Study of the Material Composition of Carbon Black Obtained as a Result of MSW Thermolysis

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Abstract This paper presents the results of design and technological developments of resource-saving technologies for processing organic solid municipal waste (MSW), which allow obtaining various types of marketable products. Commercial products were obtained using this technology in the form of: carbon black, liquid hydrocarbon fuel, synthetic hydrocarbon gas. The proposed technology and technical means allow solving a whole range of scientific and technical, research, design, technological and environmental problems. The solutions take into account the conditions of existing production facilities and are aimed at eliminating existing technical and technological limitations in the field of resource and energy saving. The results of experimental studies on the determination of the material composition, structural-morphological and textural characteristics of carbon black, including X-ray phase analysis, laser diffraction, scanning electron microscopy, low-temperature nitrogen adsorption are presented. The results of the energy dispersion analysis indicate the predominance of carbon in the analyzed material, the presence of oxygen, calcium and silicon, and chlorine is also recorded. The product studied in this work was obtained according to the low-temperature thermolysis technology for processing organic solid municipal waste developed and implemented at the company "Ecotrans TC" LLC. Promising areas of application of carbon black obtained during the processing of organic MSW by the method of low-temperature thermolysis are identified.

Keywords Ecology · Solid municipal waste · Carbon black · Material composition Thermolysis technology · Particle size distribution \cdot Energy dispersive and X -ray phase analysis \cdot Scanning electron microscopy \cdot Low-temperature nitrogen adsorption

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S. V. Klyuev (ed.), *Digital Technologies in Construction Engineering,*

Lecture Notes in Civil Engineering 173, https://doi.org/10.1007/978-3-030-81289-8_22

1 Introduction

The increase in industrial production, as well as the steady increase in energy consumption, continuously increase the anthropogenic and environmental impact on the environment [[1](#page-7-0)].

The main method of solid municipal waste management in Russia is burial [\[2\]](#page-7-1). In 2017, the volume of MSW exported to landfill sites amounted to about 50.9 million tons, or 87% of the total volume of MSW exported [\[3\]](#page-7-2).

The author's team of researchers of BSTU named after V.G. Shukhov, with the participation of National Research University BelSU and engineering and technical employees of "Ecotrans TC" LLC, conducts scientific and technical developments and the introduction of resource-saving technology and special equipment for low-temperature thermolysis of organic waste into real production [\[4](#page-7-3)].

The proposed technology and technical means allow the processing of organic MSW of various morphological and physical composition. The method of low-temperature processing of organic MSW at a temperature of up to 500 °C allows obtaining high-quality products: carbon black, liquid hydrocarbon fuel and synthetic gas.

One of the promising areas of application of carbon black is its use as a sorbent for wastewater treatment.

The most popular industrial sorbent, at present, is absorbent carbon (AC). The consumption of AC in Russia has more than doubled over the past ten years [\[5](#page-7-4)].

Product samples were obtained during the thermolysis of organic MSW at a experimental-industrial thermolysis plant [\[6\]](#page-7-5).

Figure [1](#page-1-0) shows the scheme of low-temperature thermolysis with the production of carbon black, liquid hydrocarbon fuel and synthesis gas.

Fig. 1 Scheme of low-temperature thermolysis: 1—feeder; 2—thermolysis reactor; 3—combined conveying body; 4—gear motor; 5—solid product discharge device; 6—filter separator; 7 rectification column; 8—waste gas; 9, 10—gas-liquid burners; 11—liquid hydrocarbon fuel storage tank

The crushed "tails" of MSW or rubber waste are fed into the loading chamber of the feeder 1. Then the mass moves along the reactor 2 with the help of a combined conveying body 3, kinematically connected to the gear motor 4. Under the influence of temperature, the organic mass is decomposed into a solid carbon residue and a vapor-gas mixture. Through the solid product discharge device 5, the solid carbon residue is removed from the reactor. After passing through the filter separator 6, the steam-gas mixture enters the rectification column 7. In the latter, it is divided into industrial water and liquid hydrocarbons. The uncondensed hydrocarbon gas taken from waste gas 8 is burned in gas-liquid burners 9, 10. In the burners, a part of the liquid hydrocarbon fuel taken from the reactor heating tank 11 is also burned.

2 Materials and Methods

Earlier [[7\]](#page-7-6), researchers at BSTU named after V.G. Shukhov found that carbon black, a product of thermolysis of MSW, can be recommended as a promising sorbent for the purification of aqueous solutions. However, in the presented and other works, there is no information about the material composition of the obtained material. The purpose of these studies was to determine the chemical, phase, and granulometric compositions, structural, morphological, and textural characteristics of MSW thermolysis products.

2.1 Chemical Composition

During the analysis, an analytical system based on a high-resolution scanning electron microscope Nova NanoSEM was used. The device is equipped with an analytical energy-dispersion spectrometer from EDAX. The analysis was carried out at an accelerating voltage of 30 kV in the immersion mode (immersion in certain artificially formed conditions). The sample was placed in the magnetic field of the objective lens, which reduces aberration and achieves subnanometer resolution. This mode is suitable for the study of non-magnetic samples. The energy-dispersion spectrum of the organic component of the analyzed MSW thermolysis products is shown in Fig. 2.

The chemical composition of the organic component is shown in Table 1.

The chemical composition of the inorganic (lighter) part is shown in Table [2.](#page-3-1)

According to the results of chemical analysis, it can be assumed that the particles of composition No. 1 are characteristic of the products of thermolysis of organic substances, and composition No. 2— for building materials, possibly cement stone.

Fig. 2 Energy-dispersion spectrum of the organic part of MSW thermolysis products

Element	Mass, %	Atomic, %	Element	Mass, %	Atomic, %
C	66.84	79.43	K	1.04	0.38
\circ	14.35	12.8	Ca	6.30	2.25
Na	1.27	0.79	Ti	0.44	0.13
Mg	0.3	0.17	Cr	0.03	0.01
Al	0.97	0.51	Mn	0.05	0.01
Si	3.42	1.74	Fe	1.19	0.31
P	0.33	0.15	Cu	0.23	0.05
S	0.31	0.14	Zn	0.23	0.05
Cl	2.71	1.09			

Table 1 The elemental composition of the organic component of MSW thermolysis products

Table 2 The oxide composition of the light inorganic part of the thermolysis products of solid municipal waste

Element	Mass, %	Atomic, %	Element	Mass, %	Atomic, %
Na ₂ O	6.29	6.90	CaO	27.73	33.61
MgO	1.91	3.21	TiO ₂	2.32	1.97
Al_2O_3	6.90	4.60	Cr_2O_3	0.12	0.06
SiO ₂	26.94	30.48	MnO	0.19	0.18
P_2O_5	2.95	1.41	Fe ₂ O ₃	4.90	2.08
SO ₃	2.79	2.37	CuO	0.79	0.67
Cl ₂ O	11.38	8.90	ZnO	0.81	0.67
K_2O	3.99	2.88			

2.2 Phase Composition

The phase composition was determined using the SmartLab (Rigaku) X-ray diffractometer. The X-ray tube is equipped with a Cu-anode, which gives K_a radiation (U = 45 kV, I = 200 mA). The specified diffractometer is equipped with a high-speed D/tex Ultra Hi Pix detector. The Bregg-Brentano pseudoparallel beam focusing is used. The X-ray powder diffraction pattern of MSW thermolysis products is shown in Fig. [3](#page-4-0).

The results of the phase composition of the analyzed material are presented in Table [3](#page-4-1).

2.3 Granulometric Composition

In this study, we used a laser particle size analyzer "Analysette 22 NanoTec" with a measurement range from 0.01 to 2000 microns, which allows determining the particle size distribution of the analyzed material in suspensions and aerosols. The results of the size distribution of the powder microparticles are shown in Fig. [4](#page-5-0) .

Fig. 3 X-ray powder diffraction pattern of MSW thermolysis products

Phase name	Formula	Spatial group	Card
			number
Amorphous carbon	C	143: P3	00-026-1077
Potassium magnesium	KMgH	$2: P-1$	01-078-1942
dihydrocarbonate	$(CO_3)_2(H_2O)_4$		
Halite	NaCl	$225:$ Fm-3 m	00-005-0628
Quartz	SiO ₂	152: P3121	01-085-0865
Calcite	Ca(CO ₃)	167: $R-3c$,	01-083-0577
		hexagon	

Table 3 Results of qualitative X-ray phase analysis of MSW thermolysis products

Fig. 4 Particle size distribution of MSW thermolysis products. 1— integral curve in coordinates Q3 (x) = f(μ m) (left scale)—each point on the curve indicates how much % of the sample has a particle size less than or equal to this one. 2—histogram in coordinates q3 (x) = $f(\mu m)$ (right scale) — the number of samples with a given particle size

2.4 *Textural Characteristics*

We determined the textural characteristics of MSW thermolysis products by the method of low-temperature nitrogen adsorption (BET method). The specific surface area and porosity parameters were evaluated using a TriStar II 3020 gas-adsorption analyzer manufactured by Micromeritics (USA).

The obtained nitrogen adsorption isotherms are shown in Fig. [5](#page-5-1). According to the classification of Brunauer adsorption isotherms [\[8\]](#page-7-7), the obtained isotherms belong to the IV type of isotherms that correspond to physical sorption. A characteristic feature of the type IV isotherm is the presence of a capillary-condensation hysteresis loop. According to the IUPAC classification, the obtained hysteresis loops belong to the H1 type.

Fig. 5 Isotherms of nitrogen adsorption and desorption by the test sample (The "X" axis is the relative pressure P/Po; the "Y" axis is the amount of adsorbed gas cm^3/g)

Specific surface area according to the five-point BET method	$0.1271 - 0.2281$ m ² /g
Pore volume at relative pressure $P/Po = 0.984479441$	$\vert 0.000288 - 0.001, 148 \text{ cm}^3/\text{g} \vert$
Pore size	$190.72 - 165.12$ Å

Table 4 Textural characteristics of MSW thermolysis products

Table [4](#page-6-0) shows the textural characteristics of MSW thermolysis products.

3 Results and Discussions

The XRD method revealed low-temperature trigonal quartz in the material under study, which confirms the assumption of the presence of quartz sand in MSW. The predominant ingredient in the MSW thermolysis product is amorphous carbon.

According to the chemical analysis data, it can be assumed that in the initial state during the transportation and storage of MSW, particles of cement stone, quartz sand, chalk, dust and other building materials that can give peaks characteristic of calcium and silicon could get into them.

Based on the energy-dispersion analysis, it is concluded that the analyzed material is dominated by carbon 66.84 wt.%. Oxygen, calcium and silicon are also present in large quantities. Chlorine was recorded at 2.71 wt.%. This amount of chlorine can be explained by the presence of impurity chlorine-containing substances in MSW. A significant content of calcium and silicon oxides was determined. Sodium and aluminum oxides are present.

When studying the granulometric composition, it was found that the average particle size of the analyzed material is 44.07 microns.

Analyzing the graphical dependences of the specific volume of pores on their size, it was found that most of the pores are represented by macropores, which corresponds to a carbon material. The specific surface area of the samples according to the five-point BET method is $0.1271 - 0.2281$ m²/g. These values correspond to the specific surface area of the coal before activation [\[9\]](#page-7-7).

The carbon black obtained by low-temperature thermolysis technology is in demand in various areas of industrial production. In particular, it is widely used in paint and varnish, polymer, rubber, electronic, and other industries [[10\]](#page-7-8).

4 Conclusion

1. A resource-saving technology and a technological complex for processing organic MSW were developed, and commercial products were obtained: carbon black, liquid hydrocarbon fuel, and synthesis gas.

- 2. A set of studies to determine the material composition of carbon black, including: X-ray phase analysis, laser diffraction, scanning electron microscopy, low-temperature nitrogen adsorption.
- 3. The material composition of the MSW thermolysis products, which is mainly represented by carbon (66.84 wt%), was studied. The light component is dominated by calcium oxides $(27.73 \text{ wt\%)}$ and silicon oxides $(26.94 \text{ wt\%)}$.
- 4. Promising areas of application of carbon black obtained during the processing of organic MSW by low-temperature thermolysis are identified.

Acknowledgements The work was carried out within the framework of project No. 10089447, the program of the REC "Innovative solutions in the agro-industrial complex", the scientific and production platform "Rational use of natural resources".

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