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Full range of wettability through surface modification of single-wall carbon nanotubes by photo-initiated chemical vapour deposition

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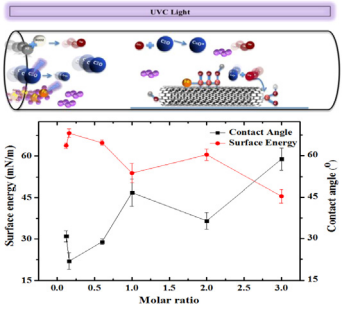
HIGHLIGHTS

- Systematic study of the functionalization of carbon nanotubes over a wide range of properties.
- Use of a solvent-free, gas-phase process initiated by UV light.
- Operation at normal temperature and pressure for increased scalability.
- Detailed chemical characterization of the modified materials, including tailored wettability.

GRAPHICAL ABSTRACT



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ABSTRACT

Single-wall carbon nanotubes (SWCNTs) have various remarkable properties, which make them a promising candidate for many applications. However, their inherent hydrophobicity has limited their commercial use in optical, biological, and electrical applications. Photo-initiated chemical vapour deposition (PICVD) using syngas is proposed as a novel, affordable, and versatile method to tailor SWCNT wettability through the addition of oxygen-containing functional groups. Following PICVD surface treatment, X-ray photoelectron spectroscopy, water contact angle measurements (CA), thermogravimetric analysis, Raman spectroscopy and transmission electron microscopy confirm controlled oxygenation of the SWCNT surface. Indeed, this novel approach allows to reproducibly make SWCNTs having surfaces properties ranging from superhydrophilic (CA < 5°) to superhydrophobic (CA > 150°), including any intermediate values, by simply varying operational parameters such as molar ratio of the syngas precursor, photo-polymerization time and reactor pressure (about normal conditions).

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1. Introduction

Single wall carbon nanotubes (SWCNTs) have various properties of interest, such as high mechanical and electrical conductivity, remarkable thermal stability (up to 2800 °C under vacuum) [1,2], proportionally lower weight than steel and titanium (typical materials in bone applications) [3,4] and the highest Young's modulus among all different types of composites and nano-materials (>1–5 TPa) [1,4]. These individual properties make them promising candidates for a wide range of applications in aerospace, nanocomposites, and biomedical and tissue engineering, to name only a few [5–7]. These materials have also shown potential to be used in bone applications due to their similarities with triple helix collagen fibrils, in terms of size and shape (diameter of SWCNTs ranges between 0.7 and 1.5 nm) [8–10]. Not only can these materials enhance the mechanical properties of biomaterials [11], they can

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Nomenclature

List of abbreviations and parameters

CA	Contact Angle
DTGA	Differential Thermogravimetric Analysis
EDS	Energy Dispersive Spectroscopy
HR	High-Resolution
HR-XPS	High Resolution X-ray Photoelectron Spectroscopy
Ozone/UV	Treatment by Ozone Under UV Light
O-T-SWCNT	Ozone Treated Single-Wall Carbon Nanotubes
PICVD	Photo-Initiated Chemical Vapour Deposition
PECVD	Plasma Enhanced Chemical Vapour Deposition
PI	Photo-Initiator
P-SWCNT	Pure Single-Wall Carbon Nanotubes
SWCNT	Single-Wall Carbon Nanotubes
Syngas/PICVD	Syngas Photo-Initiated Chemical Vapour Deposition
S-T-SWCNT	Syngas Treated Single-Wall Carbon Nanotubes
TACVD	Thermal Activated Chemical Vapour Deposition
T-SWCNT	Treated Single-Wall Carbon Nanotubes
TEM	Transmission Electron Microscopy
TGA	Thermogravimetric Analysis
UV	Ultra-Violet
XPS	X-ray Photoelectron Spectroscopy
γ_{sl}	Solid-Liquid Interfacial Energy
γ_l^d	Dispersive Component of Liquid Surface Tension
γ_l^p	Polar Component of Liquid Surface Tension
γ_s	Total Free Surface Tension of Solid
γ_s^d	Dispersive Component of Solid Surface Tension
γ_s^p	Polar Component of Solid Surface Tension
θ	Liquid Contact Angle
θ_A	Advancing Contact Angle
θ_R	Receding Contact Angle

also stimulate bone regeneration [12]. Therefore, they may be effective for use in different bone substitutes, such as scaffolds and fillers.

Despite all the above-mentioned properties and potential of SWCNTs for different applications such as reinforcements in polymer nanocomposites and biomaterials synthesis, their inherent hydrophobicity and insolubility are the most challenging features that need to be addressed [2]. For example, in the case of nanocomposites, one of the most important factors that should be considered is the homogeneous dispersion of SWCNT nanofillers [7]. Untreated SWCNTs tend to aggregate due to their high surface area to volume ratio and strong van der Waals interactions; the resulting aggregation negatively overshadows mechanical, electrical and thermal properties gained by SWCNT addition [7,13–15].

Various approaches exist to modify CNT surface properties and address such surface-based problems, including surfactants, oxidation, sonication and functionalization [1]. Of these, surface functionalization has been identified as a promising approach [1]. Functionalization implies the covalent grafting of a specific chemical functional group to the surface. Intuitively, the addition of oxygen-based functional groups would seem like a viable route to decrease hydrophobicity and, possibly, make them more compatible to be used in nanocomposites [7] and bio-applications [8].

Covalent functionalization, in which functional groups (such as amino and carboxylic acid groups) are grafted onto SWCNT walls, helps overcome attractive forces to prevent agglomeration and lead to better dispersion [7,13]. Moreover, the higher reactivity of treated SWCNTs, as well as their increased interfacial bonding and load transfer with the surrounding polymer resulting from reactive functional groups (oxygen- or nitrogen-containing groups) after treatment leads to an increase in mechanical and electrical properties of nanocomposites [7]. Given the wide range of polymer materials (hydrophobic and hydrophilic) and subsequent filler-matrix interactions, a technique capable of tailoring SWCNT wettability is required.

In the case of biomedical applications, there are significant contradictions in the literature concerning the effect of SWCNT functionalization on their cytotoxicity [16]. For example, some indicate that functionalization of SWCNTs using carboxylic acid groups reduces cell viability and proliferation [17–19], whereas Montes-Fonseca et al. (2012) reported decreased cytotoxicity for functionalized CNTs [20]. A deep examination of the literature reveals that the contradiction comes from different CNT properties (i.e. wettability, functional group charging, variation of SWCNT size, degree of purity and effect of surface energy, etc.) [21]. Furthermore, the contradictions regarding cell response to functionalized SWCNTs in the literature can likely be attributed to the fact that individual reports do not study surface treatments over wide ranges (i.e. no attention typically paid to the extent of functionalization) – essentially, a single treatment is applied and the cytotoxicity is analyzed [22,23]. Beyond the issue of cytotoxicity, applications such as the selective binding of specific blood proteins (that can have polar and non-polar components) to CNTs can necessitate hydrophobic or hydrophilic surfaces [24,25]. Therefore, before any practical biomaterial applications of CNTs can be considered, it is necessary to address the issue of surface wettability. Specifically, there is a need to study a surface modification technique capable of controlling the extent of the functionalization. Such a method should allow a "full range" of properties (from superhydrophilic to superhydrophobic, for example) using a similar reaction scheme and functional groups.

Functionalization techniques can be classified into two groups: solvent-based (wet chemistry) and solvent-free (gas-phase) methods. Solvent-based approaches suffer from multi-step and complex preparation methodologies and can damage the CNT structure. Furthermore, separation of the treated nanomaterials from the solvent, as well as the management of solvent waste, can be a time-consuming and significant processing issue, which is generally dodged in the literature [26–30]. Gas-phase surface modification techniques can be classified according to the energy source applied for reaction initiation, including thermally activated chemical vapour deposition (TACVD) [31], plasma-enhanced CVD (PECVD) [32] and photo-initiated CVD (PICVD) [27]. Table 1 presents a detailed comparison between the current functionalization methods for CNTs.

Unlike TACVD (which uses heat, problematic for temperature-sensitive substrates) and PECVD (which requires specialized and costly plasma conditions), PICVD uses light to initiate organic deposition reactions. In this process, radicals can be generated by exposure to ultraviolet (UV) light, launching a series of heterogeneous reactions on the surface of a substrate in parallel with gas phase reactions. PICVD has been gaining interest for the surface treatment of nanomaterials because of its simple procedure, low cost, possibility of operation at or near atmospheric pressure (depending on the wavelength selected) [27,32], higher intermolecular cross-linking [33], low energy consumption [27,32] and, most importantly, ability to adjust the desired degree of functionalization [33].

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