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Thermal-Oxidative Treatment to Control the Texture Characteristics of Carbon Black

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Abstract

The effect of high-temperature treatment (900 °C) of N339 carbon black in the presence of carbon dioxide, followed by low-temperature treatment (−5 °C) in a mixture of oxygen with ozone, on its properties was studied. As a result of complex oxidative action, a carbon material is formed with textural properties that are several times higher than the levels of values characteristic of the original carbon black. The oxygen content in the final material can vary from 19 to 35 wt. %, which suggests that it is promising for use as a high-performance pigment.

Keywords: carbon black, carbon dioxide, ozone, pore volume, specific surface area, functional groups

INTRODUCTION

Carbon black (CB) is a dusty or granulated carbon product composed of a set of aggregates, which are chemically bound globular particles with the turbostratic structure of graphene layers, and various oxygen-containing groups may be localised at the edge centres of these aggregates [1]. There may be both acidic (carboxylic, phenol, anhydride, etc.) and basic groups (pyrone, chromene and ketone) on CB surface, the content of specific groups depending on CB production method [1]. Channel-type CB contains about 7 wt. % and more oxygen mainly in the form of acid groups, furnace CB – only 0.5–3 wt. % oxygen in the form of both acidic and basic groups [1]. The furnace method of CB production is currently the main one, while the production of channel-type CB has been almost completely ceased due to economic and ecological reasons.

However, some branches of industry require CB with high oxygen content. For example, this is necessary for the production of rubber for special purposes with increased photostability, antioxidant, gas barrier, vibroacoustic and strength parameters [2], and for the development of high-quality pigments for the polygraphic industry [3]. The presence of oxygen-containing groups on the surface of pigment CB grades simplified dispersing in polar solvents and enhanced the stability of water-based colouring compositions [3].

In this connection, the technologies of oxidative treatment of furnace CB are under active development for the purpose of increasing the amount of oxygen-containing functional groups on its surface.

The traditional methods of carbon material surface modification are gas and liquid phase treatments with various oxidisers. Oxidative treatment at low temperatures usually results only in the changes of the surface functional composition [4–10], while treatment at high temperatures (above 800 °C) [11–15] may lead to

changes in the size of crystallites and carbon globules, surface area and porosity.

Among liquid-phase oxidisers, the most widespread ones are mineral acids [7], potassium permanganate [9, 10], hydrogen peroxide [6, 8]. However, the use of these oxidisers requires numerous stages of washing from side products. The use of hydrogen peroxide brings about the danger of explosions.

Oxidation in gaseous media, in particular in the presence of ozone [4, 5, 11, 16–19], quite contrary, appears to be a promising method to form the functional coating of CB surface in the absence of the side products of oxidation. It has been established that CB ozonation in fluidized bed is accompanied by an increase in the amount of carboxylic and quinone groups on the carbon surface by a factor of 15 and 10, respectively [5].

It is known that the treatment of CB with ozone in low concentration (up to 8 g/m^3) at room temperature involves only surface oxidation reactions with the formation of oxygen-containing groups on the reactive centres of the first graphene layer: at the edges of graphene planes and at fullerene-like regions [17, 18]. Treatment with high ozone concentration (more than 14 g/m^3) at increased temperature (60 °C), in addition to the formation of functional groups, caused the formation of partially burnt surface [17].

It has been determined that the oxidative ozone treatment of channel-type CB may allow reaching 24–33 wt. % oxygen content on the surface [5, 17]. In addition to the high oxygen content, this CB was characterised by large specific surface area (not less than 320 m^2/g), and thus it was recommended as a component of ink and polygraphic colours [5, 17]. It is known that polygraphic printing requires CB that not only contains large amounts of oxygenated groups but also possesses high colouring capacity, which is mainly due to the large specific surface area. In our work, we consider the possibility to obtain the product based on furnace CB, close in its textural characteristics to the pigment based on channel-type CB.

The goal of the present work was to study the effect of the method of thermal-oxidative treatment on the structure and physicochemical properties of furnace CB by the example of N339 grade.

EXPERIMENTAL

The object of investigation was CB of N339 grade manufactured by Omsktekhuglerod LC (Russia). The physicochemical properties of CB N339 are listed in Table 1.

Thermal-oxidative treatment of CB was carried out in two stages. At the first stage, CB was treated according to the procedure described in [12] in carbon dioxide flow at a temperature of 900 °C for 5 h (sample N339gas). At the second stage, the carbon material N339gas was treated with the oxygen-ozone mixture with ozone content 50 g/m^3 in a quartz reactor 10 mm in diameter at a temperature of (−5)±2 °C for 45 min in the fluidized bed (sample CM1), in a steady layer 20 mm thick 20 mm (sample CM2). The ozone-oxygen mixture passed through a CB layer with a flow rate of 0.05 m^3/h. The mass of the CB portion in the reactor was 0.5 g.

The mixture of ozone with oxygen was formed with the help of an OGVK-01 ozonising device (MELP, Russia). The first-grade oxygen corresponding to GOST 5583-78 was used for ozone generation. Ozone concentration at the reactor outlet was measured with an IKO-1 ozone meter (Tsyiklon-Pribor, Russia). Ozone treatment was considered to be complete in cases when ozone concentration in the input gas flow became equal to ozone concentration in the output flow after interaction. Temperature of CB portion in the reactor was measured with a chromel-alumel thermocouple according to GOST R 8.585-2001.

The X-ray diffraction (XRD) of initial and modified CB samples was carried out with a powder X-ray diffractometer D8 Advance (Bruker, Germany) in CuK_α-radiation. The samples were scanned within the angle range 10–80° over 2θ, with scanning step 0.05°, and 2 s exposure in each point. The recorded diffraction patterns were interpreted using the powder diffraction database ICDD PDF-2. It is known that the major structural parameters of carbon are the average sizes of coherent scattering regions (CSR), which are associated with graphite-like crystallites, – L_a (in the direction along graphene networks) and L_c

TABLE 1

Physicochemical parameters of carbon black, N339 grade

Parameter	Value
Total specific surface area (NSA), m^2/g	91
External specific surface area (STSA), m^2/g	86
Absorption of dibutyl phthalate (A_{DBP}), cm^3/100 g	122
pH of aqueous suspension	8.3
Oxygen content (ω_O), wt. %	0.8

(in the direction perpendicular to these networks), and interplanar spacing (d_{002}). The method to calculate interplanar spacings and crystallite sizes along the (002) plane is described in [20]. The data were processed and the peaks were decomposed using Fityk software [21].

Determination of the structural features of CB (from dibutyl phthalate absorption (A_{DBP})) was carried out in agreement with the ASTM D2414 standard. The method involves determination of the volume of dibutyl phthalate (DBP) absorbed in the CB sample under test during mixing in the mixer chamber of the absorption meter.

The morphology of CB particles was studied using transmission electron microscopy (TEM) in agreement with the ASTM D3849 standard using a JEM-2100 transmission microscope (Jeol, Japan). Computer processing of electron microscopic images was carried out with the help of the software package Digital Micrograph.

The total oxygen content (ω_O, wt. %) was determined with the help of a Vario EL Cube elemental analyser (Elementar Analysensysteme GmbH, Germany) in output gases formed during sample pyrolysis in a quartz tube filled with the catalyst, at a temperature of 1170 °C. Oxygen was quantitatively transformed into carbon monoxide (CO) during this process, with subsequent desorption and detection of CO with the thermal-conductivity detector. The parameters of analysis for oxygen content are: the temperature of pyrolysis tube – 1170 °C; the temperature of CO adsorption column during adsorption – 40 °C; the temperature of CO adsorption column during desorption – 260 °C; helium flow rate – 230 cm^3/min. Oxygen concentration was calculated relying on the calibration curve plotted with respect to the standard substance, which was benzoic acid (CAS No. 65-85-0).

The quantitative composition of functional oxygen-containing groups on the surface of CB samples was determined according to Boehm's method [22].

The acidity (pH) of the aqueous suspension of CB was determined with the help of pH-meter pH-150MI (Izmeritelnaya Tekhnika, Russia) with an ESK-10603/7 combined electrode.

The IR spectra of the samples were recorded with an IR-Prestige-21 spectrometer (Shimadzu, Japan) within the spectral range of 750–1800 cm^{-1} with the resolution of 4 cm^{-1}.

The total specific surface area (NSA, m^2/g) of initial and modified CB samples was calculated according to the BET procedure from the isotherm of low-temperature nitrogen adsorption in the negative pressure region, P/P_0 from 0.05 to 0.3. The isotherms of nitrogen adsorption were recorded with the help of a Gemini 2380 analyser (Micromeritics, USA). Specific external surface area (STSA, m^2/g) was estimated from the static layer thickness (t, nm) of CB. The value of *STSA* was calculated from the plotted dependence of the volume of nitrogen adsorbed by the sample under study on the static layer thickness t for the standard carbon black sample within the relative pressure range P/P_0 from 0.2 to 0.5. The total specific pore volume (V_Σ, cm^3/g) was determined from the maximal amount of nitrogen adsorbed at the relative pressure P/P_0 = 0.96. Micropore volume (V_{mi}, cm^3/g) was determined using the comparative t-method [23].

RESULTS AND DISCUSSION

The TEM images of initial CB N339 and modified samples CM1 and CM2 are shown in Fig. 1.

The initial CB N339 is represented by globular particles with the prevailing turbostratic structure of graphene layers (see Fig. 1, *a*). The structure of CM1 sample (see Fig. 1, *b*) is almost the same as the structure of the initial CB sample, but small pits appear on the surface of the globules. According to data reported in [15], these pits may be the throats of micropores. The CM2 sample possesses a more rarefied structure of graphene layers than the initial CB sample and CM1 sample. The surface of CM2 is more rough in comparison with the initial CB and CM1 sample, and the pits on the globule surface become more noticeable (see Fig. 1, *c*). The average size of the globules of CM1 remains almost unchanged during modification and is equal to 24 nm, in comparison with 25 nm for initial CB. The size of CM2 globules decreases substantially, to 18 nm.

It is known from literature data that the size of CB globules may change both at the stage of treatment in carbon dioxide [13, 15] and at the stage of treatment with ozone [17]. According to the data reported in [15], during the treatment in CO_2 up to 1000 °C inclusive, substantial changes of CB globule size are usually typical for burn-off about 80 wt. % and more; during ozone treatment – at high temperatures and at the same time high ozone concentration in the ozone-containing flow [17]. A decrease in the size of carbon globules in CM2 sample is likely to be caused by burn-off at the stage of ozone treatment, due to

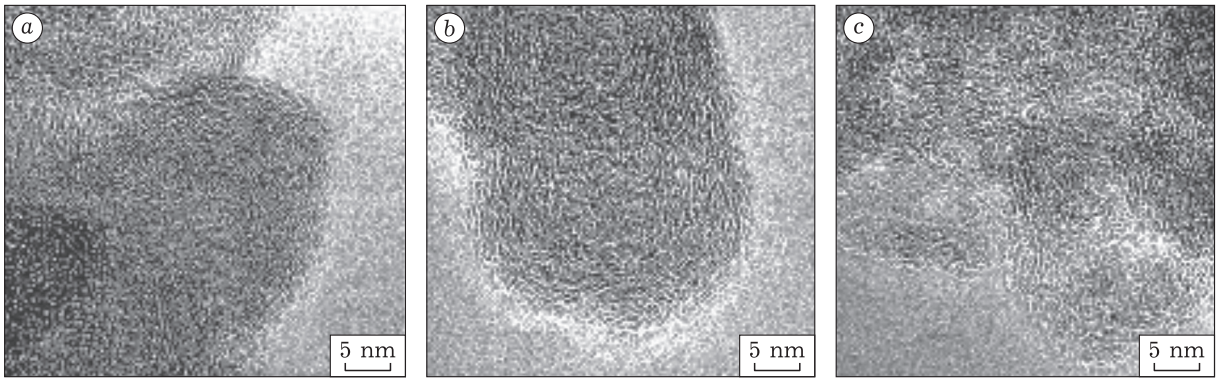

Fig. 1. TEM images of initial carbon black of N339 grade (*a*) and carbon black after gasification and ozonation stages: CM1 (*b*), CM2 (*c*).

CB self-heating in the steady layer in the ozone-oxygen flow up to 250 °C.

The results of XRD show that no substantial changes of microstructural parameters are observed during thermal-oxidative treatment: for initial N339 and modified CM1 sample, $L_a \approx 5.6$ nm, $L_c \approx 2$ nm, $d_{002} \approx 0.366$ nm. The size of crystallites in the layer plane of CM2 sample decreases substantially to $L_a \approx 4.4$ nm. The height of carbon packets $L_c \approx 2$ nm for all samples, and interlayer spacing remains almost unchanged ($d_{002} \approx 0.366$ nm). This fact confirms graphene layer burn-off in CM2 sample during its treatment.

The results obtained by means of TEM and XRD illustrate the fact that the structure of globules in CM1 sample remains almost unchanged during thermal-oxidative treatment of CB in carbon dioxide and in ozone within the chosen temperature range, while the structure of globules in CM2 changes substantially.

Then changes in the functional composition of the carbon surface during thermal-oxidative treatment were investigated.

The data presented in Table 2 provide evidence that the initial furnace CB of N339 grade contains carboxylic and phenol groups, 0.22 and 0.33 μg-equiv/m^2, respectively. Initial CB is characterized by pH of the aqueous suspension equal to 8.3, and oxygen content in this CB is less than 1 wt. %. It is known from the literature data [1] that the weakly alkaline pH of the aqueous suspension of furnace CB may be caused by the presence of basic functional groups (pyrone, chromene and ketone) on the CB surface in addition to acidic groups (phenol and carboxylic).

Gasification procedure, carried out in the atmosphere of carbon dioxide at 900 °C, leads to thermal destruction of all functional groups on the CB surface, which, in turn, promotes a decrease in pH of the aqueous suspension to 6.9 (see Table 2).

During the second stage of carbon material N339gas modification with the ozone-oxygen mixture, an increase in oxygen content is observed in CM1 and CM2 samples from 0.3 (N339gas) to 18.8 and 34.9 wt. %, respectively (see Table 2). According to the data of acid-base titration, both samples CM1 and CM2 contain carboxylic and phenol groups at a level of 1.97–2.53 and 7.25–8.00 μg-equiv/m^2, which promotes a sharp decrease in pH of the aqueous suspension from 6.9 (N339gas) to 2.0 and 1.6, respectively. In addition to carboxylic and phenol groups, some amount of oxygen may be present on the carbon surface in the form of other groups (for example, lactone, carbonyl, anhydride, *etc.*). The presence of oxygen-

TABLE 2

Functional composition of the surface of initial carbon black N339 before and after gasification and ozonation stages

Sample	pH of aqueous suspension	Content of carboxylic groups, μg-equiv/m^2	Content of phenol groups, μg-equiv/m^2	ω_O*, wt. %
N339	8.3	0.22	0.33	0.8
N339gas	6.9	0	0	0.3
CM1	2.0	2.53	1.97	18.8
CM2	1.6	7.25	8.00	34.9

* The oxygen content.

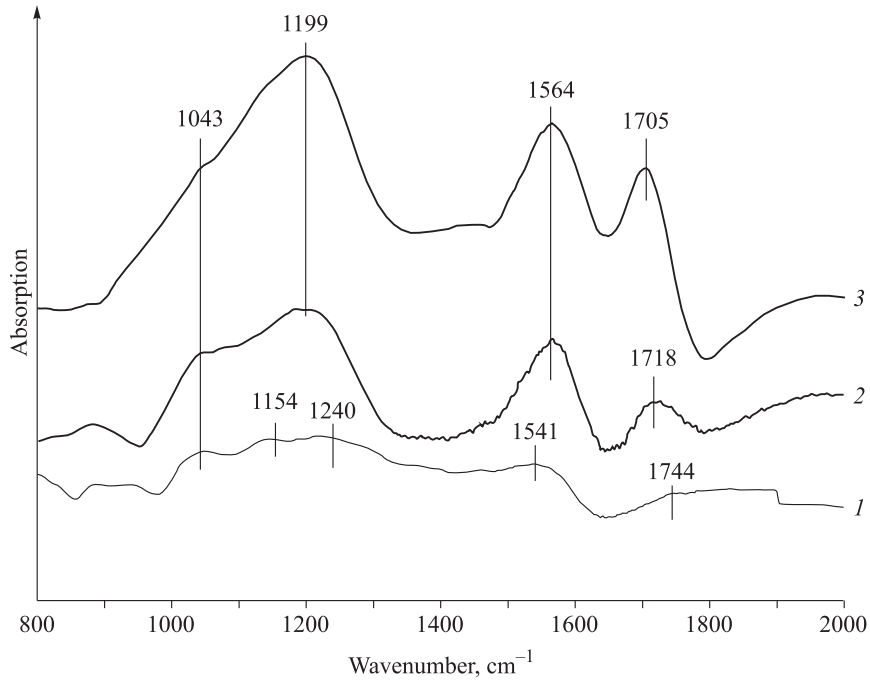

Fig. 2. IR spectra of initial carbon black of N339 grade (*1*) and samples after gasification and ozonation stages: CM1 (*2*), CM2 (*3*).

containing groups on the carbon surface was confirmed by the results of IR spectroscopy (Fig. 2).

One can see that the initial sample N339 exhibits weakly pronounced absorption bands (a. b.) within the spectral range 1680–1780 cm^{-1}, corresponding to the stretching vibrations of C=O bonds in carboxylic and carbonyl groups; a. b. in the region of 1000–1250 cm^{-1}, corresponding to the stretching vibrations of C–O bonds in phenol, lactone, and ether groups. After thermal-oxidative treatment, the intensity of a. b. increases substantially in the regions of 1680–1780 and 1000–1250 cm^{-1}, related to C=O and C–O groups, respectively, especially for CM2 sample. In addition, the IR spectra of all samples exhibit a. b. in the region of 1520–1600 cm^{-1}, corresponding to the stretching vibrations of C=C bonds in aromatic structures.

In addition to functional groups localised at the edge centres of external graphene layers, the formation of oxygen-containing groups is also possible inside the CB globules under the severe conditions of thermal-oxidative treatment (the synthesis of CM2 sample). The possibility to form narrow channels in the external graphene layers as a result of ozone reaction with CB is described in [17]; as a consequence, the internal defects of carbon globules become accessible. These internal defects may be inaccessible for evaluation by means of low-temperature nitrogen adsorption, and functional groups localised on these defects may be inaccessible for detection by titration. It is assumed that some amount of oxygen on the CM2 sample is present at the edge centres of external graphene layers, while some amount is localised at the internal defects of the globules.

For CB N339 before and after gasification and ozone treatment stages, texture characteristics and absorption of DBP were evaluated (Table 3). The texture characteristics were determined from nitrogen adsorption-desorption on CB samples before and after treatment.

According to the IUPAC classification, all isotherms (Fig. 3) recorded in the study correspond to the II type with H3 hysteresis loop. For many gases, the adsorption branch of II type isotherms points to their reversible adsorption on non-porous or macroporous solids. The shape of H3 hysteresis loop may depict either capillary processes of condensation/evaporation in non-rigid porous aggregates of adsorbents or the processes taking place in the network of pores incompletely filled with the condensate, in the presence of micropores.

A more detailed comparison between the recorded adsorption-desorption isotherms of the samples under investigation (see Fig. 3) shows that they differ from each other in the extension and width of the hysteresis loop. In the initial

TABLE 3

Texture parameters and the values of dibutyl phthalate (DBP) absorption on initial carbon black N339 before and after gasification and ozonation stages

Sample	V_{Σ}, cm^3/g	V_{mi}, cm^3/g	NSA, m^2/g	STSA, m^2/g	A_{DBP}, $cm^3/100$ g
N339	0.21	0	91	86	122
N339gas	0.55	0.13	411	177	126
CM1	1.03	0.16	482	124	120
CM2	0.48	0.08	375	189	88

Notes. 1. V_{Σ} and V_{mi} are specific total pore volume and micropore volume, respectively. 2. For designations, see Table 1.

sample N339, the hysteresis loop is the least pronounced, and micropores are absent.

For N339 gas sample, the hysteresis loop becomes broader due to the formation of new pores during thermal treatment in CO_2 at 900 °C. Indeed, the volume of micropores increases by two orders of magnitude from 0 to 0.13 cm^3/g, specific pore volume increases more than 2 times, from 0.21 to 0.55 cm^3/g (see Table 3). The total specific surface area NSA increases more than 4 times (from 91 to 411 m^2/g), and the external specific surface area STSA increases more than twice (from 86 to 177 m^2/g). However, the structural pattern of gasified CB, characterised by A_{DBP}, in spite of substantial increase in texture parameters, remains almost at the level of initial CB. A similar development of porosity and an increase in specific surface area are observed as a result of high-temperature treatment of other CB grades with carbon dioxide, which is due to burning of some amount of amorphous carbon [11, 13, 15].

For CM1 sample, a hysteresis loop in the high-pressure region becomes higher than that for the N339gas sample (see Fig. 3), which may be connected with pore size redistribution as a result of treatment with ozone. One can see in Table 3 that the ozone treatment of gasified sample N339gas in the fluidized bed (the synthesis of CM1 sample) leads to further increase in total pore volume from 0.55 (N339gas) to 1.03 cm^3/g (CM1), micropore volume from 0.13 (N339gas) to 0.16 cm^3/g (CM1), and total specific surface area from 411 (N339gas) to 482 m^2/g (CM1). The parameter of DBP absorption on CM1 sample does not change and remains at a level of 126–120 $cm^3/100$ g (see Table 3). The joint consideration of the obtained data with the results of TEM and acid-base titration allows us to assume that the fluidized-bed ozone treatment of carbon material involves the formation of many cracks and slits on the surface of globules, which, in turn, promotes and increase in micropore volume and the formation of oxygen-containing groups only at the edge centres of external graphene layers.

For CM2 sample, hysteresis loop is wider in the relative pressure region P/P_0 = 0.5–0.8 than that for CM1 sample (see Fig. 3), which may be explained by pore size redistribution during treatment with ozone, due to burn-off at some contacts in coarse CB aggregates and their decomposition into smaller aggregates, which results in a decrease in the amount of interparticle pores. Indeed, the parameter of DBP adsorption depicting the structural pattern of the aggregate decreases substantially from 120 (N339gas) to 88 (CM2) $cm^3/100$ g (see Table 3). Specific pore volume at P/P_0 = 0.96, micropore volume and a total surface area decrease from 0.55 to 0.48 cm^3/g, from 0.13 to 0.08 cm^3/g and from 411 to 375 m^2/g, respectively (see Table 3). Comparison with the data of TEM allows us to assume that ozone treatment in fluidised bed (the synthesis of CM2 sample) proceeds not only at the edge centres of external graphene layers but also promotes further destruction of the aggregates and the formation of tighter packed particles of smaller size.

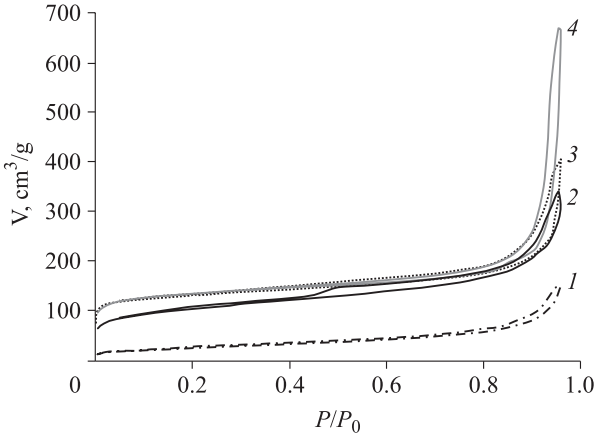

Fig. 3. Isotherms of nitrogen adsorption-desorption on initial carbon black of N339 grade (*1*), carbon black after gasification and ozonation stages: CM2 (*2*), N339gas (*3*), CM1 (*4*).

Thus, the developed two-stage thermal-oxidative treatment of initial CB in the fluidised bed allows obtaining carbon material with a total specific surface area exceeding the specific surface area of commercial CB by a factor of 1.5–5. Oxygen content in the sample increases substantially and reaches 18.8–34.9 wt. %, which allows us to recommend this material as a component for colouring compositions similarly to [5, 17]. It is shown that the structural pattern of the carbon material may be regulated at the stage of treatment with ozone.

CONCLUSION

Modification of the surface of CB N339 was carried out under the conditions of high-temperature treatment (900 °C) in the presence of carbon dioxide, and then the low-temperature (−5 °C) treatment in a mixture of oxygen with ozone in fluidised or fixed bed.

It is shown that at the first stage of high-temperature treatment of CB in the presence of carbon dioxide, thermal destruction of all functional groups on the surface of initial CB N339 is observed. This is accompanied by an increase in specific pore volume and the volume of micropores from 0.21 to 0.55 cm^3/g and from 0 to 0.13 cm^3/g, respectively, which is connected with burning of a part of amorphous carbon.

It is determined that the low-temperature treatment of CB in a mixture of oxygen with ozone in the fixed bed leads to a decrease in the average globule size from 25 to 18 nm and DBP absorption parameter from 120 to 88 $cm^3/100g$, which is caused by burning the contacts between globules in the aggregates and globule burn-off. This promotes destruction and the formation of tighter packed globules of smaller size, which causes a decrease in micropore volume from 0.13 to 0.08 cm^3/g and total specific surface area from 411 to 375 m^2/g in comparison with CB after high-temperature treatment in carbon dioxide. Ozone treatment in the fixed bed causes the formation of carboxylic and phenol groups in the amount of 7.25 and 8.00 μg-equiv/m^2, respectively, both at the edge centres of external graphene layers and at the inner defect-bearing sites.

It is established that low-temperature treatment of CB in a mixture of oxygen with ozone in fluidised bed, unlike for the fixed bed, allows conserving the average globule size and the structural pattern of aggregates at he level of initial CB N339. In this case, the total pore volume and micropore volume parameters were observed to increase from 0.55 to 1.03 cm^3/g and from 0.13 to 0.16 cm^3/g, respectively. The obtained results allow us to state that the formation of carboxylic and phenol groups in the amounts of 2.53 and 1.97 μg-equiv/m^2, respectively, proceeds mainly at the edge centres of external graphene layers.

Thus, a material possessing developed texture and high oxygen content (19–35 wt. %) has been obtained through thermal-oxidative treatment of furnace CB. The obtained carbon material may be promising for use as a highly efficient pigment component of ink for polygraphic industry.

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