Applied Chemical Engineering (2026) Volume 9 Issue 4 ISSN: 2578-2010 (O)
doi: 10.59429/ace.v9i4.5951

RESEARCH ARTICLE

Synthesis and Diagnosis of New Amides Composites a doped with nanotube assessing Their bacterial activity

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ABSTRACT

Covalent bonding composite are formed in amide bonds between the wall of oxidized nanotubes that are covalently bonded by the interaction of the carboxyl groups of nanotubes with the amine groups of other amino compounds and the formation of highly effective Composite against some bacteria. It can also have other applications. The composite Create (K3) (N- (benzathiazol-2-yl) nanotube hydrazide. From the treatment of oxidized nanotubes (K1) with some derivatives amino compounds (K2), The Synthezid composite was subjected to a comprehensive and rigorous characterization to evaluate its structural, morphological, and thermal properties. Fourier-transform infrared spectroscopy (FT-IR) was employed to identify the functional groups and confirm the chemical cross-linking between the composite components. These findings were further supported by X-ray diffraction (XRD) analysis to investigate changes in the crystalline structure and calculate the average crystallite size. Morphologically, Scanning Electron Microscopy (SEM) was utilized to examine the surface topography and particle distribution, while Thermogravimetric Analysis (TGA) revealed the thermal stability and degradation behavior of the final product."

Keywords: Amide, Nanotube, Composite, diagnosis, Bacterial activity.

ARTICLE INFO

Received: 20 March 2026
Accepted: 25 May 2026
Available online: 02 November 2026

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1. Introduction

Important technological applications of nanomaterials, especially single-wall nanotubes, are widespread [1] and their use as chemical sensibilities are currently in full swing [2,3,4,5]. Many polymeric compounds have been integrated with carbon nanotubes, increasing their physical properties and strength [6,7]. Carbon nanotubes have also contributed to the design of devices for nanotubes [8]. Due to the improvement of the properties of carbon nanotubes, progress has been made in their various applications [9,10]. Proper binding of the nanotube groups to form complex networks of nano electronic circuits [11]. One of the most important bonding forces for nanotubes is ester and amide bonds [12]. Through the interaction of nanotubes, strong peptide covalent bonds can be formed. For the purpose of binding the amine groups of amino compounds with the carboxyl groups of carbon nanotubes, it is moderately [13,14,15,16]. It may be chemically binding to functional groups [17, 18, 19,20,21,22,23]. The result achieved in this research paper could be an innovative starting point for application.

2. Experimental

When the nanotubes are oxidized under strong acidic conditions, the carboxylic groups [24] are obtained. 100 g of pure nanotubes were added to which a ratio of 3:1 of concentrated sulfuric acid (75 ml to 25

ml of nitric acid) was added to 700 ml of distilled water and then the mixture was placed in an ultrasonic device for a time of 1.40 hours and forty-one minutes. The solution is filtered and dried at room temperature. A ratio of 4:1 (15-60) ml of the previously prepared sulfuric acid to hydrogen peroxide mixture is added to the mixture, then the mixture is placed in an ultrasonic device for a quarter of an hour. The solution filter and then collect the resulting and dry below 70 degrees in an air-free atmosphere. 50 Mg of oxidized nanotube was taken with 47 Mg of the compound and 15 mL of DMF solvent was mixed in an ultrasonic device for 50 min and then placed in an oil bath for 48 hours [25] filtered the result, washed with methanol and dried at 80 degrees under vacuum atmosphere. The purification and juice process terminates the open ends and sites of the side wall defects of the nanotubes [26,27,28]. The percentage of yield for the prepared composites K1, K3 are 92% and 87% respectively. Used ThermoIR100 Spectrometer, Thermogravimetric analysis (TGA)2050.

3. Discussion

The reaction between the amino groups of the prepared compounds and the carboxylic groups of nanotubes to form amides [29] that have the potential to inhibit some types of bacteria. The covalently formed raw material and amides have been identified using attenuated total reflection (ATR) technique in FT-IR infrared spectroscopy, which is widely used in the study of carbon nanotubes to determine functional groups [30,31]. According to the following mechanism.

The FT-IR spectrum as shown in Figure (1) appearing that the range of carbon nanotubes are variable wavelengths, for instance, at 445 cm^{-1}, the peak is strong due to the type of bonding of the C-H and N-H groups.

On the other hand, the presence of peaks (490.50 and 960.50) Cm^{-1} is due to the vibration in the carbon nanotube, and the absorption peaks of 1633.97 and 1026.55 Cm^{-1} are corresponding with stretching of the C=O group, these results confirm the preparation of the nanocarbon tube.

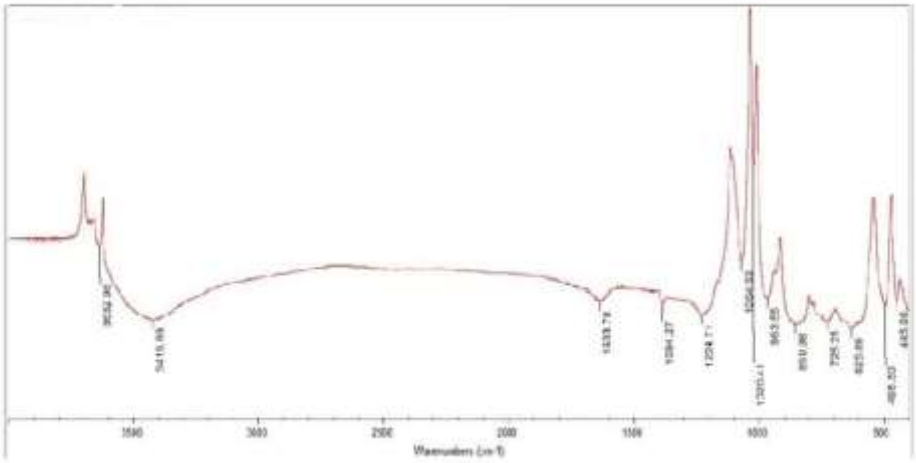


Figure 1. representing the FT-IR spectrum of the resulting compound K1

Figure (3) showing the FT-IR spectrum of the compounds (K2 and (K3).

The XRD spectrum Figure (2) showing the presence of carbon nanotubes and the presence of the peak at 2ø $43,0^0$ and $57,0^0$ which corresponds to ($100,1^0$) of the prepared nano carbon tubes [25] The crystal structures of the prepared nano carbon tubes have been proven through the sharp peak, (002) The absence of amorphous carbon has been confirmed.

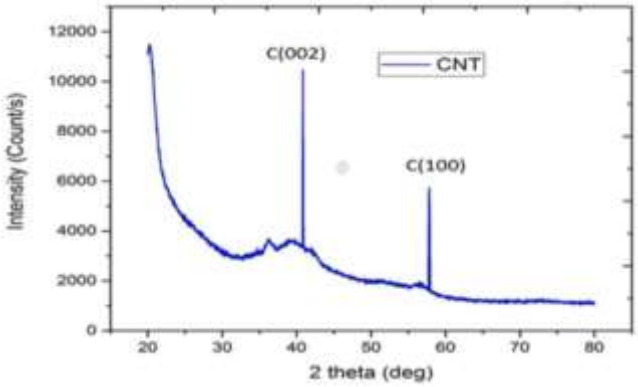


Figure 2. XRD showing the prepared nanocarbon(K1)

XRD pattern of the (K2) compound and the (K3) nanotube hybrid with the organic compound show the presence of distinct peaks that describe the slight displacement of the levels causing the structure to change as in the a peak 2ø = 26.37 The hybrid composite patterns with the nanotubes show a good crystal structure; this means that the incorporation of the carbon nanotubes has altered the structure of the amino compound with the hybrid nanotubes resulting in a change in the amine binding [32] At the specific a peak of the 2ø = 25.84 composite, it did not contribute much, but it shows multiple peaks in the extreme intensity X-ray diffraction patterns that correspond to the addition of carbon nanotubes.

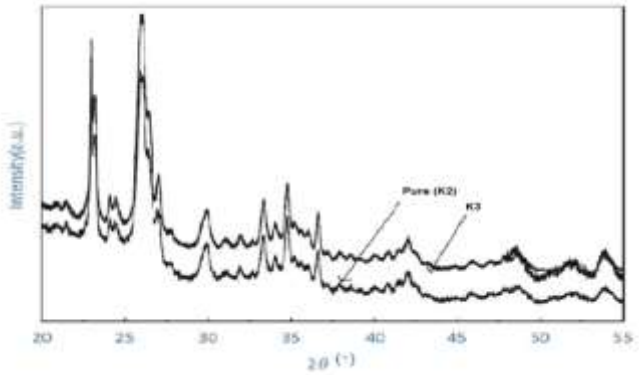


Figure 3. showing the XRD of K2 and K3

The resulting samples showed the aggregation of carbon nanotubes with a size of (m5 μ) while the molecules with a size of (1 μm) showed clearly dispersed nanotubes forming the contact area between the nanotubes and the organic matter by means of amides and is useful in the implantation process, which is important for hybrid nanocomposites.

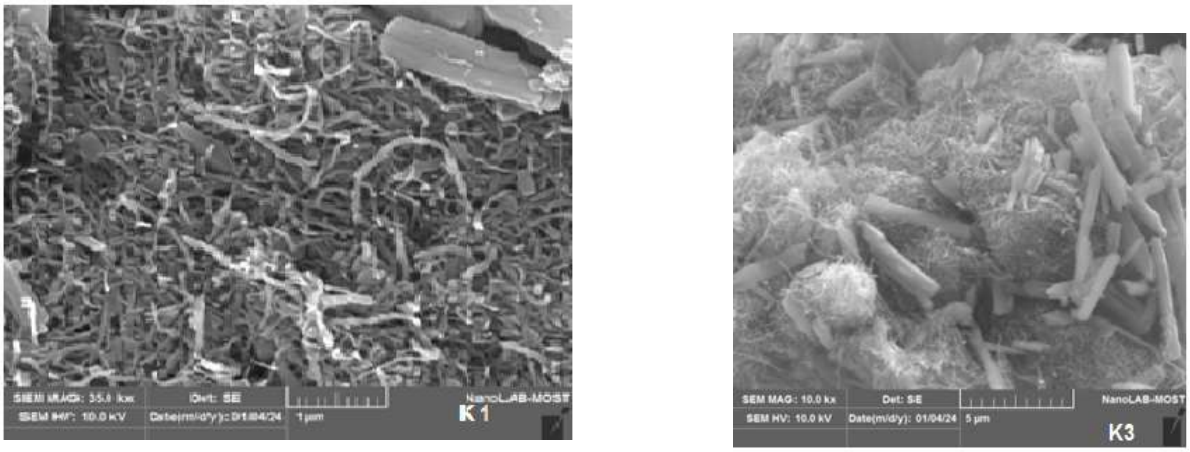


Figure 4. Representing the SEM images of the carbon nanotube (K1), (K3)

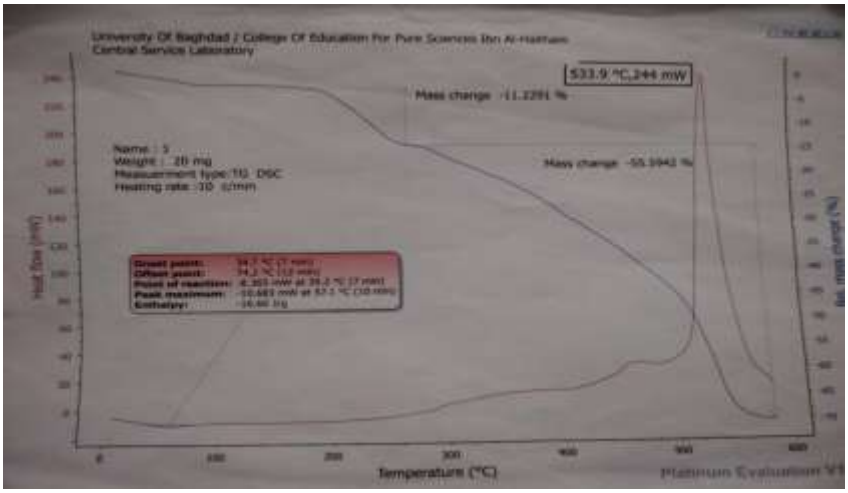


Figure 5. representing the TG, DSC of the K3 complex

4. Study of the thermal behavior of the prepared composite (K3)

The thermogravimetric curve of the prepared TG composite as shown in Figure (5) shows the thermal range of approximately 30.600C0 as well as the DSC differential thermal scan with a heating rate of 10 Degre per min using a 20 mg pattern. We observe that there are two distinct phases of mass loss indicated by the multi-stage pyrolysis behavior.

1. The first stage is the loss of mass between (200-300) which is offset by the weight loss by 11.23%, which was due to the elimination of moisture or from weakly related aggregates.
2. The second stage of mass loss between 600-320 C0 where the loss of mass was about 55.59%, which is due to the thermal decomposition of the volatile organic compound of the decomposition products. The loss in the compound was about 66.8%. At about 57C0, DSC showed a thermal absorption value with a change in enthalpy of 16.6J/g, which indicates the removal of physically absorbed water or a low-energy phase transition at high temperatures, especially associated with 540-530 in TG confirming that this process is related to the disintegration of the compound structure.

5. Biological Activity

The biological activity of the prepared composite (K3) was measured compared to the organic compound (K2) and the results are shown in Table (1) which shows that the prepared compound has a clear effect against positive and negative bacteria compared to the organic compound (K2).

- Gram-positive: *Staphylococcus aureus* (ATCC 25923), *Bacillus subtilis* (ATCC 6633)

- Gram-negative: *Escherichia coli* (ATCC 25922), *Pseudomonas aeruginosa* (ATCC 27853)

Culture Conditions: Strains maintained on nutrient agar (NA) at 4°C. Fresh cultures in nutrient broth (NB) incubated at 37 ± 1°C for 18-24 h, standardized to OD600 = 0.5 (~10^8 CFU/mL).

Testing Method: Agar well diffusion (CLSI M02-A12). MHA plates inoculated with 100 µL bacterial suspension. Wells (6 mm) filled with 50 µL test solution (100%, 50%, 25% in DMSO). Incubation: 37°C, 24 h, aerobic.

Controls: Positive (ciprofloxacin 5 µg/disc, gentamicin 10 µg/disc); Negative (DMSO 50 µL); Growth control.

Interpretation: No activity (-): <6 mm; Weak (+): 6-10 mm; Moderate (++): 11-15 mm; Strong (+++): 16-20 mm; Very strong (++++): 21-25 mm; Extremely strong (+++++): >25 mm.

Table 1. showing the differences between (K2and K3)

Concentration	25%	50%	100%
K2	+	+	++
K3	+++	++++	+++++

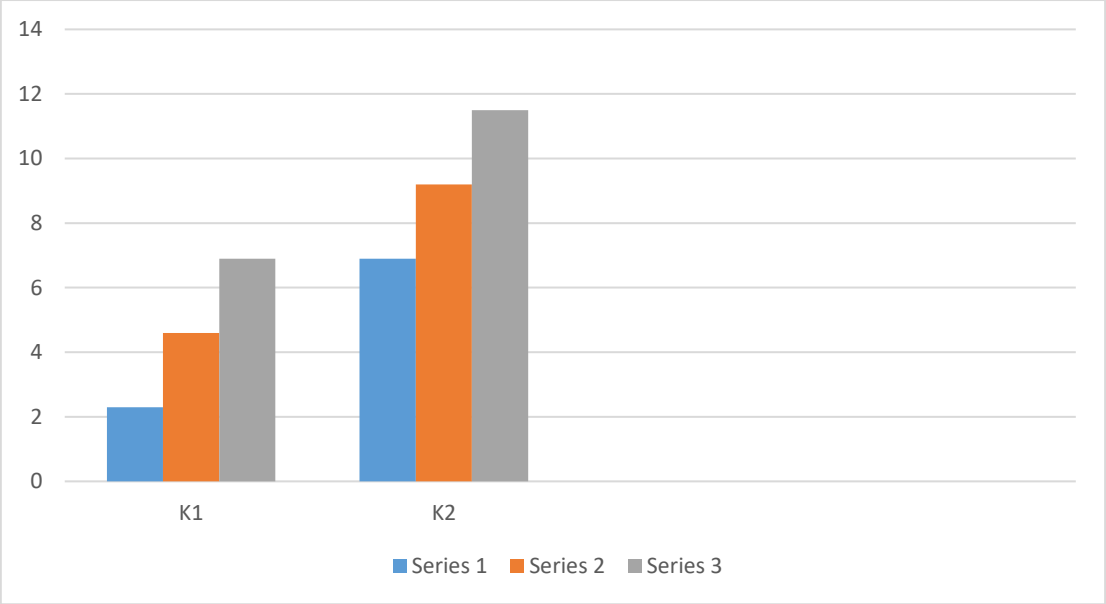


Figure 6. showing the comparative antibacterial activity of K2 and K3 against tested bacterial strains at concentrations of 100%, 50%, and 25%.

The significantly enhanced antibacterial activity of composite K3 compared to K2 can be explained by a multi-faceted mechanism:

(1) Electrostatic Interaction: Carboxylic acid and amide groups on K3 interact with positively charged bacterial surface components (lipoteichoic acids in Gram-positive, divalent cations in Gram-negative LPS), facilitating initial adsorption.

(2) Physical Membrane Disruption (Nano-Knife Effect): The rigid cylindrical nanotubes (10-20 nm diameter) physically pierce the phospholipid bilayer, causing irreparable membrane damage and intracellular content leakage.

(3) Oxidative Stress: Functionalized CNTs generate ROS (superoxide, hydroxyl radicals, hydrogen peroxide) leading to lipid peroxidation, protein oxidation, and DNA damage.

(4) Metabolic Inhibition: Amide groups hydrogen-bond with active sites of respiratory enzymes (ATP synthase, NADH dehydrogenase), disrupting the electron transport chain and depleting cellular ATP.

(5) DNA Intercalation: The planar benzothiazole ring intercalates between DNA base pairs, interfering with replication and transcription.

(6) Synergistic Effect: CNTs act as nanocarriers concentrating the amide agent at the bacterial surface, while amide groups improve nanotube dispersibility - the combined effect exceeds individual contributions.

(7) Differential Gram Response: Against Gram-positive bacteria, nanotubes directly pierce the thick peptidoglycan; against Gram-negative bacteria, carboxylate/amide groups chelate LPS-stabilizing Ca^{2+}/Mg^{2+} ions, destabilizing the outer membrane for penetration.

6. Conclusion

The synthesized composite exhibits significant potential as abroad spectrum antibacterial agent, demonstrating promising inhibitory activity against both Gram – positive and Gram- negative bacterial strain. These findings position the composite as a viable Candidate for further pharmaceutical development.

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