

# Surface modification and functionalization of meso-sized carbon tubes

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**Abstract.** In this article, the specific surface area ( $S_{sol}$ ) and pore size distribution were determined by BET absorption at a low temperature of 77K. The purpose of the work is to determine the level of functionality and surface modification of meso-sized carbon tubes. According to the BET model, the specific surface area was calculated and the BJH model of the pore size distribution in the material was used. The morphology of mesoporous carbon was studied using transmission electron microscopy and scanning electron microscopy. Differential scanning calorimetry and thermogravimetric analysis of the synthesis products were performed in the air to determine the leaching conditions. According to thermogravimetric data, the amount of unstructured forms of meso-sized carbon in the samples of meso-sized carbon tubes with a multi-walled mesoporous structure decreases several times, from 2.0-8.0% to 0.5-2%. Electron microscopy confirmed the effectiveness of air annealing. A comparison of different methods of processing meso-sized carbon tubes with a multi-walled mesoporous structure shows the advantages of using nitric acid as a modifying agent, since it promotes the formation of structural fragments of the most molecules among the oxidizing agents used, which are easy to use and by simple washing with water is easily removed from the system without absorption in meso-sized carbon tubes with a multi-walled mesoporous structure. Meso-sized carbon tubes with a multi-walled mesoporous structure processed in nitric acid, as well as the first ones, showed stability up to 450-500 °C when heated in air. Their structural and electronic characteristics, calculated from Raman spectra and diffraction data, remained virtually unchanged. The method of X-ray diffraction of oxidized samples indicates almost complete removal of the metal.

## 1 Introduction

It is known that the replacement of traditional fuels with hydrogen alternative fuel mixtures can increase efficiency and reduce the emissions of gas-piston thermal power plants, autonomous power plants, cars and other types of transport [1]. Conventional methods of

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hydrogen production, such as methane steam reforming, coal gasification, or water electrolysis, currently cannot ensure the economic feasibility of using hydrogen in power [2]. Therefore, the urgent task of developing small-scale hydrogen energy today is to develop a technology that allows obtaining hydrogen fuel with a high concentration of hydrogen without carbon monoxide admixture. This method is catalytic pyrolysis of hydrocarbons [3].

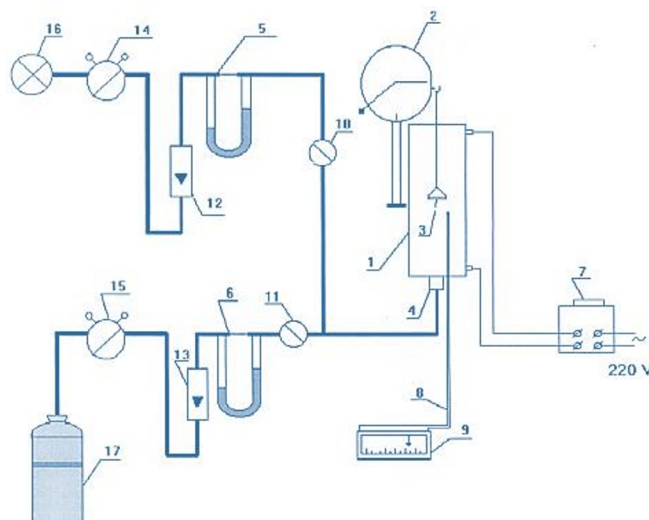
Carbon nanostructures such as fullerenes [4], nanotubes [5-7], and hybrid graphene/carbon nanotubes [8] have many excellent mechanical, optical, electrical, and thermal properties that make them useful in many industrial applications, especially in nanotechnology, make it a promising material for use in the field. Many studies have successfully attempted to precisely control the physical shape of carbon nanotubes [5,6].

In particular, the influence of various growth parameters, such as diameter, number of graphene layers, defect density, length, etc., on the properties of the resulting nanotubes was studied [9, 10]. In addition, self-assembled graphene flakes of mesoporous carbon tubes [11], chemical evaporation of graphene layers in mesoporous carbon tubes [13-14] and simple mechanical mixing, hybrid (graphene/carbon nanotubes) many approaches have been proposed to prepare the structures.

In this work, the process of obtaining hydrogen by catalytic pyrolysis of methane according to the  $\text{CH}_4 \rightarrow 2\text{H}_2 + \text{C}$  reaction on different catalysts is considered. In addition to hydrogen, a valuable product - nanofiber carbon - is formed during the reaction, which can be used, for example, as filler for composite materials, and as a reagent for the synthesis of refractory materials. Today, in addition to obtaining mesocarbons from natural gas, the scientists of the world can obtain ethylene from natural gas in one step, to obtain mono- and bicyclic aromatic hydrocarbons by aromatizing methane, to obtain mono- and bicyclic aromatic hydrocarbons from propane-butane fractions and petroleum satellite gases, to obtain synthesis gas from methane and based on it the reactions of obtaining high-octane methanol and dimethyl ether, and obtaining lower molecular weight olefins from methanol and dimethyl ether are of interest [15-25]. Mesoporous carbon and mesoporous zeolites act as catalysts in the above-mentioned reactions.

## 2 Experimental part

To study the kinetics of obtaining mesoporous carbon, a flow reactor with an inner diameter of 60 mm and a length of 400 mm, filled with a 100 mm nozzle for heating the feed gas mixture at once, and equipped with a nozzle for gas injection from the bottom side, was assembled. The diagram of the reactor is shown in Figure 1 below.



**Fig. 1.** Schematic of an experimental device for studying the kinetics of mesoporous carbon formation: 1 – reactor; 2 – torsion balance VT-500; 3 – a boat with a catalyst; 4 – nozzle for entering the gas phase; 5, 6 – diffmanometer; 7 – laboratory autotransformer; 8 – chromel-aluminum thermocouple; 9 millivoltmeter; 10, 11 – one-lane crane; 12, 13 – rotameter RM-0.25G; 14, 15 – pressure reducers; 16, 17 – gas source.

## 2.1 Determination of pore size and the comparison surface

The Comparison surface area ( $S_{sol}$ ) and pore size distribution were determined by BET absorption at a low temperature of 77K. To determine the relative surface area, 400 mg of material was placed in an ampoule in the analyzer, a vacuum was created with a standard pump, and a graph representing the relationship between the amount of absorbed gas and the relative pressure of a certain amount of nitrogen adsorption gas at a temperature of 77°K during the measurement process was constructed, according to the BET model using the instrument software, the specific surface area was calculated and the BJH model of the pore size distribution in the material was used.

Purification of amorphous meso-sized carbon impurities from meso-sized carbon tubes with multi-walled mesoporous structure. The meso-sized carbon tubes with the original multi-walled mesoporous structure were cleaned of meso-sized carbon impurities by calcination in air at a temperature of 350-400 °C determined by thermogravimetric data for 2 hours.

## 3 Results and discussion

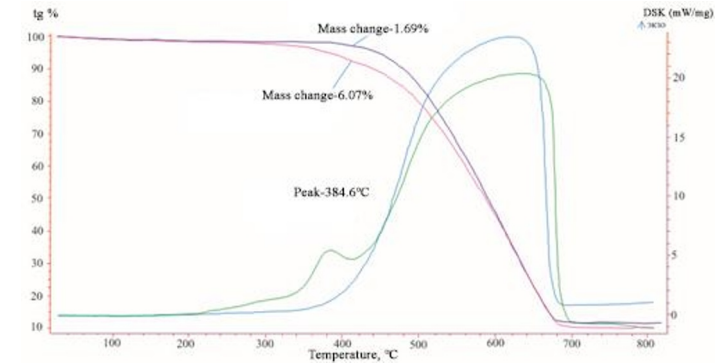
### 3.1 Cleaning and surface modification of meso-sized carbon tubes with multi-walled mesoporous structure

According to transmission (illumination) electron microscopy and scanning electron microscopy data, the main impurities in the synthesis products of pipe samples with a pore size of 2÷50 nm are amorphous and partially structured meso-sized carbon inclusions and metal with a pore size of 2÷50 nm consists of particles. The simplest and most acceptable methods are air burning to remove meso-sized carbon impurities, because it is known that amorphous meso-sized carbon impurities are oxidized at lower temperatures than meso-

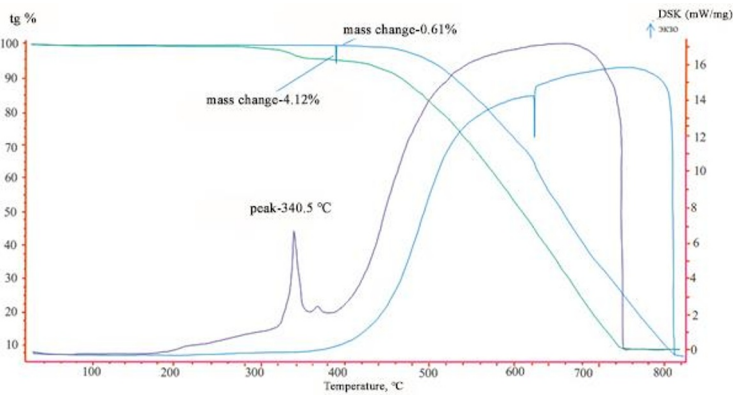
sized carbon tubes with a porous structure with a size of  $2\div 50$  nm, and treatment with strong mineral acids metal can be removed through.

**3.2 Surface cleaning and modification of meso-sized carbon tubes with multi-wall mesoporous structure**

Differential scanning calorimetry and thermogravimetric analysis of the synthesis products were performed in the air to determine the leaching conditions. In the thermogravimetric curves of meso-sized carbon tubes with a multi-walled mesoporous structure (Figures 2 and 3), two stages of mass loss were observed: the first, up to  $400^{\circ}\text{C}$ , corresponds to the combustion of amorphous forms of meso-sized carbon; the second corresponds to the combustion of meso-sized carbon tubes with a multi-walled mesoporous structure up to  $700^{\circ}\text{C}$ .



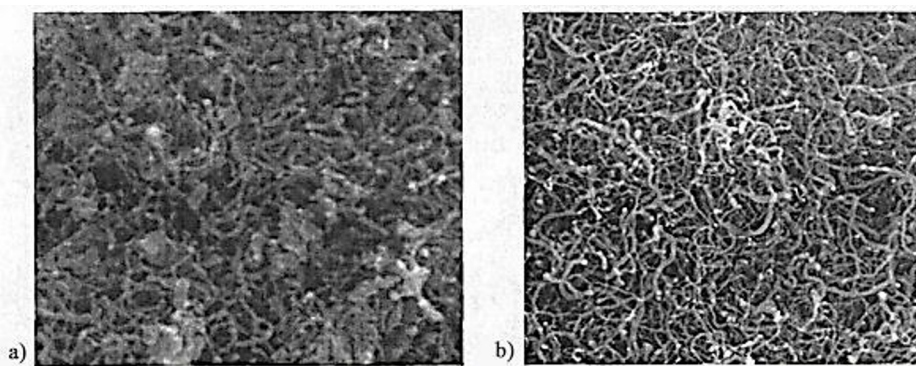
**Fig. 2.** Differential scanning calorimetry and thermogravimetric analysis of pure and burned-in air cylinders



**Fig. 3.** Differential scanning calorimetry and thermogravimetric analysis of pure and burned-in air conical-multiwall mesoporous meso-sized carbon tubes

Differential scanning calorimetry with maximum temperatures of  $340^{\circ}\text{C}$  and  $630^{\circ}\text{C}$  (conical-multiwalled mesoporous carbon tubes) and  $380^{\circ}\text{C}$  and  $650^{\circ}\text{C}$  (cylindrical-multiwalled mesoporous carbon tubes) coincides with the peaks in the curves. Thus, to remove impurities of amorphous meso-sized carbon, it was determined that meso-sized carbon tubes with a multi-walled mesoporous structure should be calcined in air at  $T \sim 350$ -

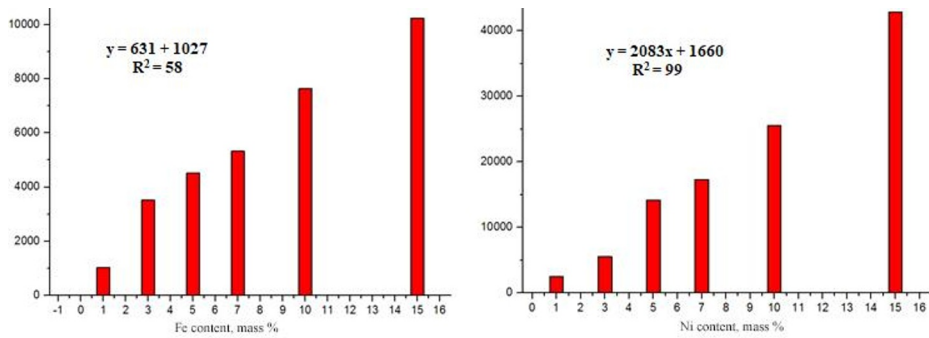
400 °C. The differential scanning calorimetry curves of samples of meso-sized carbon tubes with sintered multi-walled mesoporous structures do not have the first peak, which indicates the selective removal of impurities. The ignition temperature of meso-sized carbon tubes with cylindrical and conical-multi-walled mesoporous structures differs: in the latter, it is lower by 20-40 °C, which is due to a larger surface area and a larger number of meso- and pores with a size > 50 nm, which facilitate air diffusion related to the presence of In addition, this may be related to the specific features of their structure: oxidation of the surface of meso-sized carbon tubes with a conical-multi-walled mesoporous structure saturated with sp<sup>3</sup>-hybridized and valence-unsaturated meso-sized carbon atoms turns into a cylindrical-multi-walled mesoporous structure consisting of graphene planes is significantly easier than the oxidation of the surface of meso-sized carbon tubes with. According to thermogravimetric data, the amount of unstructured forms of meso-sized carbon in the samples of meso-sized carbon tubes with a multi-walled mesoporous structure decreases several times, from 2.0-8.0% to 0.5-2%. Electron microscopy (Fig. 4) confirmed the effectiveness of the cleaning by burning in the air. Micrographs of a sample of meso-sized carbon tubes with a refined conical-multi-walled mesoporous structure clearly show the morphology of the tube with a porous structure with a size of 2÷50 nm, and it can be seen that the amorphous meso-sized carbon derivatives have been removed.



**Fig. 4.** Micrographs of meso-sized carbon tubes with a conical-multi-walled mesoporous structure a) - before and b) - after burning in the air at 350 °C.

Cleaning of metal from particles with a porous structure with a size of 2÷50 nm. The most convenient and simple way to clean meso-sized carbon tubes with a porous structure of 2÷50 nm from the metal catalyst is treated with strong mineral acids. Nitric, sulfuric, and hydrochloric acids and their mixtures are often used.

We used EDXFA and thermogravimetric (TGA) methods to determine the amount of metal in samples of meso-sized carbon tubes with an initial multi-walled mesoporous structure and after acid treatment. Both methods are acceptable in terms of speed and convenience. The advantage of X-ray fluorescence analysis over other methods is that it allows determining the amount of metal in a small part of the sample (equal to 1 g) much faster and without preliminary preparation. Calibration curves for the determination of the content of metals in meso-sized carbon tubes with a multi-walled mesoporous structure are presented in Fig. 5.



**Fig. 5.** Calibration curves for determination of iron and nickel by energy dispersive X-ray fluorescence analysis

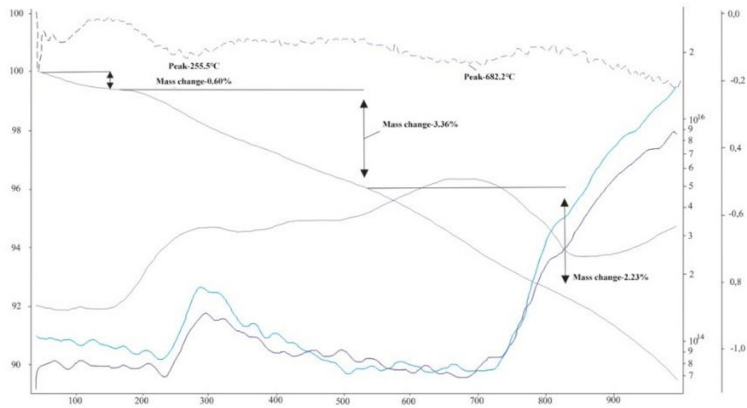
**Table 1.** Metal content in samples of conical (meso-sized carbon tubes with multi-walled mesoporous structure -Ni) and cylindrical (meso-sized carbon tubes with multi-walled mesoporous structure -Fe) tubes with a porous structure with a size of 2÷50 nm

|                                                                     | The amount of Me in meso-sized carbon tubes with an initial multi-walled mesoporous structure, Mass. % |         | The amount of Me in the treated 10M HNO3 meso-sized carbon tubes with multi-walled mesoporous structure, Mass. % |         | Me content in meso-sized carbon tubes with a multi-walled mesoporous structure treated with 10M HCl, Mass. % |         | Me content in meso-sized carbon tubes with a multi-walled mesoporous structure treated with 10M HNO3 + 10M HCl, Mass. % |         |
|---------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------|---------|------------------------------------------------------------------------------------------------------------------|---------|--------------------------------------------------------------------------------------------------------------|---------|-------------------------------------------------------------------------------------------------------------------------|---------|
|                                                                     | TGA                                                                                                    | EDXFA   | TGA                                                                                                              | EDXFA   | TGA                                                                                                          | EDXFA   | TGA                                                                                                                     | EDXFA   |
| Meso-sized carbon tubes with multi-walled mesoporous structure - Ni | 6.8±0.6                                                                                                | 7.2±0.4 | 1.8±0.3                                                                                                          | 1.5±0.2 | 0.9±0.2                                                                                                      | 0.7±0.2 | 1.9±0.3                                                                                                                 | 1.8±0.2 |
| Meso-sized carbon tubes with a multi-wall mesoporous structure - Fe | 4.0±0.5                                                                                                | 3.8±0.3 | 1.7±0.3                                                                                                          | 1.6±0.2 | 1.2±0.2                                                                                                      | 1.0±0.2 | 1.4±0.4                                                                                                                 | 1.5±0.3 |

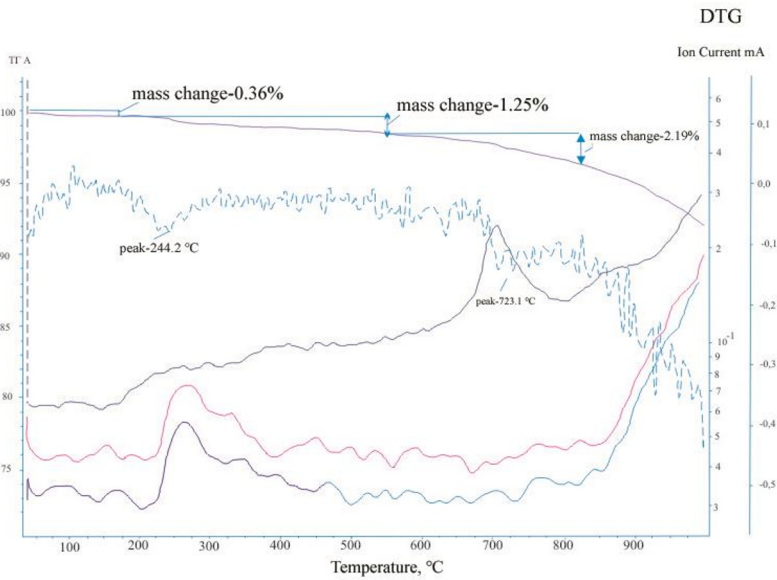
According to the results of measurement of the metal content of samples of meso-sized carbon tubes with multi-walled mesoporous structure after washing with nitrate, chloride, hydrochloric and nitric acid mixtures, hydrochloric acid treatment is the best result for meso-sized carbon tubes with conical-multi-walled mesoporous structure. It can be said that it gives mesosized carbon tubes with a cylindrical-multi-walled mesoporous structure, and in the case of -Fe, it is not oxidizing and does not passivate these metals.

Surface modification and functionalization of meso-sized carbon tubes with multi-walled mesoporous structures with oxygen-containing groups. Giving hydrophilic properties to tubes with a 2÷50 nm pore structure to form stable dispersions in polar solvents and polymeric materials, then stabilizing particles with a metal pore structure 2÷50 nm on the surface of meso-sized carbon tubes with a multi-walled mesoporous structure, also, to provide the possibility of further functionalization of meso-sized carbon tubes with multi-walled mesoporous structure with organic fragments, their surface was modified with

oxygen-containing groups, and the effect of various oxidizing agents on the properties and structure of meso-sized carbon tubes with multi-walled mesoporous structure was studied. To work with oxygen-containing groups, the surface of meso-sized carbon tubes with a multi-walled mesoporous structure was treated with the following reagents: nitric acid, a mixture of sulfate and nitric acids, 37% hydrogen peroxide, liquid ozone, and gaseous ozone in oxygen plasma. Meso-sized carbon formed by oxidation with a mixture of sulphate and nitric acids was determined by thermogravimetry and mass spectroscopic analysis in an inert atmosphere (Fig. 6 and Fig. 7).



**Fig. 6.** Thermogravimetry and mass spectroscopic analysis of meso-sized carbon tubes with conical-multiwall mesoporous structure treated with HNO<sub>3</sub>/H<sub>2</sub>SO<sub>4</sub> mixture.



**Fig. 7.** Thermogravimetry and mass spectroscopic analysis of meso-sized carbon tubes with cylindrical-multiwall mesoporous structure treated with HNO<sub>3</sub>/H<sub>2</sub>SO<sub>4</sub> mixture.

IR spectroscopy study of meso-sized carbon tubes with oxidized multi-walled mesoporous structure. For qualitative analysis of the formed groups, IR spectra of meso-

sized carbon tubes with conical- and cylindrical-multi-walled mesoporous structures were recorded. The spectra of all meso-sized carbon tubes with a multi-walled mesoporous structure contain lines associated with the valence vibrations of the meso-sized carbon-meso-sized carbon bonds of the tube skeleton with a porous structure with a size of 2÷50 nm: in the IR spectrum, to a cylindrical-multi-walled mesoporous structure for meso-sized carbon tubes (C-C) with broad absorption maxima at 1235 cm<sup>-1</sup> and narrower bands at 1171 cm<sup>-1</sup> for meso-sized carbon tubes (C-C) with conical-multiwalled mesoporous structure in the IR spectrum, as well as size (C=C) at 1580 cm<sup>-1</sup> for both types of tubes with a porous structure of 2÷50 nm.

**3.3 Study of meso-sized carbon tubes with multi-walled mesoporous structure oxidized by nitric acid by X-ray photoelectron spectroscopy method**

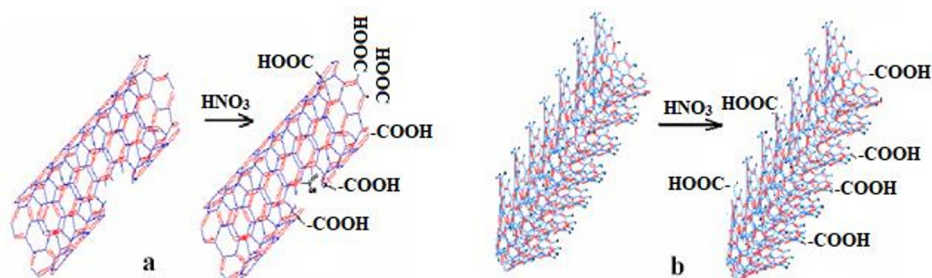
The samples were studied by X-ray photoelectron spectroscopy to study in more detail the types of groups and their distribution on the surface of meso-sized carbon tubes with an oxidized multi-walled mesoporous structure. Raman spectra and photoemission spectra of tubes with a porous structure with an initial size of 2÷50 nm, confirming data obtained from X-ray diffraction, show that both types of tubes are defective (they have sp<sup>2</sup>-unhybridized meso-sized carbon atoms).

**Table 2.** Results of titrimetric determination of carboxyl groups in the presence of barium acetate

| No | Mass of meso-sized carbon tubes with multi-walled mesoporous structure, mg | ΔV, ml | C(NaOH), n | Degree of functionalization (mass %) | Number of carboxyl groups, mmol/g<br>Meso-sized carbon tubes with multi-walled mesoporous structure -COOH |
|----|----------------------------------------------------------------------------|--------|------------|--------------------------------------|-----------------------------------------------------------------------------------------------------------|
| 1  | 50                                                                         | 2.55   | 0.01       | 2.87                                 | 0.64                                                                                                      |
| 2  | 52                                                                         | 2.5    | 0.01       | 2.7                                  | 0.6                                                                                                       |
| 3  | 51                                                                         | 2.46   | 0.01       | 2.71                                 | 0.6                                                                                                       |
| 4  | 49                                                                         | 2.47   | 0.01       | 2.83                                 | 0.63                                                                                                      |
| 5  | 51                                                                         | 2.53   | 0.01       | 2.79                                 | 0.62                                                                                                      |

**3.4 Determining the level of functionality of meso-sized carbon tubes with conical- and cylindrical-multi-walled mesoporous structures treated with different oxidizing agents.**

The most effective oxidation of tubes with a conical and cylindrical mesoporous structure of 2÷50 nm size with nitric acid. This is shown schematically in Figure 8.



**Fig. 8.** Carboxylation scheme of (a) meso-sized carbon tubes with cylindrical-multi-walled mesoporous structure and (b) meso-sized carbon tubes with a conical-multi-walled mesoporous structure under nitric acid treatment.

Ozone treatment in oxygen discharge plasma and liquid ozone resulted in significantly lower functionality. At the same time, meso-sized carbon tubes with a porous structure with a size of 2÷50 nm were not oxidized to CO<sub>2</sub> by liquid ozone, which, respectively, meso-sized carbon tubes with a conical-multi-walled mesoporous structure ( $2.5 \pm 0.2$  and  $2.0 \pm 0.2$  % showed a higher level of functionality for meso-sized carbon tubes with a cylindrical-multiwall mesoporous structure than o). The opposite reaction was observed in the oxidation of HNO<sub>3</sub>. This may be due to the difference in the mechanisms of interaction of oxidants with tubes having a porous structure with a size of 2÷50 nm: nitric acid mainly oxidizes sp<sup>3</sup>-hybridized meso-sized carbon atoms, while ozone can attack double bonds and meso-sized carbon-oxygen bond causes them to break as soon as they are formed.

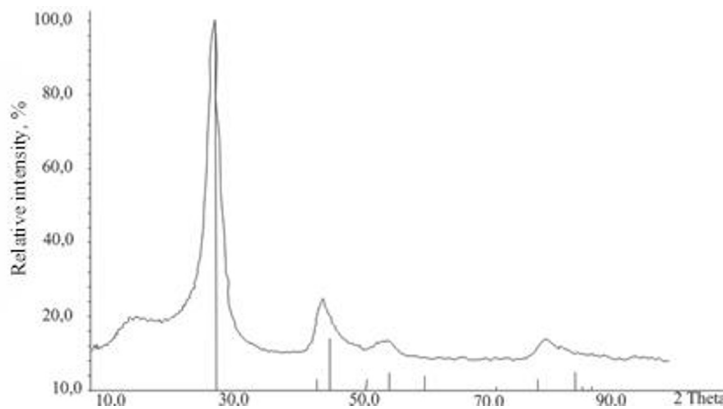
The degree of carboxylation of meso-sized carbon tubes with a conical-multiwalled mesoporous structure oxidized with H<sub>2</sub>O<sub>2</sub> was lower than that oxidized with HNO<sub>3</sub> and was  $3.3 \pm 0.3$  % ( $0.78 \pm 0.01$  mmol/g) in the opposite direction to  $6.5 \pm 0.4$  % ( $1.5 \pm 0.02$  mmol/g) and  $1.4 \pm 0.1$  % ( $0.320 \pm 0.005$  mmol/g) for meso-sized carbon tubes with a cylindrical-multi-walled mesoporous structure in the opposite direction  $2.9 \pm 0.2$  % ( $0.64 \pm 0.01$  mmol/g).

Undoubtedly, this is explained by the decomposition of peroxide on the surface of meso-sized carbon tubes, which have a multi-walled mesoporous structure due to the large specific surface area of the material. As a result, H<sub>2</sub>O<sub>2</sub> is a weaker oxidant than meso-sized carbon materials.

A comparison of different methods of processing meso-sized carbon tubes with a multi-walled mesoporous structure shows the advantages of using nitric acid as a modifying agent, since it promotes the formation of structural fragments of the most molecules among the oxidizing agents used, which are easy to use and by simple washing with water is easily removed from the system without absorption in meso-sized carbon tubes with a multi-walled mesoporous structure. The latter conclusion was made based on elemental analysis data on the absence of nitrogen in samples of meso-sized carbon tubes with multi-walled mesoporous structures.

The effect of acid treatment on the morphology of tubes with a porous structure with a size of 2÷50 nm. Meso-sized carbon tubes with a multi-walled mesoporous structure processed in nitric acid, as well as the first ones, showed stability up to 450-500 °C when heated in air. Their structural and electronic characteristics, calculated from Raman spectra and diffraction data, remained virtually unchanged.

The method of X-ray diffraction of the oxidized samples indicates almost complete removal of the metal (Fig. 9).



**Fig. 9.** X-ray image of meso-sized carbon tubes with conical-multi-walled mesoporous structure in oxidized 6M HNO<sub>3</sub>

Only broadened reflections from the graphite structure were observed in the diffraction pattern, shifted to smaller angles due to increased interplanar voids in meso-sized carbon tubes with a multi-walled mesoporous structure compared to graphite. The surface area of meso-sized carbon tubes with multi-walled mesoporous structure -COOH is on average slightly larger than that of meso-sized carbon tubes with original multi-walled mesoporous structure by 20-50 m<sup>2</sup> (Table 3), which is probably due to the opening of pores due to metal leaching, depends and the resulting increase in surface area, while the distribution of diameter holes is similar to the original sample.

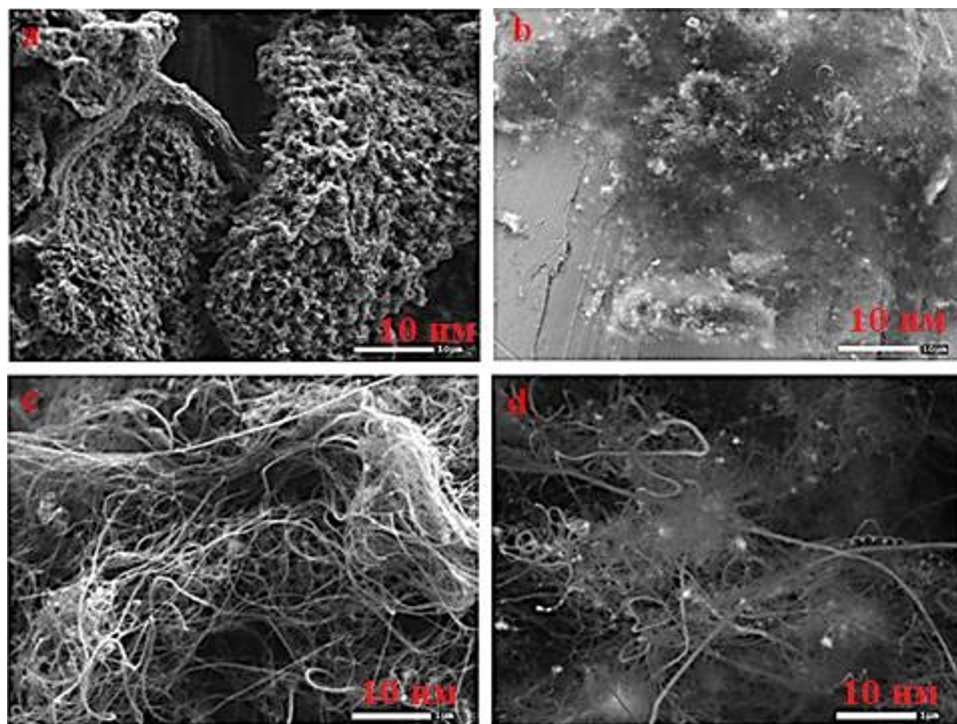
**Table 3.** The comparative surface area of meso-sized carbon tubes with conical- and cylindrical-multiwalled mesoporous structures measured by nitrogen absorption using the BET method

| Sample № | S <sub>sol</sub> (meso-sized carbon tubes with conical-multiwall mesoporous structure - COOH), m <sup>2</sup> /g | S <sub>sol</sub> (meso-sized carbon tubes with a cylindrical-multi-walled mesoporous structure - COOH), m <sup>2</sup> /g |
|----------|------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------|
| 1        | 282±11                                                                                                           | 212±8                                                                                                                     |
| 2        | 258±10                                                                                                           | 215±8                                                                                                                     |
| 3        | 403±16                                                                                                           | 223±9                                                                                                                     |
| 4        | 305±12                                                                                                           | 205±8                                                                                                                     |
| 5        | 276±11                                                                                                           | 232±9                                                                                                                     |

As an example, in meso-sized carbon tubes with a single-walled porous structure with a size of 2÷50 nm, the functionality of tubes with a porous structure with a size of 2÷50 nm increases with increasing acid treatment time, but destructive processes occur at the same time can be. This is especially true for meso-sized carbon tubes with a conical-multi-walled mesoporous structure because their structure is more susceptible to the influence of strong agents than meso-sized carbon tubes with a cylindrical-multi-walled mesoporous structure, where the size is 2÷50 nm graphite planes forming the tube walls with a porous structure are less reactive, and the C-C bond energy is higher due to the formation of a single aromatic system through hexagonal meso-sized carbon rings.

In order to study the changes in the structure of meso-sized carbon tubes with a multi-walled mesoporous structure during oxidation, several samples treated with 12 M HNO<sub>3</sub> for 3, 6, 9 and 15 hours were analysed by transmission electron microscopy. The resulting

images are presented in Figure 10. It can be seen that after 9 hours of oxidation, tubes with a porous structure with a size of  $2\div 50$  nm begin to disintegrate, and after 15 hours, their almost destruction occurs, leaving only tubes with a porous structure with the largest size of  $2\div 50$  nm. Therefore, this type of long-term processing has not been carried out because of the possible destruction of meso-sized carbon tubes with multi-wall mesoporous structures.



**Fig. 10.** Micrographs of meso-sized carbon tubes with conical-multiwalled mesoporous structure oxidized in 12M HNO<sub>3</sub> at different times, a - 3, b - 6, c - 9 and d - 15 h

## 4 Conclusion

Specific surface area ( $S_{sol}$ ) and pore size distribution were determined by BET absorption at a low temperature of 77K. According to the BET model, the specific surface area was calculated and the BJH model of the pore size distribution in the material was used.

Meso-sized carbon tubes with an initial multi-walled mesoporous structure were identified based on thermogravimetry data for 2 h. It was cleaned of meso-sized carbon impurities by burning in the air in a furnace at a temperature of 350-400 °C.

According to the results of measurement of the metal content of samples of meso-sized carbon tubes with multi-walled mesoporous structure after washing with nitrate, chloride, hydrochloric and nitric acid mixtures, hydrochloric acid treatment is the best result for meso-sized carbon tubes with conical-multi-walled mesoporous structure gives A comparison of different methods of processing meso-sized carbon tubes with a multi-walled mesoporous structure shows the advantages of using nitric acid as a modifying agent, since it promotes the formation of structural fragments of the most molecules among the oxidizing agents used, which are easy to use and by simple washing with water is easily removed from the system without absorption in meso-sized carbon tubes with a multi-walled mesoporous structure. The latter conclusion was made based on elemental analysis

data on the absence of nitrogen in samples of meso-sized carbon tubes with multi-walled mesoporous structures.

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