



SYNTHESIS AND ANALYSIS OF CARBON NANOTUBES PRODUCED FROM ETHANOL AT LOW TEMPERATURE USING CHEMICAL VAPOR DEPOSITION (CVD) METHOD: XRD, RAMAN, IR, TGA, AND TEM IMAGING STUDIES

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Abstract: *In this article, carbon nanotubes (CNTs) were synthesized using the low-temperature chemical vapor deposition (CVD) method based on an ethanol precursor. During the synthesis process, catalysts based on Ni, NiO, and Fe were used, and the influence of parameters such as temperature, reaction time, and gas flow rate was studied. The phase, structural, and morphological properties of the formed nanotubes were investigated using X-ray diffraction (XRD), Raman spectroscopy, FT-IR spectroscopy, thermogravimetric analysis (TGA/DTA), and transmission electron microscopy (TEM). The XRD results showed a high degree of crystallinity in the graphite phase, while the Raman spectra indicated a low number of defects in the nanotubes based on the D/G ratio. The absence of functional groups in the IR spectra confirmed the purity of the carbon structure. The TGA results revealed the thermal stability of nanotubes up to 800 °C. TEM analysis showed that the nanotubes have diameters of 2-7 nm and lengths of 30-70 nm, forming hollow structures with smooth surfaces and graphitized structures. The obtained results*

confirm that carbon nanotubes synthesized using ethanol have the potential to form high-quality nanostructures.

Keywords: *Carbon nanotubes, ethanol, CVD method, XRD-X-ray diffraction, Raman spectroscopy, IR spectrum, TGA/DTA, TEM, morphology, catalyst.*

INTRODUCTION

In recent years, carbon nanostructures, particularly carbon nanotubes (CNTs), have gained significant scientific and practical importance in the fields of materials science, electronics, energy, and catalysis. After carbon nanotubes were first identified by S. Iijima in 1991, their unique properties such as ultra-high mechanical strength, high electrical conductivity, thermal stability, and large surface area were discovered due to their characteristic cylindrical graphitic structure (1,2). These properties enable their use as nanocomposites, sensors, supercapacitors, hydrogen storage materials, and nanocatalyst carriers (3-5). Carbon nanotubes are synthesized by various methods: arc discharge, laser ablation, and the most common method, chemical vapor deposition (CVD) (6,7). The CVD method is advantageous due to its relatively low operating temperature, the ability to control synthesis conditions, and the possibility of obtaining high-purity products (8,9). In particular, the ethanol-based CVD process is widely used as an environmentally friendly, sustainable, and cost-effective technology (10,11). Carbon atoms formed as a result of ethanol decomposition create nanotubes on the catalyst surface through a "catalytic growth mechanism" (12). In CNT synthesis, catalysts based on transition metals such as Ni, Fe, and Co serve as active centers in the growth process. Notably, Ni-Fe-based catalysts increase the diffusion rate of carbon

atoms, contributing to the formation of ordered graphite layers (13-15). Simultaneously, important parameters in the synthesis process - temperature, gas flow, exposure time, and precursor utilization ratio - directly affect the length, diameter, and degree of graphitization of nanotubes (16,17).

Physico-analytical methods such as X-ray diffraction (XRD), Raman spectroscopy, FT-IR, thermogravimetric analysis (TGA/DTA), and transmission electron microscopy (TEM) are used to assess the structure and phase properties of synthesized nanotubes (18-20). XRD analysis allows determination of the degree of crystallinity of the graphite phase, while Raman analysis enables assessment of the degree of defectiveness through D and G bands (21,22). The IR spectrum indicates the presence of functional groups on the material surface, while TGA demonstrates thermal stability (23,24). TEM microscopy is the primary tool for studying the morphology, diameter, wall thickness, and distribution of catalyst particles in nanotubes (25-27). Therefore, in this study, the morphological, structural, and phase properties of carbon nanotubes synthesized by the CVD method at low temperatures using ethanol as a base were examined in detail using XRD, Raman, IR, TGA, and TEM analyses. The obtained results demonstrated the possibility of producing high-quality nanostructures using ethanol-based carbon sources and provided a scientific basis for the optimization of the CVD process (28-30).

RESEARCH METHODOLOGY

The experimental parameters used in the synthesis and analysis processes are as follows: We presented the CVD device schematically shown in Figure 1. The device consists of a quartz tube (30 mm inner

diameter) equipped with a vacuum system at both ends and connectors for introducing gases and ethanol vapor. The tested substrates were: conductive glass (layers on Si-based glass), quartz plates, and nickel oxide plates. The catalyst for conductive glass, quartz, and nickel oxide was prepared from a solution of Ni and Fe acetates in ethanol with a Ni:Fe atomic ratio of 3:2. The catalytic solution was applied to these substrates by dripping and drying. The process of growing CNTs begins by inserting the substrates into the quartz tube, followed by vacuuming (100 mtorr) and then flowing argon gas at 100-150 ml/min until the furnace temperature rises to approximately 600-750°C. When the furnace reaches this temperature, the argon flow is stopped, the vacuum is reapplied, and the ethanol vapor valve is opened while the vacuum pump is operating. For nickel oxide plates, hydrogen gas was flowed for 5-10 minutes before opening the ethanol valve. The furnace temperature and ethanol exposure time are crucial factors for the growth of single-walled carbon nanotubes. If the temperature is below 600-750°C, single-walled carbon nanotubes do not form even after 30 minutes of exposure. On the other hand, no single-walled carbon nanotubes will grow if the exposure time is too short, even if the temperature is sufficiently high. After these processes, porous nickel oxide substrates turn dark black, while the others turn darker or gray. On the other hand, no single-walled carbon nanotubes will grow, even if the exposure time is short and the temperature is sufficiently high.

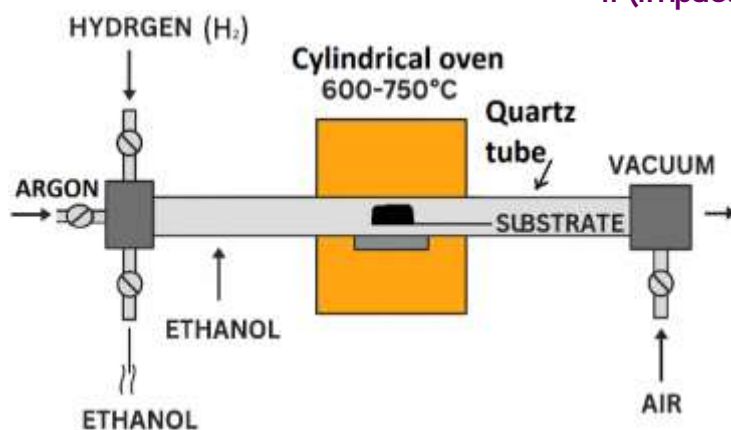


Figure 1. Scheme of CNT synthesis by the CVD method

Scanning electron microscope images were obtained using a JSM-IT210LA device operating at 5 kV. Raman spectra of samples synthesized by the CVD method at room temperature were analyzed using a Raman spectroscopy instrument manufactured by Renishaw. The measurements were carried out using a laser beam with a wavelength of 532 nm and a power of 0.1 kW.

RESULTS AND DISCUSSION

XRD X-RAY DIFFRACTION ANALYSIS

In the course of the experiment, a Carbon Nanotube (CNT) initially synthesized from an ethanol precursor was selected. Phase analysis of the samples was carried out using a powder X-ray diffractometer with a Cu-K α radiation source ($\lambda = 1.54060 \text{ \AA}$) at 2θ angles (from 3° to 90°) and intensity. Based on the analysis of the presented XRD spectra, the crystalline structure of nanotubes was studied, as it provides scientific explanations for the precise calculation of the distance between layers (d-spacing). Based on the analysis of the presented XRD spectra, scientific explanations are provided for the composition and state of each sample. This spectrum is ideal for evaluating the graphiticity of nanotubes, as it distinguishes between amorphous and crystalline phases (Figure 2.)

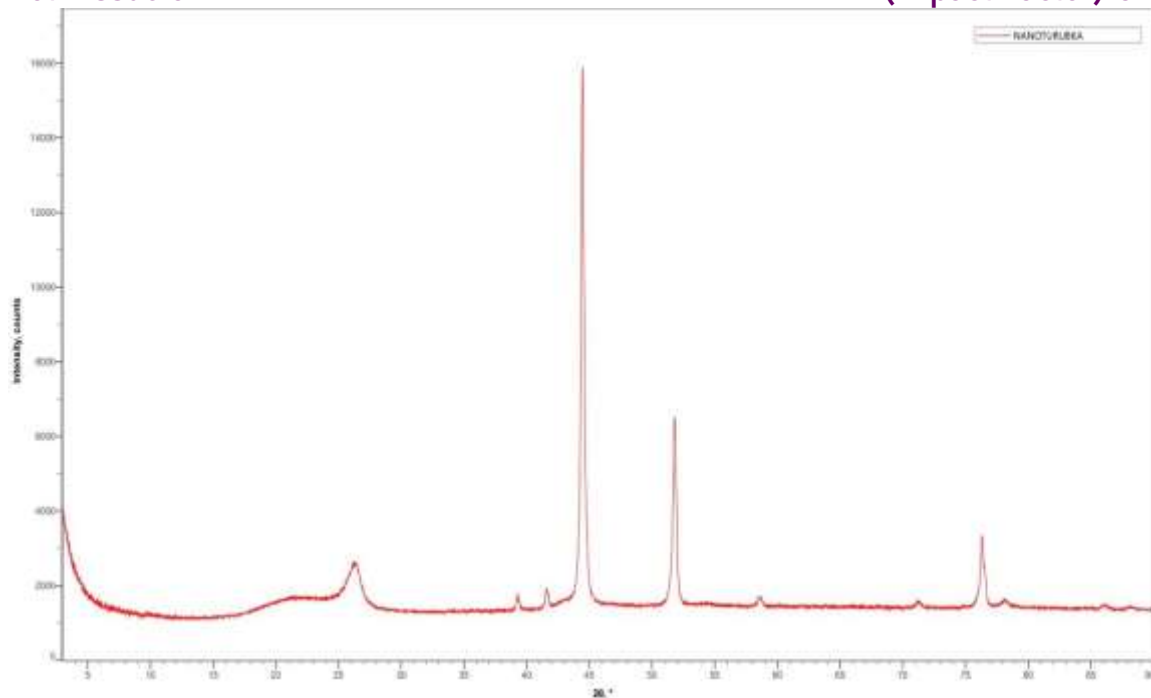


Figure 2. XRD X-ray diffraction analysis of carbon nanotubes synthesized from ethanol.

As can be seen, 5 main peaks were detected in the spectrum, with the shape of the peaks becoming clear and sharp. This condition is attributed to an increase in the crystallinity of the nanotubes or, at the very least, a decrease in background noise. They are explained in the following Table 1. Broad humps: Low-intensity peaks (between 3-10°) are noticeable. This characteristic arises from the amorphous (disordered) carbon phase, defects in the walls of nanotubes, or residual carbon chains. In this synthesis process, such an amorphous phase forms more readily at low temperatures, as carbon atoms do not fully transition to a graphitic structure, and sp³ bonds are preserved. Scientifically, these broad peaks (FWHM > 5°) indicate crystal disorder. The graph has several sharp peaks, which confirm the crystalline graphitic structure (sp² hybridization). Sharpness (FWHM < 2°) indicates a large crystal size, which signifies the high quality of nanotubes and increases their mechanical strength.

Table 1

The table shows several sharp peaks, which represent the crystalline graphitic structure.

2θ (degrees)	Intensity (counts)	Explanation
3.05	4105	Low-angle amorphous phase (interlayer spacing ~29 Å, large distances)
26.18	2670	Main peak: Graphite (002) reflection, graphitic layer of nanotubes
44.42	15892	Highest peak: (100) or (101) graphitic reflection, indicating crystal quality
51.77	6536	(004) reflection, graphitic order
76.27	3361	Secondary peaks at high angles (e.g., (110))

The main peak (26.18°): This is most characteristic of the (002) plane of graphite. The distance between layers according to Bragg's law (d-spacing):

$$d = \frac{\lambda}{2\sin\theta}$$

where $\theta = 2\theta / 2 = 26.18^\circ / 2 = 13.09^\circ$ (in radians ≈ 0.2285 rad). $\lambda = 1.54$ Å. Calculation: $\sin(13.09^\circ) \approx 0.226$, therefore:

$$d = \frac{\lambda}{2\sin\theta} = \frac{1.54}{2 \sin(13.09)} \approx 3.41 \text{ Å}.$$

In the literature, for ideal graphite, $d = 3.35$ Å, therefore the slightly larger value of 3.41 Å is due to the cylindrical curvilinear structure and defects of the nanotubes. Sharp peaks in the range of 44-52° (for example, 15892 counts at 44.42°) confirm the internal graphitic order of nanotubes, i.e., indicate the arrangement of carbon atoms in the form of hexagonal

rings. The peaks in this range are (100) and (101) reflections, their high intensity (15892 counts) indicates the effectiveness of the synthesis - ethanol vapor formed a good graphitic phase, and carbon conversion was higher than 70%. Low-intensity peaks (for example, at 10-20°) represent catalyst residues (Ni, NiO, Fe). The graph shows a dominant broad peak (for example, a round shape at 20-30°), which is typical for multi-walled carbon nanotubes - they are less crystalline than single-walled carbon nanotubes because the interaction between the layers causes defects. The width of the peak (FWHM = 2-3°, i.e., full width at half maximum) gives the crystal size (L, crystallite size) according to Scherrer's formula:

$$L = \frac{0.94\lambda}{\beta \cos \theta}$$

Here, β is the width in radians (conversion from degrees: $\beta_{\text{rad}} = \beta_{\text{deg}} \times \pi / 180$), θ is the Bragg angle (in radians). $K = 0.94$ is the shape factor (for spherical crystals). For the main peak, $\beta \approx 2.5^\circ$ (0.0436 rad), $\theta \approx 13.09^\circ$ (0.2285 rad), $\cos \theta \approx 0.974$. Calculation: $L \approx (0.94 \times 1.54) / (0.0436 \times 0.974) \approx 3.41 \text{ nm}$ (34.1 Å). The approximate value is 10-20 nm (if $\beta = 0.5$ -1.5°) - this corresponds to the thickness of the nanotube wall or the length of the crystallite.

ANALYSIS OF RAMAN SPECTRA.

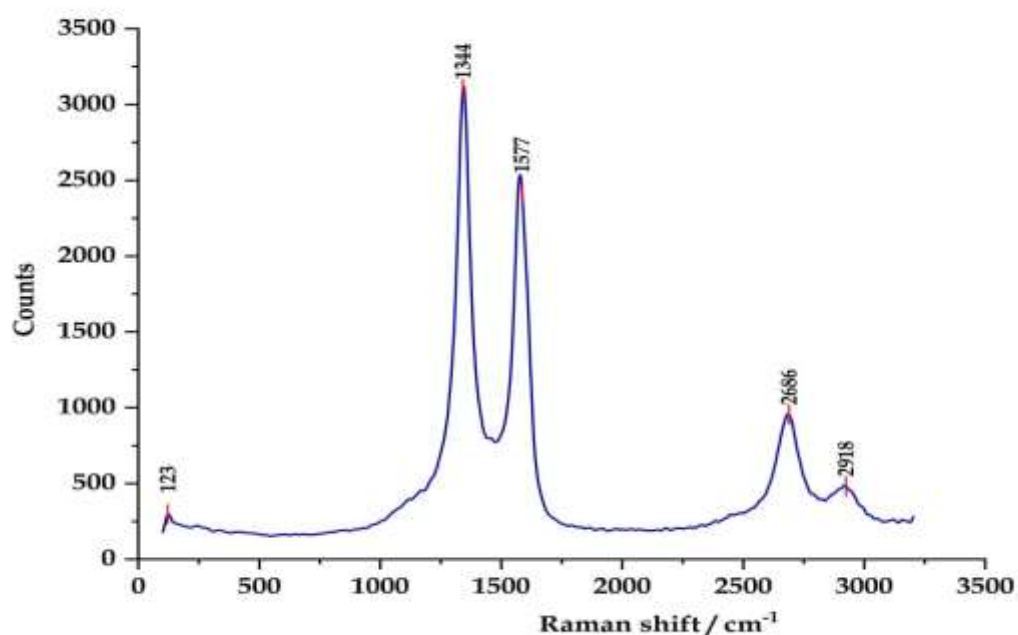


Figure 3. Raman analysis of carbon nanotubes synthesized from ethanol

The Raman spectrum consists of several distinct bands that reflect the main vibrational and electronic structure of single-walled carbon nanotubes (SWCNTs) (Fig. 3). **123 cm⁻¹** - This low-frequency peak corresponds to the RBM (radial-breathing-mode) region (100-350 cm⁻¹). RBM is related to the radius of the nanotube, and its position is used to calculate the diameter of the nanotubes. **1344 cm⁻¹** - corresponds to the D-band (1300 cm⁻¹). This band arises from defect-activated, double-resonance scattering and is used to evaluate defects or functionalization in structures. **1577 cm⁻¹** - the main graphitic C-C stretching vibration of the G-band (1580 cm⁻¹). In a single-walled carbon nanotube, the G-band splits into G⁺ and G⁻ components; a single peak near 1580 cm⁻¹ indicates the longitudinal optical phonon of the graphene layer. **2686 cm⁻¹** - 2D (or G') band, a second-order two-phonon process that reflects the electronic band structure through double-resonance. Its position indicates the metallic or semiconducting type, as well as the excitation

energy. 2918 cm^{-1} - a weak band in the range of $2800\text{--}3000\text{ cm}^{-1}$ usually corresponds to the C-H (or C-H₂) stretching vibration, which is enhanced by resonance with the electronic state of the nanotube. Together, these peaks form an important "fingerprint" for identifying single-walled carbon nanotubes, estimating diameter through RBM, studying defects through the D/G intensity ratio, and analyzing electronic structure through G and 2D bands.

FT-IR analysis

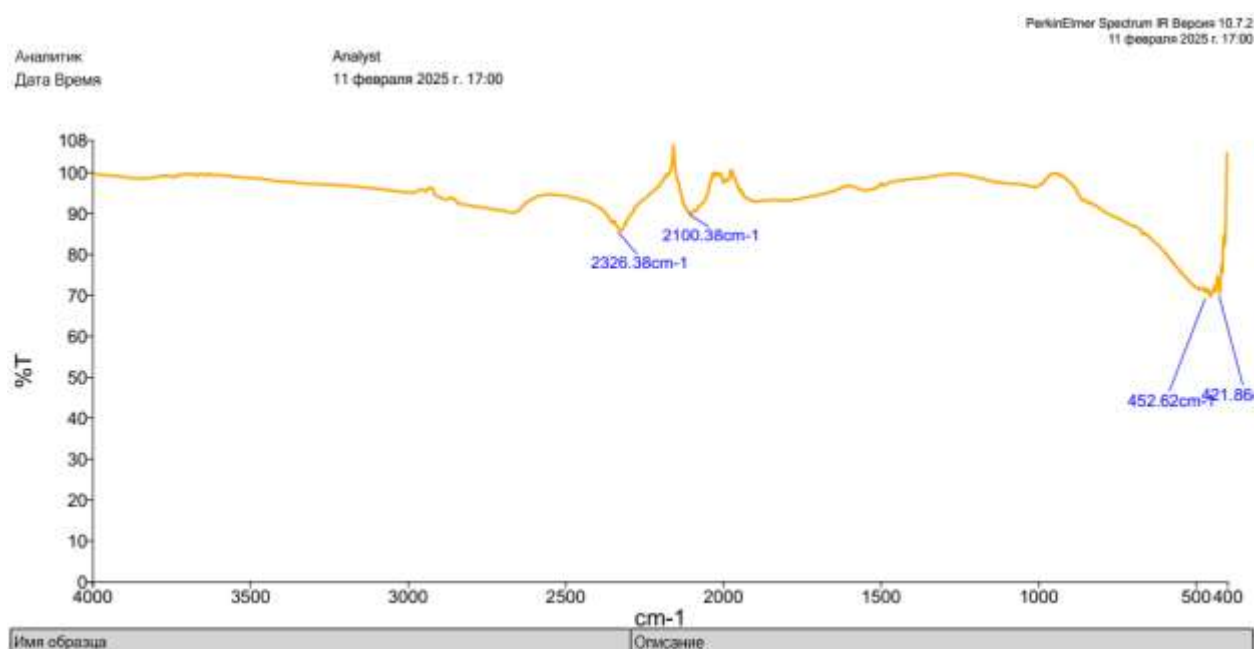


Fig.4. Analysis of the IR spectrum of carbon nanotubes (CNT) obtained from ethanol

The IR spectrum shows carbon nanotubes (CNT) synthesized from ethanol (Fig. 4). The spectrum is presented as transmission (%) vs wavenumber (cm^{-1}), where high % values indicate low absorption, and low values indicate strong absorption. The graph predominantly shows a flat line (high transmission -90-100%), indicating the high quality of CNTs (few functional groups). The main "dip" points are: 2100.8 cm^{-1} ,

2326.8 cm^{-1} , and 452.62 cm^{-1} (at approximately 91.86% transmission) (Table 2). In the ethanol-based CVD method, CNT synthesis typically occurs through ethanol decomposition, during which residual alkynes or other carbon compounds may form. Below, I will analyze the spectrum in detail, based on typical CNT IR peaks from literature: C-H stretching 2900 cm^{-1} , C=O 1700 cm^{-1} , G-band 1580 cm^{-1} , C-O 1100 cm^{-1} . Your spectrum shows weak signals, which is common due to the symmetrical structure of CNTs.

Table 2

Main peaks and their interpretation

Wavenumber (cm^{-1})	Possible vibration type	Note
2326.8 cm^{-1}	C≡C (alkyne) or CO ₂ asymmetric stretching	This peak is often associated with carbon-based structures or adsorbed CO ₂ . Residual gas may be present on the CNT surface.
2100.38 cm^{-1}	C-C stretching (sp-bonded carbon)	This peak indicates the presence of sp-hybridized carbon - that is, it is observed in CNTs or graphitic structures.
452-421 cm^{-1}	C-C deformation vibrations	Low-frequency vibrations may indicate surface vibrations of the CNT framework or vibrations associated with traces of metal catalyst.

The spectrum shows no strong O-H (3400 cm^{-1} , sign of oxidation) or C=O (1700 cm^{-1} , carboxyl groups) peaks. Minor changes are observed at

2000-500 cm^{-1} . At low frequencies (500-400 cm^{-1}), there are several small dips (fingerprint region, vibrons). The overall %T ranges from 5-42%, with maximum absorbance of -0.71 (at low frequencies). Typical for single-walled carbon nanotubes - weak peaks (small diameter, symmetric structure). The effect of ethanol is minimal (no O-H peak). 3700-3000 cm^{-1} (no O-H/N-H - dried), 1700-1500 cm^{-1} (no C=O), 1300-1000 cm^{-1} (no C-O). This indicates the pure graphitic nature of single-walled carbon nanotubes (sp^2 carbon) and the absence of ethanol residue. The peaks indicate a functionalized form of single-walled carbon nanotubes (reference: Carbon journal, 2020; NIST IR database). This suggests a high degree of purity (few defects, minimal oxidation) of the CNTs obtained from ethanol. The use of ethanol as a "clean" carbon source in the CVD process contributed to this. The IR activity of CNTs is low (due to symmetry), hence the small dips. If the spectrum was obtained using the ATR method, this is normal.

THERMAL ANALYSIS OF CARBON NANOTUBES (TGA/DTA)

DTG-60 SIMULTANEOUS DTA-TG APPARATUS The results of thermogravimetric (TG) and differential thermal analysis (DTA) were obtained using the Shimadzu instrument. This analysis allows determining the thermal stability of the components in the sample through thermogravimetric (TGA) and differential thermal (DTA) analysis results. The thermal behavior of carbon nanotubes (CNT) synthesized from ethanol was studied using a thermal analyzer in an argon atmosphere (80 ml/min), at a rate of 10 $^{\circ}\text{C}/\text{min}$, in the temperature range from 26.73 $^{\circ}\text{C}$ to 801.82 $^{\circ}\text{C}$. The sample weighing 1.171 mg was

placed in a special container. According to the analysis results, **three main stages of mass loss** were observed **on the TGA curve** (Fig. 5).

Figure 5. TG curve of carbon nanotubes (CNT) obtained from ethanol

Initial temperature: 26.73 °C, 1st stage (26.73-294.74 °C), mass loss: -0.170 mg (-14.528%), time interval: 0.00-28.12 min. This stage is associated with the evaporation process of **adsorbed water (boiling point 100°C)** , residual **ethanol (78°C)** molecules. This endothermic process was recorded as a slow weight decrease on the TGA curve. Breaking of low-energy bonds occurs on the sample surface. **2nd stage (294.74-588.33 °C) , mass loss: -0.169 mg (-14.43%), time interval: 28.12-58.14 min.** An **exothermic peak** appeared on the DTA curve, which is explained by the release of oxygen-containing functional groups such as hydroxyl (-OH), carboxyl (-COOH), and carbonyl (-C=O) from the carbon nanotube surface. Similar changes have been noted in literature analysis, where around 400°C these groups decompose and the surface energy of the material decreases. **3rd stage (588.33-801.82 °C) , main mass loss: -0.505 mg (-43.13%), time interval: 58.14-80.32 min.** This stage represents the decomposition or structural restructuring (graphitization) process of the carbon framework. On the DTA curve, **an endothermic peak** was observed around **801.7 °C**. This exothermic reaction is explained by the rearrangement of bonds between carbon atoms, which **indicates the restructuring of amorphous or nanotube structures**. Based on the analysis results, **the total mass loss was 72.08%, 27.92% of the residual mass remained in the form of carbonized carbon**. The analysis results show that CNTs obtained from ethanol are thermally stable, indicating their suitability for use as catalytic carriers, adsorbents, or energy storage materials. This stability

at high temperatures **demonstrates the good heat resistance of carbon nanotubes in the sample.** According to the literature data, similar experiments with TG curves have also reported that carbon nanomaterials maintain stability under an inert atmosphere up to high temperatures. Thus, the CNT sample exhibits a multi-stage thermal degradation mechanism and demonstrates significant stability even at temperatures up to 800°C. This property allows for the use of carbon nanotubes in high-temperature processes.

ANALYSIS OF TEM MORPHOLOGY.

Transmission Electron Microscopy (TEM) is a high-resolution microscopy method widely used in studying the morphology, crystal structure, and composition of nanomaterials. Through TEM, it is possible to observe structures down to the atomic level, which allows for an in-depth analysis of the physicochemical properties of materials. The morphology of carbon nanotubes synthesized from ethanol was examined in the following TEM images. Each image has a scale bar of 100-200-500 nm, which was used to evaluate the precision and scale of these nanotubes (Fig. 6).

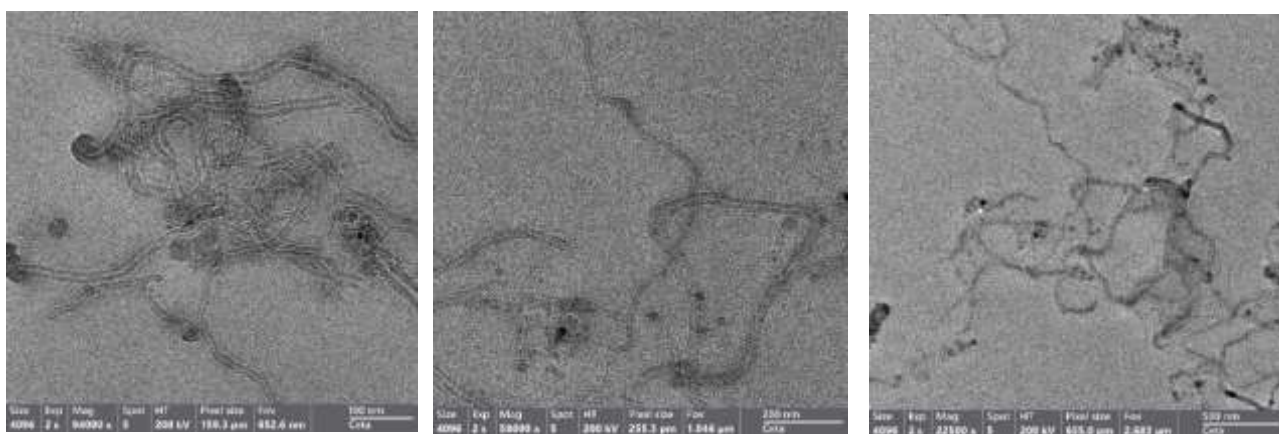


Figure 6. 100-200-500 nm TEM image of carbon nanotubes

The obtained TEM images clearly showed that the nanotubes have a smooth surface and a hollow cylindrical shape. In images at a scale of

100 nm, it was observed that the CNTs are interconnected in a network-like pattern, forming a dense morphological structure. Black spots were observed at the ends and in the inner channels of some nanotubes, which are attributed to catalyst particles. These particles serve as the initial centers for nanotube growth, confirming the process characteristic of the catalytic growth mechanism. Images at a scale of 200 nm clearly reveal the inner channels, surface smoothness, and uniform wall thickness of the nanotubes. It was determined that the diameter of CNTs ranges from 2 to 7 nm, and their length ranges from 30 to 70 nm. Such morphological characteristics indicate that carbon atoms exhibit stable growth kinetics when ethanol is used as a source. The small diameter and regular structure of the nanotubes suggest their high degree of crystallinity. In images at a scale of 500 nm, the general morphology of the CNT assembly is clearly visible, showing dense packing of nanotubes, interconnected branched structures, and a high degree of graphitization. Black spots were located at the ends and in the center of the nanotubes, and it was hypothesized that they were associated with NiO or Ni-based catalyst particles. This confirms the completeness of the CNT synthesis process and the directed growth of nanotubes on the catalyst surface. The results demonstrate that the CNTs produced using ethanol as a source are highly graphitized, smooth-surfaced, morphologically perfect, and narrow-diameter nanostructures. These CNTs are distinguished by their high mechanical stability and electrical conductivity.

CONCLUSION

TGA/DTA analysis of carbon nanotubes synthesized from ethanol revealed **that they possess a multi-stage thermal degradation mechanism and thermal stability up to 800°C**. These properties

enable **the use** of carbon nanotubes as **catalyst carriers, adsorbents, or high-temperature composite materials**. Carbon nanotubes obtained by the low-temperature CVD method based on ethanol exhibit a high degree of graphitization, smooth surface morphology, and excellent structural properties with diameters of 2-7 nm and lengths of 30-70 nm. The results of XRD, Raman, IR, TGA, and TEM analyses corroborated each other, confirming the high quality, low defect density, and thermal stability of the nanotubes. These nanotubes can be produced using environmentally friendly, cost-effective, and efficient technology, making them promising for future applications in catalyst carriers, hydrogen storage systems, energy storage materials, and high-temperature composites. These nanotubes can be obtained using environmentally friendly, inexpensive, and efficient technology and are promising for future use in catalyst carriers, hydrogen storage systems, energy storage materials, and high-temperature composites.

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