbon, hydrophilicity, porous structure.

1. Introduction

Intensive development of industry and agriculture caused worsening of environmental problems. One of the important directions of the environment cleaning systems development is the elaboration of cheap and effective adsorbents. However, the materials used for these purposes now have high price, complex technology of production and regeneration [1]. Today, waste from food and agricultural products processing is more often used for manufacturing adsorbents [1–4]. This direction is promising, since vegetable waste is not amenable to regeneration, but their stocks are constantly replenished. However, the waste from sugar beet processing for this purpose is hardly used. As an adsorbent, it is proposed to use only thermally modified filtration sediment – defecation residues, the effectiveness of which is shown in the work [5].

It is known [6] that sugar production is a complex material and energy consuming production; moreover, the quantities of raw materials and auxiliary substances are several times higher than the output of finished products. So on average, the production of 1ton of sugar consumes 8–10 tons of sugar beet, about 25–35 m^3 of water, 0.6 ton of limestone, 0.53 ton of standard fuel. With the average yield of sugar of 10–12 % by weight of the processed beet about 83 % of fresh sugar beet pulp, 5.4 % of molasses, 12 % of the filtration sediment, 15 % of conveyor wash sediment, up to 350 % of sewage, etc. are produced. Thus, the main solid by-product in sugar preparation is sugar beet pulp. The pulp contains pectin substances, cellulose, sucrose, nitrogenous compounds, etc. 35–40 % of it is used for livestock feed, 30 % of pulp is dried, and the rest often goes sour losing the great part of the fodder value and forming other kind of waste – sour beet pulp water [6].

The main directions of sugar beet pulp utilization at present is its usage as an active substance in the production of biogas, obtaining pectin concentrate, pectin glue and dietary fibres, nanocrystalline cellulose and as fuel of CHP of sugar plant [7–9]. However, these measures do not allow solving completely the problem of sugar beet pulp processing.

Therefore, the aim of this work was to investigate the hydrophilicity and structural characteristics of activated carbon obtained from sugar beet pulp.

2. Material and methods

Dried sugar beet pulp, ground to 5–8 mm size was used as a feedstock. For the pyrolysis of raw materials, a steel crucible placed in a tubular reactor made of stainless steel was used. Activation was done by means of water vapour, the carrier of which was argon. The duration of activation was 2 hours at the temperature of 800 ± 5 °C. The output of activated carbon was approximately 25–27 % of the initial mass of pulp. The received carbon samples were washed with distilled water and dried.

Photographing of the obtained carbon was performed using a scanning electron microscope with low vacuum chamber and a system of energy dispersive microanalysis REM-106. This microscope is designed for direct study of the surface relief of various materials in the solid phase and the determination of their elemental composition by X-ray microanalysis according to quanta energy of characteristic X-ray radiation in the regime of low and high vacuum.

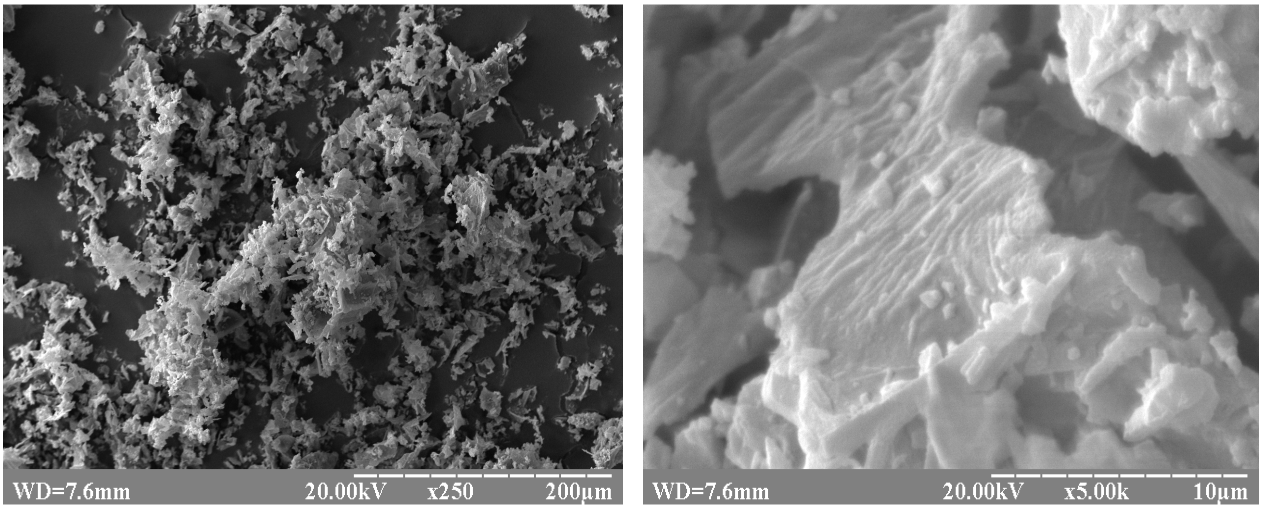
X-ray diffraction curves of carbon materials were obtained on diffractometer DRON-3 at Cu K_{α}-radiation ($\lambda = 0.1542$ nm), monochromatised by the reflection from the flat (002) of the single crystal pyrolytic graphite. The diffractograms were measured in continuous scanning of the detector in the range of angles of diffraction $2\theta = 5$–$100°$ (speed of the detector movement was 1.75 rpm, the sampling frequency of the output signal – 1 s^{-1}). To study the porous structure of the activated carbon, a method of small-angle X-ray scattering (SAXS) was used. The experiments were conducted in the mode of the X-ray beam passing through the sample. The radiation registration was performed in scan mode with a step of $0{,}05^{o}$, the exposure time $\tau = 125$ s.

The proportion of hydrophilic pores was determined by the amount of water absorbed by the sample, made of activated carbon, during a long-term exposure. The total volume of the pores available for the liquid was determined at an exposure in the liquid, wetting the activated carbon surface. Heptane was used for this purpose, and the calculation of hydrophilic-hydrophobic properties was made by the method [10].

3. Results and discussion

Activated carbon materials are the universal adsorbents, which are used in sugar manufacturing, either on their own [11] or mixed with other adsorbents [12] for the removal from the syrup of impurities, colloidal and other substances negatively affecting the quality of the product and its output. Given that in recent years more and more industrial consumers of sugar are starting to use white sugar of the I and II categories, the problem of improving the quality of the syrup as a determining factor affecting the quality of sugar, becomes extremely important. This makes us search for additional methods of purification of syrups and juice filtration in sugar production, in particular with the use of activated carbon.

Electronic photos of the obtained carbon are shown in Fig. 1, *a* and Fig. 1, *b*. As shown in Fig. 1, *a*), the form of obtained activated carbon particles is similar to the dried pulp particles. This suggests the possibility of pulp carbonization because the weight loss occurs due to the development of the porous structure.



a b

Fig. 1. Electronic photos of the activated carbon made of beet pulp with a scale of 200 µm (a) and 10 µm (b)

In Fig. 1, *b* large macro-pores are not observed, that most likely suggests formation of a well-developed mesoporous and microporous structure.during the activation

The Roentgen diffraction methods can be used for the analysis of such structural characteristics. In Fig. 2 the diffractogram of the obtained activated carbon samples is given. Such X-ray diffractogram is characteristic for quite strongly unidirectional carbons. The peak in the region of 40–50° corresponds to the superposition of two maxima at $2\theta \approx 44.5°$ and $2\theta \approx 51.9°$ due to the diffraction from the sample holder.

The main maximum in the region of 15–30° has an asymmetric shape, due to the influence of the maxima from the side of smaller scattering angles. We can assume that this sample has a nature of micro non-homogenous short-range order, which is manifested in the formation of minor amounts of graftobian clusters of 2–3 nm size distributed in a less ordered carbon phase. On the diffractogrammes of carbon materials studied in works [13, 14], in the area of angles of 15–30° a broad peak was observed, the presence of which the authors attribute to the presence in samples of small size domains, which have a coherent and parallel order of graphene layers. Thus, during the carbonization of sugar beet pulp at 800 ºC nanocrystalline graphite clusters are formed, which leads to an increase of graphitization degree of carbon material.

Since the received activated carbon is characterized by a misdirected microstructure, more detailed information on its porous structure can be obtained using SAXS data. In Fig. 3, ***a*** the curve of scattering intensity ***I(s)*** of the studied samples after the introduction of collimation amendment is shown. The resulting SAXS spectrum has the shape of a curve, which decreases monotonically in the entire range of angles.

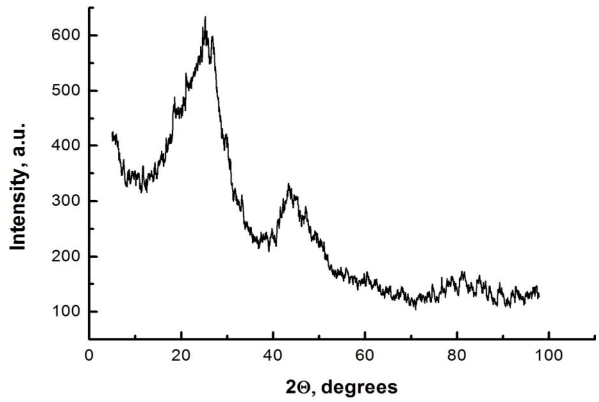
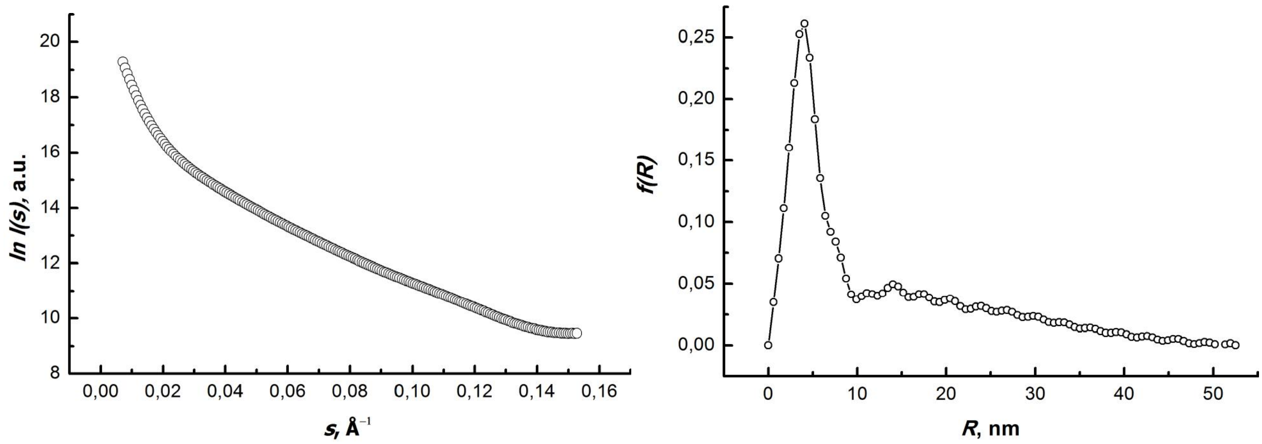


Fig. 2. The diffractiogram
of the activated carbon made of sugar beet pulp



a b

Fig. 3. SAXS range (a) and the pore distribution function according to sizes (b)
for the activated carbon obtained from sugar beet pulp

This behaviour indicates the chaotic distribution of the scattering heterogeneities (pores) and respectively the absence of correlation in their relative placement. Extrapolation of the curves of intensity to the value of the wave vector s = 0 was carried out using the Guinier law that allowed estimating the distribution function of pores according to the size. The calculations of this function were carried out using GNOM software package; the results are shown in Fig. 3, ***b***. It was found that for activated carbon, obtained on the basis of sugar beet pulp, the main contribution to the pore structure is made by the pores with average radii of inertia of 3,5 nm. In addition, the obtained carbon has a certain amount of mezzo-pores with a radius of 10–50 nm, allowing this carbon to adsorb not only individual molecules but also colloidal particles.

Determination of pores hydrophilicity is an important step in the study of the activated carbon properties, since the increase of hydrophilicity improves the efficiency of aqueous solutions adsorption. In the work [11], it was noted that as the reference carbon for the discoloration of the syrup, you can use the carbon of Norit type, and in [12] this carbon is among the major carbon adsorbents used in the sugar industry. Therefore, the comparison of hydrophilic properties of the obtained carbon made from sugar beet pulp was carried out with the traditional birch charcoal of BAU-A brand and carbon of Norit DLC30 Supra brand. In Table 1. the dependence of the fraction of hydrophilic pores compared to the free volume of the investigated carbon materials is shown.

Table 1

Hydrophilic-hydrophobic properties of activated carbon

Activated carbon types	Free space filling of heptanes, %	Free space filling of water, %	Hydrophilic pores, %	Hydrophobic pores, %
BAU-A	69	31	46	54
Norit DLC30 Supra	80	31	39	61
Activated carbon from sugar beet pulp	73	39	53	47

Table 1 shows that activated carbon sugar beet pulp has a lower porosity than the carbon of Norit type, but its hydrophilicity is almost 1.4 times higher. This allows absorbing a greater volume of aqueous solution, and, consequently, working more effectively as the adsorbent. Compared to BAU charcoal both the porosity and the hydrophilicity of the surface are bigger.

Therefore, the further study of adsorption properties of activated carbon based on beet pulp will allow determining additional features and its suitability to the solution of certain problems of both industrial and environmental areas. An additional modification, for example, by ultrasonic radiation of both output raw materials and already obtained activated carbon, will allow changing structural and adsorption characteristics of such carbon. Considering that improved pulp squeezing is one of the measures to increase the energy efficiency of sugar production [15], a fairly dried pulp can be used as an output raw material for carbonization without additional grinding and drying.

4. Conclusion

Thus, the analysis of literature data and experimental studies revealed that a by-product of sugar production – the sugar beet pulp – is a promising material for activated carbon obtaining, which can have a wide range of applications.

The hydrophilic-hydrophobic properties of the surface of the obtained activated carbon have been investigated. They proved to be the best for similar properties of commercial activated carbons. Other adsorption properties require an additional research, but now it is worth noting the prospects of using activated carbon made of sugar beet pulp as an adsorbent.

In most countries with a high level of industrial development, the rate of production of activated carbon is around 0.5 kg per person per year, while in Ukraine – less than 0.02 kg. Therefore, Ukraine regularly imports activated carbon from abroad for industrial and environmental applications. The development of technologies for low-cost high porous materials, based on agricultural products waste recycling, will give the opportunity not only to reduce the load on the environment, but also to meet the needs of the state in the qualitative activated carbon.

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