Original papers
Nanostructured, nanoscale materials and nanodevices

УДК 535.341.08 DOI: 10.17277/jamt.2022.02.pp.104-112

Effect of the carbon nanotubes size and oxidative treatment on their optical properties

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Abstract: Optical properties of carbon nanotubes depend on their geometric dimensions, presence of defects and availability of functional groups; all this is important to consider when choosing carbon nanotubes for specific photo-thermal and other processes. The present study investigated temperature differences between colloidal suspensions under the influence of sunlight, due to the difference in the rate of photo-thermal transformation (as a result of optical absorption). It was found that as the size of carbon nanotubes decreased, the optical absorption increases and its peak shifted to the blue side. Oxidized carbon nanotubes demonstrated an increase in overall optical absorption compared to non-oxidized carbon nanotubes. The optical absorption peak corresponding to π-plasmon in oxidized and non-oxidized carbon nanotubes of the Taunit-M and Taunit types was at values of 1.96, 1.25 and 1.13, respectively. Also, the temperature of colloidal suspensions and the rate of their heating increased with a decrease in the size of carbon nanotubes. As a result of the research, the carbon nanotubes type most suitable for specific use in optical absorbing devices (such as solar collectors) was determined based on the carbon nanotubes ratio to the target electromagnetic spectrum.

Keywords: antenna effect; nano-antenna; light absorption; multi-walled carbon nanotubes; optical properties; interaction of light and matter.

For citation: Ezzeddin B, Tugolukov EN, Dyachkova TP, Chapaksov NA. Effect of the carbon nanotubes size and oxidative treatment on their optical properties. *Journal of Advanced Materials and Technologies*. 2022;7(2):104-112. DOI:10.17277/jamt.2022.02.pp.104-112

Влияние размеров углеродных нанотрубок и окислительной обработки на их оптические свойства

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Аннотация: Оптические свойства углеродных нанотрубок зависят от их геометрических размеров, степени дефектности и наличия функциональных групп, что важно учитывать при выборе углеродных нанотрубок для использования в конкретных фототермических и других процессах. Исследованы температурные различия между коллоидными суспензиями при воздействии солнечного света, обусловленные разницей в скорости фототермического преобразования (в результате оптического поглощения). Показано, что с уменьшением размеров углеродных нанотрубок оптическое поглощение увеличивается, а его пик смещается в синюю сторону. Окисленные углеродные нанотрубки демонстрируют повышение общего оптического поглощения по сравнению с неокисленными углеродными нанотрубками. Пик оптического поглощения, соответствующий π-плазмону, в окисленных и неокисленных углеродных нанотрубках типа «Таунит-М» и «Таунит» находился при значениях 1,96, 1,25 и 1,13 соответственно. Показано, что температура коллоидных суспензий и скорость их нагрева увеличиваются с уменьшением размера углеродных нанотрубок. В результате проведенных исследований определен тип углеродных нанотрубок, наиболее подходящий для конкретного использования в оптических поглощающих устройствах (таких как солнечные коллекторы) на основе отношения размеров углеродных нанотрубок к целевому электромагнитному спектру.

Ключевые слова: антенный эффект; наноантенна; поглощение света; многостенные углеродные нанотрубки; оптические свойства; взаимодействие света с веществом.

Для цитирования: Ezzeddin B, Tugolukov EN, Dyachkova TP, Chapaksov NA. Effect of the carbon nanotubes size and oxidative treatment on their optical properties. *Journal of Advanced Materials and Technologies*. 2022;7(2):104-112. DOI:10.17277/jamt.2022.02.pp.104-112

1. Introduction

Unique optical and thermal properties of carbon nanotubes (CNTs) determine the possibility of their use in various fields of industry, medicine, and energy [1–3]. In medicine, CNTs are used in the thermal treatment of tumors as local heat sources [4, 5] activated by electromagnetic radiation with certain wavelengths. In CNTs, surface plasmons will got excited when the frequency of electromagnetic waves correspond to the frequency of surface plasmons in CNTs. The excitation of surface plasmons is associated with the release of Joule heat as a result of ohmic losses during longitudinal electrical oscillations in CNTs. In the industrial field, CNTs have found application for creating selective chemical sensors, since it has been proven that the optical properties of CNTs change when a chemical reaction occurs between them and some chemical compounds [7]. The change in the optical response of these sensors is related to the change in their electronic structure. This, in turn, leads to a change in the nature of the interaction of CNTs with electromagnetic waves. In the field of renewable energy, CNTs are used to convert solar energy. In a study by Wang et al. [3], CNTs were used to produce water vapor by absorbing sunlight and converting it into thermal energy. However, this study did not report the effect of CNT parameters (such as diameter, tube length, and surface chemistry) on their temperature and heating (evaporation) rate. Also in the field of renewable energy, CNTs have been used to improve the efficiency of converting photon energy into electrical energy in photovoltaic cells. In [8], a new model of a solar cell based on CNTs in the form of a light-absorbing layer was proposed. The results of this study showed that the optimal thickness of the light-absorbing layer is 4 μm, and the optimal concentration of CNTs are $3 \cdot 10^{18}$ cm^{-3}.

These optical and thermal properties of CNTs are partially explained by their electronic structure, since these properties change significantly with a change in the electronic structure. In a study by Iskender Muz et al. [9], it was found that the modification of CNTs with various elements leads to a change in their optical properties, and their metal-coating by atoms of various elements leads to a change in the dipole moments and polarization of these CNTs (as a result of a change in its electronic structure). The optical response of these CNTs is complicated, since they obey, on the one hand, the Bouguer-Lambert-Beer law [10], where it was discovered in a study by Mohammad Mustafa Gafuryan et al. [11], namely, with an increase in the concentration of nanotubes in a colloidal suspension consisting of water/multi-walled carbon nanotubes, the optical absorption of the colloidal suspension increased. On the other hand, the optical response of CNTs is also related to antenna theory [12], so their interaction with electromagnetic waves (including light of different wavelengths) depends on their spatial orientation and length. In a study by Binkai Chen et al. [13], it was found that CNTs interact with infrared radiation in such a way that their optical absorption in the case of polarization of electromagnetic waves parallel to the CNT axis (the electric component E of the electromagnetic wave is parallel to the CNT axis) was greater than their optical absorption when electromagnetic waves are polarized perpendicularly the CNT axis.

Several studies have been carried out to study the interaction between CNT arrays and light. A study conducted by Hua Bao et al. [14] examined the effects of volume fraction, tube length, tube spacing, and angle of incidence on the radiative properties. It has been found that low volume fraction and long tubes are more favorable for achieving low reflectance and high absorption, and for a fixed volume fraction and a finite tube length, greater periodicity results in greater absorption. The results also show that even higher absorbance material can be obtained using CNTs with good alignment on the top surface. In another study by Wood B.D. et al. [15], the relationship between the height and density of forests of multi-walled CNTs and their optical properties in the mid-infrared range was studied. The study found that at a suitable density for a carbon nanotube forest, a randomly modified forest less than 20 μm high could generate a reflection of less than 0.002 in the mid-infrared range.

Several researches have also been done to study the interaction between individual carbon nanotubes and light. In a study conducted by Jean-Christophe Blancon et al. [16], the spectrum and amplitude of absorption by the cross section of individual single-walled CNTs were studied. A non-resonant background comparable to the optical absorption of ideal graphene was also identified. This study showed that the same single-walled CNT, whether standing alone or lying on a substrate, demonstrates a significant broadening of exciton resonances with increasing oscillator strength, as well as a sharp weakening of polarization-dependent antenna effects. Despite the aforementioned interactions of CNTs with electromagnetic waves, only experimental results were presented without a full explanation of the observed effects, taking into account all the factors influencing the interaction between electromagnetic waves and CNTs. For example, the process of CNT oxidation affects not only the chemical composition of the CNT surface, but also its size (the CNT diameter and length decrease).

In this paper, was presented the research results of the impact of dimensions of multi-walled CNTs and the oxidative treatment of the surface of these tubes on their optical properties.

2. Materials and Methods

2.1. Materials

"Taunit-M" CNTs (inner diameter is about 5–15 nm, outer diameter is about 10–30 nm, length $l \geq 2$ μm, specific surface ≥ 270 m^2g^{-1}), "Taunit" CNTs (inner diameter is about 10–20 nm, outer diameter is about 20–50 nm, length $l \geq 2$ μm, specific surface ≥ 160 m^2g^{-1}) and oxidized "Taunit-M" CNTs were obtained at Nano-Technical Center LLC (Tambov).

The dispersant sodium dodecyl-benzene-sulfonate (SDBS) was prepared in the form of a 40 % aqueous solution (Rus-chem-trade LLC (Dzerzhinsk)).

2.2. Suspension preparation methods

For preparing colloidal suspensions the following operations were performed: first, 0.6 g of CNTs (powder) was placed in a glass container, then 3 g of a dispersing agent (sodium dodecyl-benzene-sulfonate "SDBS") was added, followed by 96.4 g of distilled water.

After this, the mixtures were stirred using a mechanical mixer with at speed of 1000 rpm for two minutes. Then the mixtures were subjected to ultrasonic treatment using an ultrasonic generator (I10, St. Petersburg). The mixtures were sonicated for 60 min in a cold water bath. Figure 1 shows the colloidal suspension preparing process.

In order to calculate the mass concentration of CNTs after the completion of the ultrasound treatment, colloidal suspensions were left for a week

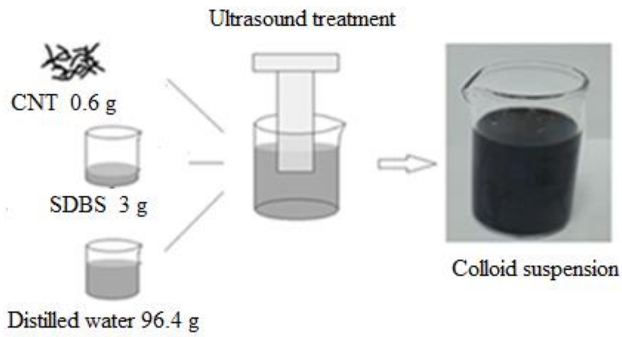


Fig. 1. The colloidal suspension preparing process from CNTs

to give enough time for the deposition of poorly dispersed CNTs, since the presence of CNT agglomerates in a colloidal suspension causes a red shift in the optical absorption spectra of the samples [17, 18].

To avoid sediment agitation, the liquid was slowly taken up with a syringe, and after removing the colloidal suspension, the glass vessel was placed in an oven (Sanyo Convection Oven Mov 210F, Osaka, Japan) for three hours to dry the carbon nanotubes and then determine their weight. Thus, it was possible to determine the mass of CNTs suspended in a colloidal suspension.

After determining the mass concentration of CNTs in colloidal suspensions, these concentrations were corrected by adding distilled water to obtain a concentration of 0.4 wt. %. The purpose of standardizing the concentration of CNTs in colloidal suspensions was to avoid the concentration effect in optical absorption.

In order to determine the temperature differences between colloidal suspensions of CNTs of different types due to the different rate of photo-thermal transformation in them, all samples were kept in sunlight (to reproduce the conditions corresponding to the operating conditions of solar thermal equipment) for one and a half hours, and the temperature of colloidal suspensions was measured every 15 minutes.

2.3. Methods for studying the CNTs properties

Optical absorption spectra of three samples of a colloidal suspension of CNTs various types with a 0.4 wt. % concentration were determined using a UV-Vis spectrophotometer (Lambda 35, Waltham, MA, USA).

Raman spectroscopy was performed using a DXR Raman Microscope Thermo Scientific, Madison, Wisconsin, USA (irradiation laser wavelength 532 nm). Structural defects of CNTs before and after oxidation were determined, as well as the influence of the size factor on the optical absorption of CNTs.

FT-IR spectroscopy was performed using an FT-IR spectrometer (Jasco FT/IR-6700, Sennincho Hachioji, Japan). The optical transmission in the middle and far infrared ranges of suspensions containing various types of CNTs was studied.

The temperature was measured using a thermometer (digital thermometer TR-101) in order to determine the temperature difference between colloidal suspensions with different photo-thermal conversion.

3. Results and Discussion

3.1. UV-visible spectroscopy

Measurement of the samples optical absorption showed that the optical absorption of oxidized “Taunit-M” CNTs was higher than the optical absorption of non-oxidized “Taunit-M” CNTs, which, in turn, was higher than that of “Taunit” CNTs (Fig. 2). This means that the Taunit type has the highest optical transmittance, followed by the non-oxidized “Taunit-M” and then the oxidized “Taunit-M”.

The increase in optical absorption with a decrease in the size of nanotubes (mainly as a result of a decrease in the average diameter of CNTs) can be explained as follows: on the one hand, this increase is due to the fact that, at the same mass concentration of CNTs in a colloidal suspension, the number of CNTs in the case of tubes of smaller diameter is greater, than the number of tubes of larger diameter per unit volume of liquid, provided that the lengths of nanotubes are commensurable, which increases the value of the filling factor and the surface area of interaction between CNTs and photons, and this, in turn, increases the probability of absorption of photons passing through the sample (the Bouguer-Lambert-Beer law). On the other hand, CNTs interact with electromagnetic waves as antennas [20], and with an increase in the quantitative concentration of nanotubes, the probability of absorbing electromagnetic waves passing through the sample increases due to an increase in the number of nanotubes of the appropriate length to absorb these electromagnetic waves.

Also, with a decrease in the CNT size, a “blue shift” was observed in the absorption peaks corresponding to the excitation of surface plasmons of carbon nanotubes (π plasmon). In the UV range, we detected optical absorption peaks at 318 nm for the Taunit type, at 293 nm for the non-oxidized Taunit-M type, and at 281 nm for the oxidized

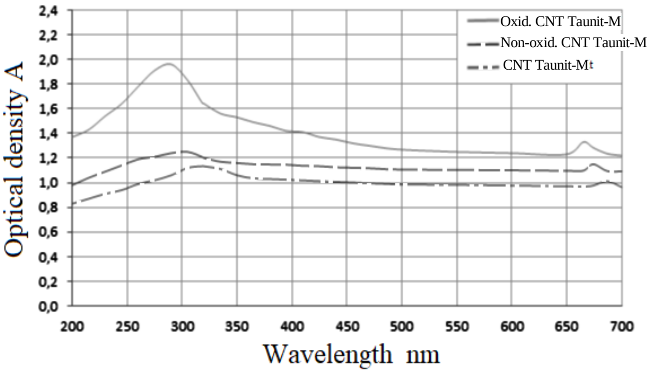


Fig. 2. Optical density of samples with oxidized “Taunit-M” CNTs, non-oxidized “Taunit-M” CNTs and “Taunit” CNTs

Taunit-M type. This can be explained as follows: decreasing in the diameter of nanotubes leads to an increase in the effect of quantum confinement [21]. As a result, the frequency (energy) of electromagnetic waves required to excite plasmons in nanotubes increases.

It should be noted that the oxidation process reduces the length and thickness of CNTs, which leads to an increase in the intensity of optical absorption and a blue shift in the absorption peak compared to non-oxidized CNTs for the reasons described above.

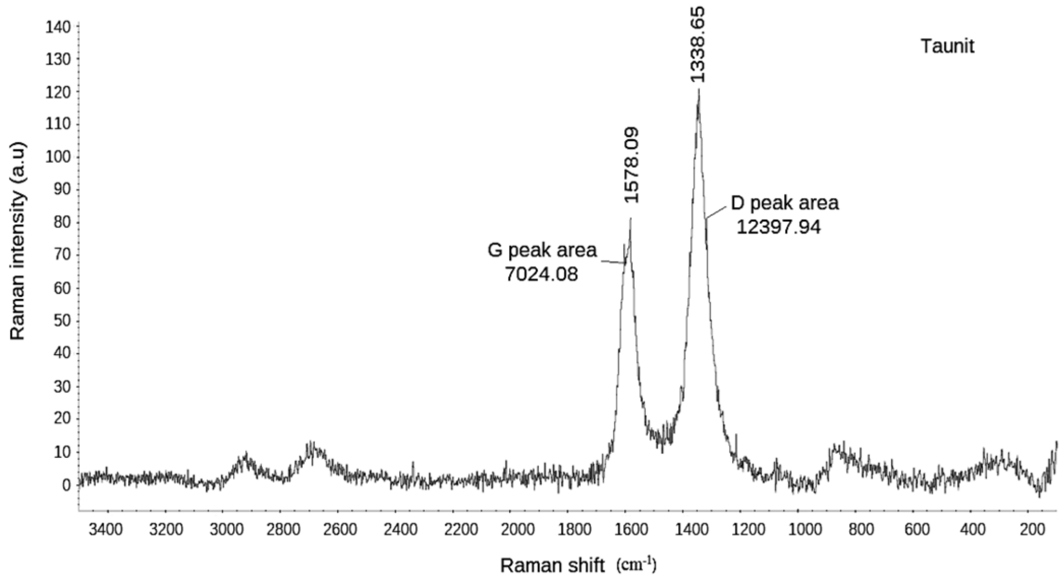
In addition, the oxidation process increases the number of C—O bonds at the expense of C—C bonds, which also leads to a “blue shift” in the optical absorption spectrum, since the C—O bond has a higher electronegativity (polarity) than C—C [22], which requires higher energies of photons to separate charges, so the frequency of photons that can cause excitation in the electron cloud must be higher.

3.2. Raman spectroscopy

The results of Raman spectroscopy of various types of CNTs showed a “blue shift” at the G-peak, which was observed when the average value of the CNT diameter decreased. The G-peaks for “Taunit”, non-oxidized “Taunit-M” and oxidized “Taunit-M” were observed at 6337 (1578.09 cm^{-1}), 6270 (1595.50 cm^{-1}) and 6258 (1598.92 cm^{-1}) nm, respectively (Fig. 3).

It is also worth noting that the Raman intensity in a CNT sample of the non-oxidized “Taunit-M” type was higher than that of the oxidized “Taunit-M” type.

It can be said that the CNTs used in this paper are a package of concentric single-walled nanotubes (i.e., all single-walled carbon nanotubes that make up a multi-walled CNT have one longitudinal axis).



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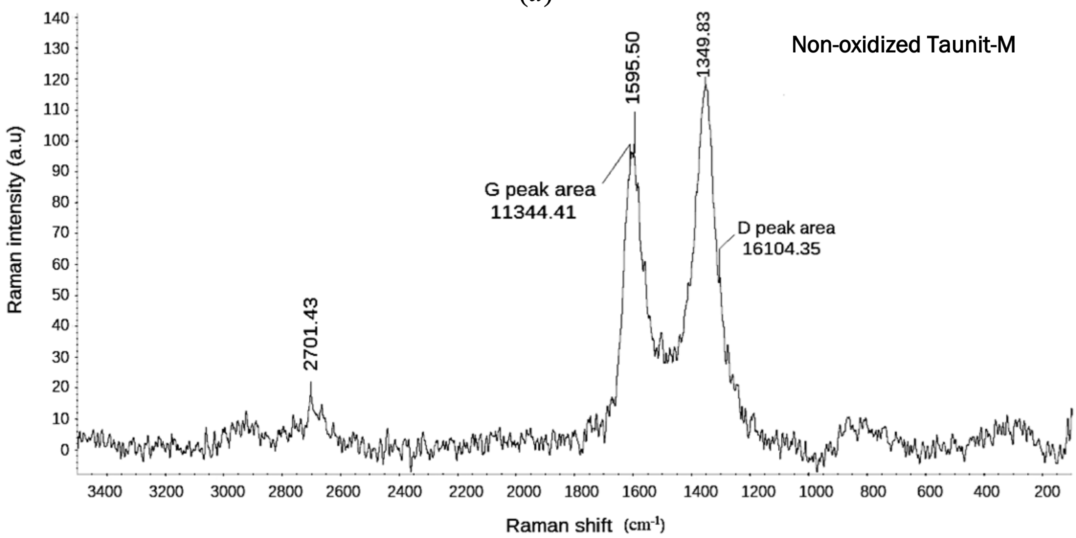

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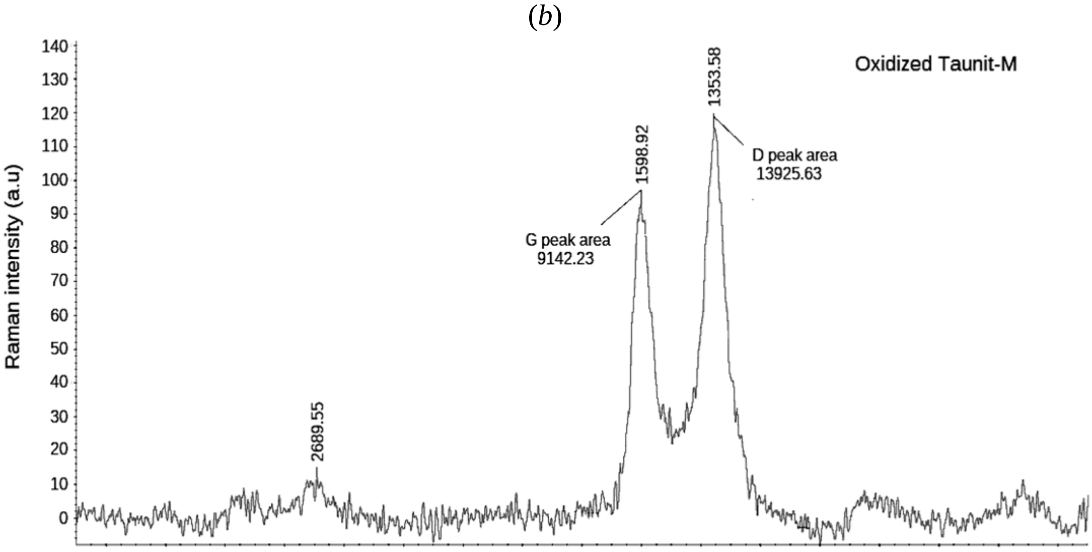

(*c*)

Fig. 3. Raman spectra of samples “Taunit” (*a*), non-oxidized “Taunit-M” (*b*) and oxidized “Taunit-M” (*c*)

With a decrease in the CNT diameter and due to an increase in the quantum confinement effect, the energy required for the occurrence of Stokes-Raman excitation increases in the carbon atoms that make up the graphene layer, where the van der Waals force between the graphene layers in a multilayer nanotube affects the collective vibrations of these layers [24], and this affects the phonon effects in CNTs. As the number of layers in CNTs decreases (i.e., their diameter decreases), the vibrational coupling of the graphene layers that make up the wall of carbon nanotubes shifts towards shorter electromagnetic waves. Thus, a "blue shift" of the G-peak appears in the Raman spectrum. The decrease in the intensity of Raman light scattering in the oxidized "Taunit-M" compared to the non-oxidized "Taunit-M" can be explained by the fact that the oxidation process affected the graphene layers and led to an increase in defects in these layers, which, in turn, led to a change in electron-phonon interaction in graphene layers [25].

It is also noted that the I_D/I_G ratio of 1.523 and 1.419 for oxidized "Taunit-M" and non-oxidized "Taunit-M", respectively, confirms the fact that the oxidation process causes a greater number of defects in the graphene layer. It should be noted that the oxidation process leads to a decrease in CNT layers, which, in turn, affects the position of the G peak in the Raman spectrum, since with a decrease in the number of CNT layers, the plasticity of bonds between graphene layers forming CNTs decreases, and this, in turn, is reflected in the photon energy required for excitation.

3.3. FT-IR spectroscopy

The results of FT-IR spectroscopy of various types of CNTs showed that a decrease in the diameter of CNTs leads to an improvement in absorption in the mid- and far-IR wavelength range, where the transmittance values of oxidized "Taunit-M" are lower than the corresponding values for non-oxidized "Taunit-M" and "Taunit".

It can be said that the increase in absorption is associated with an increase in the fill factor, since the diameters of the non-oxidized"Taunit-M" and oxidized "Taunit-M" CNTs are smaller than the diameters of the "Taunit" CNTs, which means that the number of nanoantennas interacting with electromagnetic waves, more. As for the increase in absorption in oxidized "Taunit-M" compared to non-oxidized "Taunit-M", it occurs as a result of the fact that the length of oxidized CNTs can be somewhat less than the length of non-oxidized CNTs, therefore, the localization of surface plasmons in them is greater. As for the three considered types, the absorption decreases with an increase in the length of electromagnetic waves due to a decrease in the number of nano-antennas capable of interacting with these electromagnetic waves, since with an increase in the wave length of an electromagnetic, the number of carbon nanotubes satisfying the condition $|\varepsilon_1'| >> |\varepsilon_1''|$ (were ε_1' – the real part of the CNTs permittivity; ε_1'' – the imaginary part of the CNTs permittivity), decreases, which reduces the formation of surface plasmons [26]. Figure 4 shows the FT-IR spectrum of these samples.

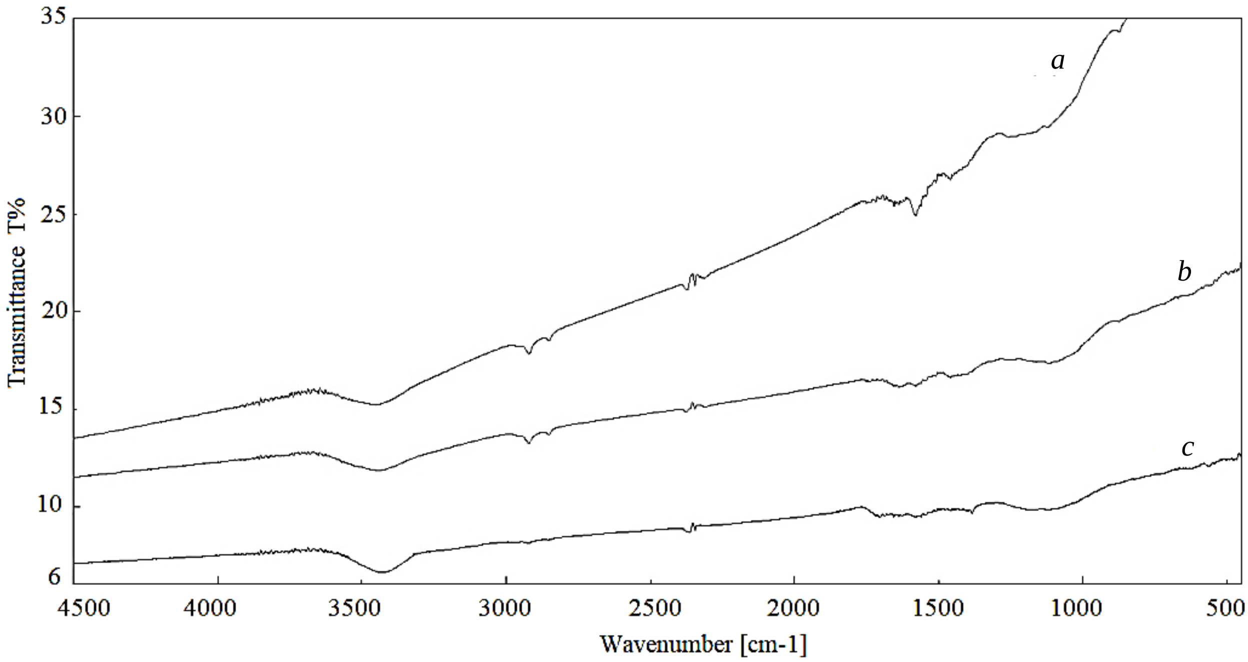

Fig. 4. FT-IR spectra for "Taunit" (*a*), non-oxidized "Taunit-M" (*b*) and oxidized "Taunit-M" (*c*) samples

3.4. Photo-thermal conversion

The results of photo-thermal conversion measurements showed the difference in temperatures of the three studied samples under identical illumination. The temperature of the colloidal suspension containing oxidized “Taunit-M” CNTs was maximum and reached 42.7 °C, while the temperature of the colloidal suspension containing non-oxidized “Taunit-M” CNTs reached 40.2 °C, and the temperature of the colloidal suspension, containing “Taunit” CNTs, reached 38.4 °C.

The difference in sample temperatures can be explained as follows: on the one hand, a change in CNT diameters leads to a change in their quantitative concentration at the same mass concentration. This, in turn, leads to a change in the total surface area of CNTs interacting with photons per unit volume, as well as to a change in the number of heat sources per unit volume, which affects both the heating rate of the colloidal suspension and the final temperature. It is worth noting that smaller diameter CNTs can absorb photons of higher frequency (see Fig. 2) and therefore higher energy, which means more light energy, is converted into heat and an increase in the temperature of the colloidal suspension.

On the other hand, a decrease in the CNT diameter increases the imaginary part of the complex permittivity of carbon nanotubes (which is the extinction coefficient) and the imaginary part of the wave number of surface plasmons, where the imaginary part of the wave number determines the damping* (ohmic losses, i.e. energy loss of surface plasmons).

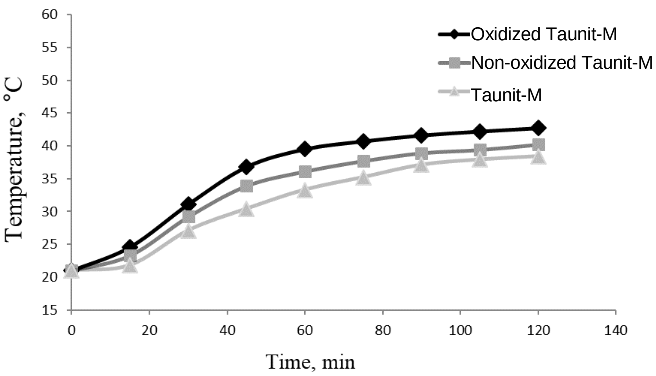

Fig. 5. Temperature dependence of colloidal suspensions for various types of CNTs on the irradiation time

We also observed that the temperature growth rate in colloidal suspensions containing oxidized “Taunit-M” CNTs is greater than the temperature growth rate in colloidal suspensions containing non-oxidized “Taunit-M” CNTs, which, in turn, is higher than the growth rate temperature in colloidal suspensions containing CNT “Taunit”. This is also explained by an increase in the quantitative concentration of CNTs. Figure 5 shows the temperature curves of colloidal suspensions for various types of CNTs depending on the irradiation time.

4. Conclusions

The optical properties of colloidal suspensions, each of which contains different types of multi-walled CNTs, have been studied. The influence of the dimensions of carbon nanotubes (diameter and length) and the oxidative treatment of their surface on the optical properties of suspensions has been studied. The study showed that with a decrease in the size of nanotubes, optical absorption increases, and a “blue shift” of absorption peaks occurs. An increase in the number of defects in carbon nanotubes reduces the intensity of Stokes-Raman scattering. It is shown that the oxidation process improves the overall optical absorption**. It was also found that the temperature and heating rate of the suspension increase with a decrease in the diameter of carbon nanotubes due to an increase in the number (at the same mass concentration) of nanoparticles involved in the process of photothermal conversion and an increase in the absorption of photons with higher energy. As a result of the research, the carbon nanotubes type most suitable for specific use in optical absorbing devices (such as solar collectors) was determined based on the carbon nanotubes ratio to the target electromagnetic spectrum.

5. Funding

This study did not receive external funding.

6. Conflict of interests

The authors declare no conflicts of interest.

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* The vibrational motion of the excited electrons along the CNT.

** Due to an increase in the quantitative concentration of nanoparticles.

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Received 02 March 2022; Accepted 14 April 2022; Published 01 July 2022