

The study of synthesis and functionalized single-walled carbon nanotubes with amide group

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Abstract This study includes of syntheses and characteristics functionalized single-walled carbon nanotubes (SWCNTs) with amide group using thionyl chloride and NH_3 . First SWCNTs in H_2SO_4 and HNO_3 , solved and the solution obtained ultrasound was to reach the equilibrium temperature to functionalization of carboxylate single-walled carbon nanotubes (SWCNT-COOH). Then using thionyl chloride with (SOCl_2) and DMF the mixture was refluxing. SWCNT-COCl was obtained from the previous step with ammonia (NH_3), and DMF as solvent, and the mixture was refluxing. The black solid obtained was placed overnight in the oven to dry. Carbon nanotubes were expected at this stage to have a functional group CONH_2 . All new chemical bonding products were identified by FT-IR and observations using scanning electron microscopy (SEM) were confirmed. SWCNT-COOH functionalized carbon nanotubes have a relatively smooth surface and thin Stowe and the SEM image of the SWCNT- NH_2 ; a thin layer of hope is clearly placed on the surface of SWCNT-COOH and its diameter is increased.

Keywords Single-walled carbon nanotubes (SWCNTs) · Amide · Chemical modified

Introduction

Grafit, diamond, opals and carbon nanotubes (CNTs) are diverse feature of carbons but the word Nanotubes usually

signifies carbon nanotubes that have attracted worldwide attention [1].

Carbon nanotubes (CNTs) are new [2]. From the time of their discovery in 1991 by Sami Iijima [3, 4], (CNTs) Carbon nanotubes have attracted much attention due to specific characteristics such as [3, 5, 6], thermal [7], mechanical, optical and chemical properties [3, 5, 6].

However, its poor solubility in many solvents and chemicals and weakened biocompatibility greatly hinder CNT applications in actual systems [8–12]. Carbon nanotubes (CNTs) are of two types: single-walled (SWCNTs) and multi-walled (MWCNTs) [13]. Se espera que los nanotubos químicamente funcional izado pared lateral para agregar nuevas funciones a su comportamiento original. [14, 15]. Also, Oxidation of the nanotubes has been widely reported [16–26]. Because of a weak solubilised inorganic solvents water and solvents, SWNTs oxygen possessing carboxyl acid moieties as the starting materials for functionalized substrates have been used in many studies [14]. That means that these procedures of correction of shortcomings of in SWNTs are considered heavy [15]. Epoxy resins are well established as thermosetting matrices of advanced nanocomposites, displaying a series of promising characteristics for a wide range of application [27–30].

During the oxidation of the surface features, new functional groups (e.g., COOH, COCl, CNH_2) change [16–26].

Functionalization of Carbon nanotubes to the other molecules was only be achieved to the solution problems which was the lack of interfacial adhesion and critical for load to the transfer in the nanocomposites [31–39]. The conventional method to functionalize Carbon nanotubes with functionalization groups amino was generally a complicated process involving much reaction time, toxic materials such as SOCl_2 , and strictly controlled reaction conditions [40–42]. The using of amino group involved

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reaction of the specific characteristics of the amino groups [5, 43]. Carboxylic acid groups and amino groups played a major role in the functional adaptation, cell tend, peptide synthesis, and ammonia games, sometimes in combination with other gases, which is often considered an effective precursor employing amine functionality on carbon and other materials, increasing their hydrophobicity and biocompatibility [43–48]. The using this date and reported with functionalization groups amino on the surface of carbon nanotubes were to limited due to the relatively difficulty of the modification. Some research studies have exploited an approach to obtain the sidewall amino-modified single-walled carbon nanotubes (SWNTs) through fluorination and subsequent reactions with terminal diamines [49]. Also hans be reported a three-step amino-modification process involving the formation of chloroanhydrid from carboxylic [50], amidation using the Hofmann rearrangement of carboxylic acid amides [51, 52].

In particular, the role of amino features to build complex structures with a combination of other functional groups comes in to play. To date, chemical modification of CNTs surfaces with amino substituents has been studied by the use of ammonia with ball-milling method [53] and substitution of fluorinated SWNTs with diamines [54]. In this research, for preparation of nanocomposite SWCNTs-CONH₂, first SWCNTs were functionalized; then they were reacted with NH₃ and reflux process.

Materials and methods

Materials

SWCNTs 79 (purity >95 %, diameter 1–2 nm, length 5–30 nm surface area 400 m²/g) and manufacturing method, catalytic chemical vapor deposition (CVD) was purchased from NanoAmor Nanostructured & Amorphous Materials, Inc. (USA). Ammoniaque 25 % with a molecular weight of 17.03, was purchased from Merck KGaA and used. All solutions were prepared with deviations of <0.1 % from the desired concentrations. All supplementary chemicals were of analytical grade and all solution mixed by deionized water. Their concentrations were measured using UV-2550 UV–visible spectrophotometry (SHIMADZU, Japan).

Synthesis of SWCNT-COOH

The amount of 1 g SWCNTs in 20 ml H₂SO₄, 98 % and 20 mL HNO₃, 70 % will solve the solution obtained; the ultrasound took 3 h to reach the equilibrium temperature. After Sonic the substances dissolved in deionized water were added and the pH was measured. As far as pH

SWCNT's, dissolved in deionized water to reach pH (i.e., pH 7). SWCNTs, filtered and the filter that was placed overnight a vacuum for dry. The carbon nanotubes used in this step is a functional group COOH.

Synthesis of SWCNT-COCl

SWCNT-COOH obtained from the previous step thionyl chloride with 280 ml (SOCl₂) and 3 ml of DMF and the mixture was refluxing at 60 °C for 52 h. After refluxing and cooling the mixture at room temperature with a solution containing DMF, THF and ethanol and rinsed then was filtered. The black solid obtained was for 1 day in the oven to dry. The carbon nanotubes used in this step is a functional group COCl.

Synthesis of SWCNT-CONH₂

SWCNT-COCl obtained from the previous step with 100 ml of ammonia (NH₃), 25 % and 20 ml of DMF, and the mixture was refluxing at 75 °C for 96 h. After refluxing and cooling the mixture at room temperature with a solution containing DMF, THF and washed with ethanol and then filtered. The black solid obtained was placed overnight in the oven to dry. Carbon nanotubes are expected at this stage has a functional group CONH₂ (see Fig. 1 for reaction scheme).

Results and discussion

Characteristics of functionalized carbon nanotube

To study the functional groups, the SWCNT samples were characterized by FTIR spectroscopy. Also, a morphology study of the functionalized SWCNTs was conducted using SEM [55].

Fourier transforms infrared spectroscopy studies

The FT-IR spectroscopy studies provide clear evidence for the functionalization of the carbon nanotubes [56]. FTIR spectroscopy studies were used to identify the chemical groups that were attached to SWCNTs.

In the FTIR measurement, the amount of carbon nanotubes added to KBr must be strictly controlled since the black carbon nanotubes absorb infrared rays [57]. Pure nanotube moderate or strong absorption in the region of 1,450–1,650 cm⁻¹ often represents an aromatic ring. The bands at 1,600–1,660 demonstrated C–H stretching absorption peak [58].

The FT-IR spectra showed Fig. 2. Absorption bands in the range 3,000–3,500 cm⁻¹ (3,440) related to

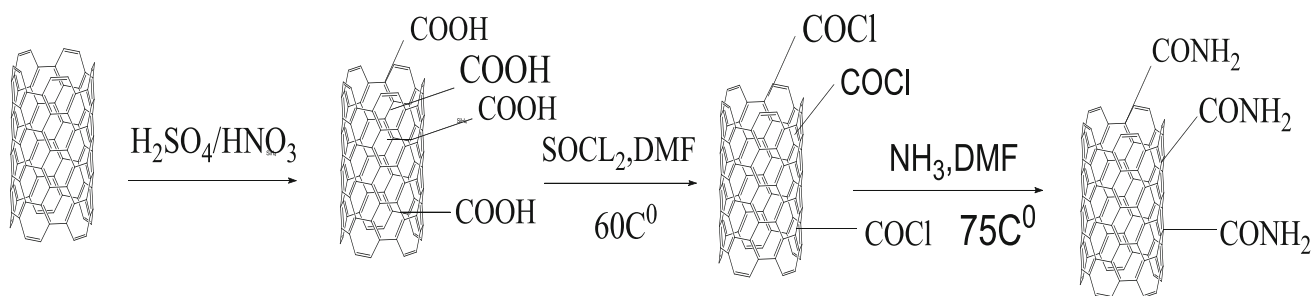


Fig. 1 Reaction scheme for functionalization

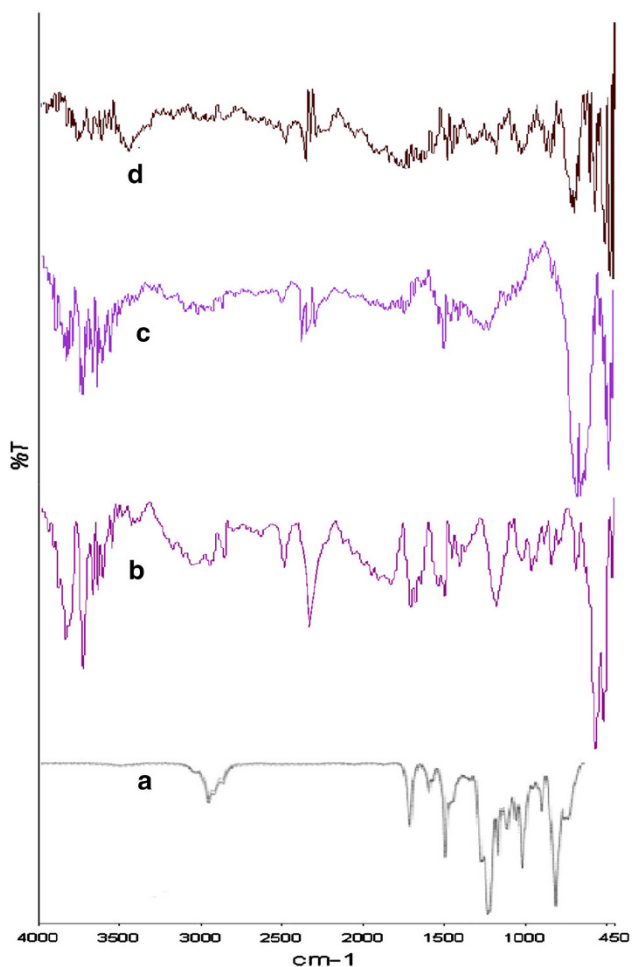


Fig. 2 FTIR spectra for **a** pure SWCNT, **b** SWCNT-COOH, **c** SWCNT-COCl and **d** SWCNT-NH₂ functionalized

presence of the hydroxyl group (O–H Tensional vibration) were observed on the surface of oxidized SWCNTs, which may be caused due to either ambient moisture or purification initial SWCNTs. The presence of carbonyl group C=O confirmed the peaks around 1,737 cm⁻¹ (C=O stretching) and peaks around 1,311 cm⁻¹ related to (C–O stretching vibration) of the –COOH carboxylic acid group (Fig. 1a). This result

clearly showed presence of carboxylic acid groups on the surface of oxidized SWCNTs [59].

The converting of the carboxylic acid groups (SWCNT-COOH) into the acyl chloride intermediates (SWCNT-COCl) by treatment with thionyl chloride was confirmed by the appearance of peak near 1,778 cm⁻¹ stretching incurve [60].

The broad band in 1,630–1,380 cm⁻¹, is due to the (C=O stretch) of the amide and peaks at 3,180, 3,480 and 1,640 cm⁻¹ for NH (N–H Tensional) of amid group. A sharp peak 1,098 cm⁻¹ was attributed to (C–N) amines [59].

Scanning electron microscopy (SEM)

To investigate the effect of different factors SEM observations were carried out SWCNT-NH₂. Morphology, distribution and orientation of the SWCNT-NH₂ with SEM were studied.

To study the surface morphology of the samples, plates and Share nanocomposites prepared by hot-pressing were broken in liquid nitrogen. Then sample was covered with a thin layer of gold using SEM study.

The difference in dispersion and adhesion can be clearly seen. SEM image of the SWCNT surface was smooth and uniform, and the SEM image of SWCNT-COOH was observed. Carbon nanotubes have been found that the surface was relatively smooth and thin, and the SEM image of the SWCNT-NH, a thin layer of organic compounds (amide) is clearly on the surface of SWCNT-COOH (thicker part) has been its diameter was increased.

Conclusion

In this research, we have dealt successfully introduced into the NH₂ in nanotubes. Structural analysis FT-IR and SEM image of the SWCNT surface is smooth and uniform, and the SEM image of SWCNT-COOH was observed that the surface of carbon nanotubes is a significant factor in the relatively flat and thin and the

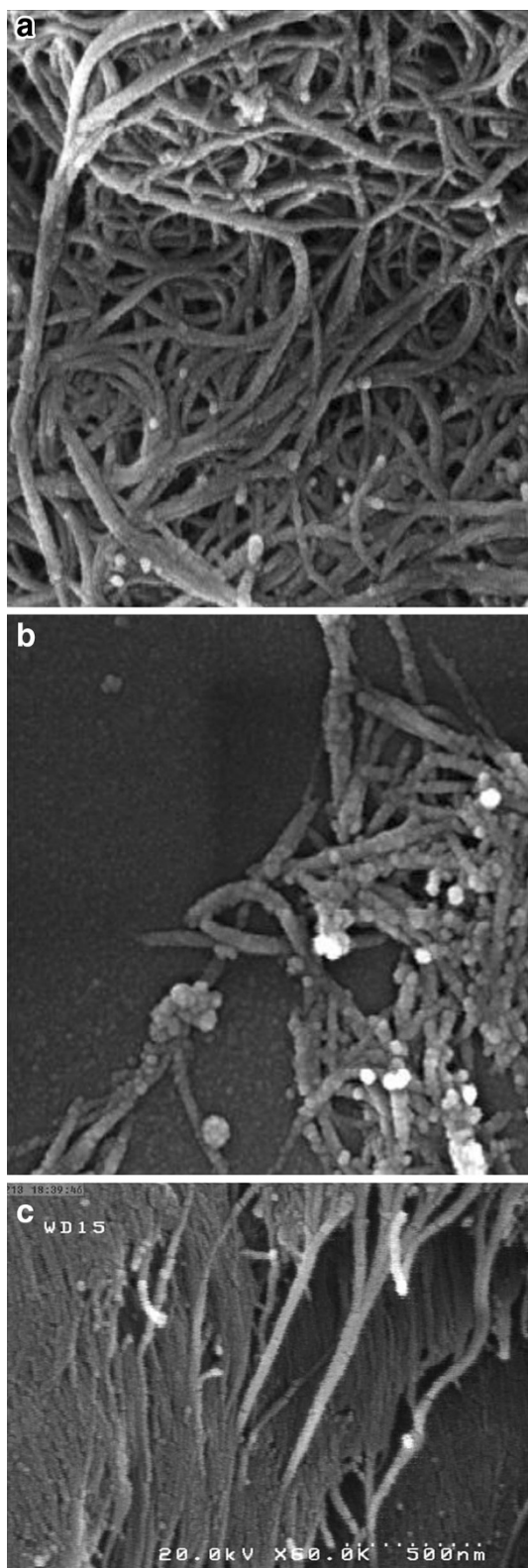


Fig. 3 SEM images of SWCNT functionalization. **a** SEM images of SWCNTs, **b** SEM images of SWCNT-COOH, **c** SEM images of SWCNT-CONH₂

SEM image of the SWCNT-NH₂, a thin layer of organic compounds (amide) is clearly on the surface of SWCNT-COOH (thicker part) has been increased in diameter. The SEM images of the carbon nanotubes, thus having to confirm.

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