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X-ray diffraction analysis for main peaks and the noises of single and multi-walled carbon nanotubes

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Abstract

The XRD for carbon nanotubes shows the main two peaks at 2Ө ≈ 25∘ and 43∘ with many noises for all the line from 5∘ to 80∘ which depend on nature of structure. The noises produce from instrumental sources and the physical properties of tubular structure and that represent by inter and intra Van Der Waals forces. Most of the noises were removed by using computerized programs while it is referred to nature of tubular structure with one or many sheets of graphene. The most of this noises represent the nature of structure which reduces with increase graphene sheets, thus removing it delete the specific proprieties of CNTs. The graphite behave the same properties of tubular structure which variant with number of sheets.

Keywords: SWCNTs; MWCNTs; XRD; Noises; Inter-VDW

1. Introduction

A new generation for nanoscience and nanotechnology after 1991[1] is carbon nanotubes CNTs which encourage to use rare physiochemical properties in many fields. CNTs commonly classified depending on the number of graphene sheets to single-walled SWCNTs, double walled DWCNTs, few walled FWCNTs and multi-walled carbon nanotubes MWCNTs. The CNTs behavior's [2] influence by number and length with nature of rolling the sheets when decided the center diameters of tubes and the distance between the sheets. in our previous work [3] we classified CNTs to graphite Nano tubes GNTs which refer to MWCNTs and graphene nanotubes gNTs and that refers to the ether types SW, DW and FWCNT. This confirms, close partnership between graphite, graphene and CNTs in many behavior and gives reasons for variance in the essential physio-chemical properties. Many apparatus were used to analysis CNTs which may images spectroscopy such scanning electron spectroscopy SEM, transmission electron spectroscopy TEM and helium ion microscopy HIM. The functional and structural groups were analysis by X-ray photoelectron spectroscopy XPS, Energy-dispersive X-ray spectroscopy EDX and X-ray diffraction XRD. The X-ray diffraction [4] commonly depend to analysis tubulars structure and assessment the quality and crystalline/ amorphous nature of nanotubes in addition to the purities. Yung et al. assessment the [5] interlayer spacing and crystallite size can change the diffraction angle and the

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line width when studies many carbon materials with a laser. In our previous works [6-11] XRD was used to characterized pristine and modified SWCNTs or MWCNTs with TiO_2 and Pt, also after activation with different chemical compounds. Osaimi et al. reported that increase the number of inner shells causing more intensity for MWCNTs than SWCNTs when studied [12] the effects of CNTs types on structure of CNT/poly-methyl methacrylate composite. The attentions concerned with identification the two main characteristic peaks of CNTs at 2Θ≈ 25° for (hkl = 002) and 43° for (hkl = 100) [3,13-14]. The first peak can be related to reflections from hexagonal carbon atomic networks and stacking the parallel of tubular structure, while the second peaks refers to graphitic crystalline structure of lattice [3,6,8,10]. Most literates mentions the two characteristic peaks without noise by draw the XRD patterns for CNTs without noise by computerized programs.

Dealing with the results of XRD then convert those into forms which can explain the nature or properties of the structure, it should be include all the details without neglected any part. Previous theoretical and experimental studies [13-14] refers directly or indirectly to two facts:

- The noise can be related to low count of results or, mostly increase X-ray flux by raises the slit size and increase the time of count , that produce greater signal/noise ratios. The disadvantage will be expense the resolution of some mains peak and more time required to complete the measurements. Time factor , represent the best ways to get better quality and quantities data by doing longer scan and reduced step size while, sometimes combinations the two sections may produces the ideal solution.

-XRD pattern entity include noise which appears large or small in comparison to main peaks data of material in this case the noise when plotting with peaks typically level of figure is very high. Thus the researchers solve this problem by two ways, the first: re-set the XRD apparatus by make zero background for sample which succeed to minimize the noise in the amorphous sample to produce clear image with eliminate necessary peaks which is the problem. The second remove or reduces the noises when applying acceptable software such Matlab, Excel, and X'pert High score. This review confirm, the noise refers to distinguish for types of CNTs thus remove the noise hides the true identity for the structure.

1.1. Theories of X-Ray Diffraction

The X-ray diffraction technique uses on powder or microcrystalline samples, where every possible crystalline orientation is represented typically.Calleja and Vonk reported that X-rays can be produced from X-ray generators or synchrotron facilities [13] for different materials. The metal used as an anode such copper which produces spectral line at 0.154 nm, while X-ray synchrotron radiation, besides a much higher radiation flux, a tuning of λ-values are possible. Garcia et all reported that the value of angles [2] that produce from scattering X-ray provide information about the structural such inter-atomic distances when reaching to few nanometers and evaluate the number of layers of materials. Bragg's law [$n\lambda = 2d_{hkl} \sin\theta$] [14] represent the characteristic equation for crystal structure which used commonly to estimate synthesized materials. Briefly the wavelength (λ) of X-rays scattering with diffraction angle (2θ) influenced by [15-16] the abilities to re-diffraction for many times (n) which refer to order of diffraction. All of these parameters can calculate the distance (d) between the successive identical in the crystal as shown in Fig. 1. Some materials appear a polycrystalline state when characterized by the different orientation of the crystallites.

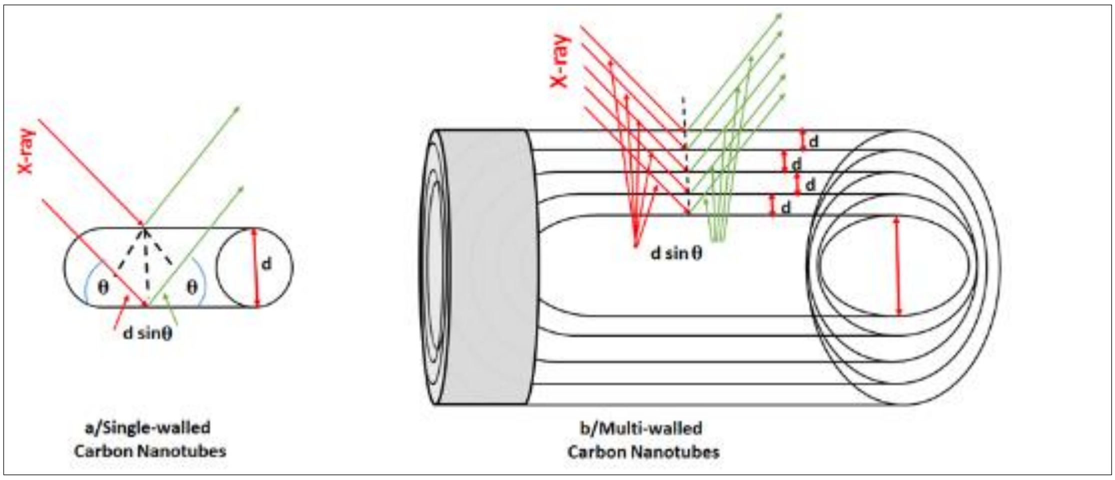

Figure 1 Schematic for applied Bragg's law with (a) SWCNTs and (b) MWCNTs

In Bragg view, crystal planes act as mirrors, could constructive the image when the path difference between the two reflected beams in the same plane of incident light. The image could be more strong and clear when the incident and diffracted beams were repeated more than twice with constructive interference, causing stronger peaks as shown in figure 1b. The unclear image accrue, when reduce the number of constructive interference and decrease number of material's layers mostly causing reduce the beam with increase the abilities to rise destructive beams as a noise signal. Theoretical studies of XRD patterns of the plane and tubular structure of graphite had shown an increase in the curvature of the graphite sheets leads to increase in the Bragg angle and decrease in the full width at half maximum FWHM [14-17]. Experimentally the patterns of Nano powder or in less-micron range need for specific treatments with use absolutely dry powder and longer scan time to avoid moisture to get ideal image. However, XRDs pattern influence by many parameters such as nature of material (particle size and proprieties) , preparation of sample for measurement, types of detector, power of tube and slit size [18].

2. Material and methods

2.1. Materials

Single-walled carbon nanotubes SWCNTs and multi-walled carbon nanotubes MWCNTs were purchased from Aldrich. The two types were fabricated by chemical vapor deposition method CVD. The purity of SWCNTs 77% SWCNTs, surface area S_{BET} was 1327 m^2/g and mode diameter 1.1nm, while MWCNTs was 83% in purity and 5.5 nm in diameter mode with 276 m^2/g in S_{BET}.

2.2. Characterization

XRD powder analysis were taken on a Rigaku Rotalflex (RU-200B), using Kα for Cu radiation (λ= 0.15405) nm and a Ni filter. The tube current was 100 mA with a tube voltage 40 kV. The 2θ angular data were estimated for 10° and 80° applying 5° min^{-1} for a scan rate with resolution equal to 0.02° and slow scan. The XRD pattern of CNTs were represent in figure 2 which shows two characteristic peaks at 2θ=25.6° , 44.3° and 2θ= 26.3°, 43.8° [3-6] related to the C (001) and C (002) planes for SWCNTs and MWCNTs respectively. The two peaks of SWCNTs are less intensity than MWCNTs peaks as shown in fig. 2.

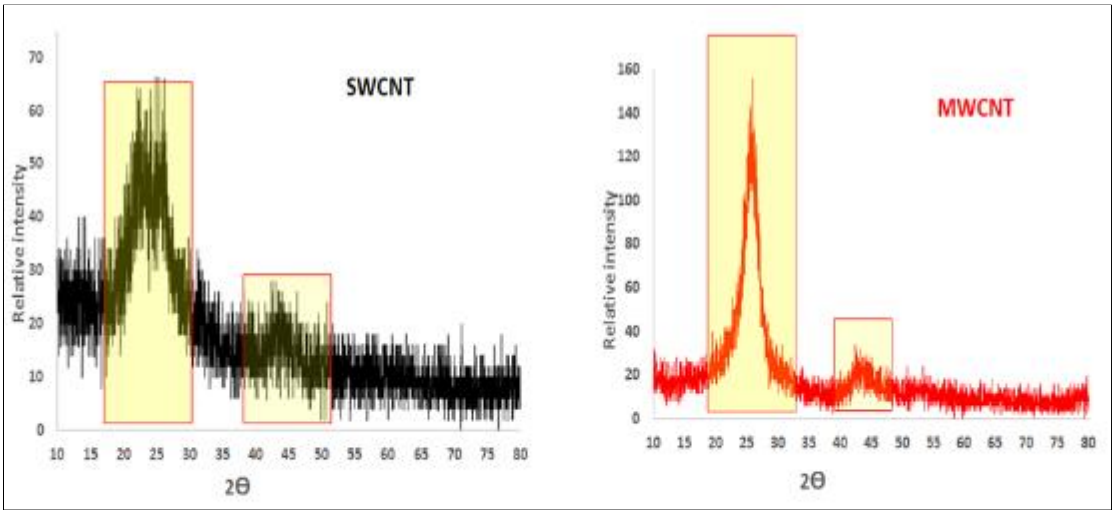

Figure 2 XRD patterns for SWCNTs and MWCNTs

The first peak at 2θ≈ 26 ° of SWCNTs and MWCNTs are listed in fig.3, appears variance in area under the curve with high ratios of noises for SWCNTs as compare with MWCNTs. The same behavior were seen in fig. 4 for the second peak at 2θ≈ 44∘ for the two types of CNTs.

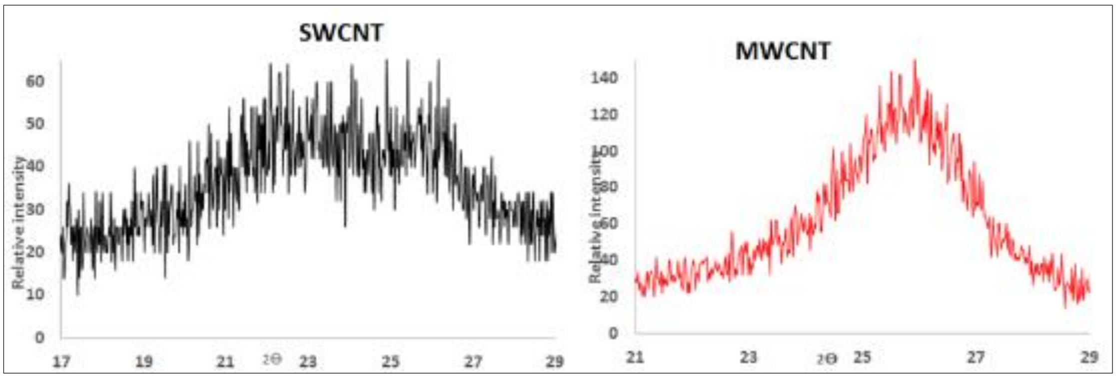

Figure 3 XRD pattern for the first peaks of SWCNTs and MWCNTs at 2θ ≈26°

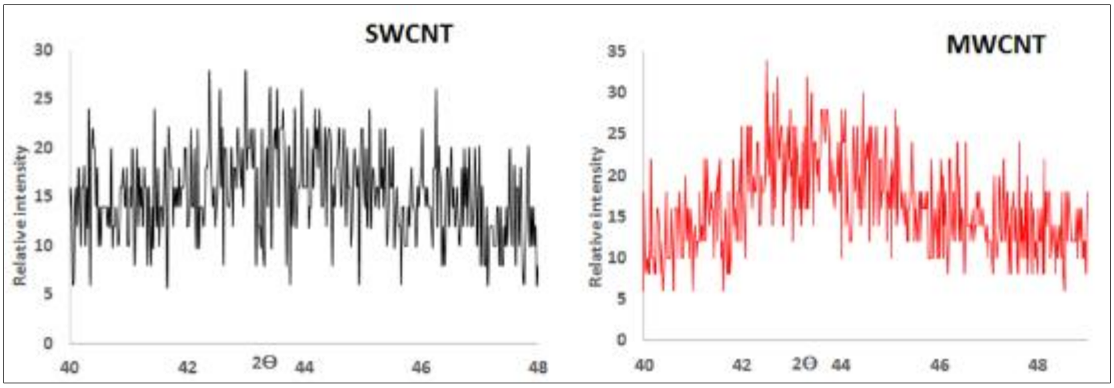

Figure 4 XRD pattern for the second peaks of SWCNTs and MWCNTs at 2θ ≈44°

3. Discussion

Prove the noise important in XRD patterns of CNTs which require compare the experiments results with many reported litterateurs in this field that used XRD for characterized CNTs. The mechanism of XRD depends on the interaction of X-ray with the CNT surface, when we have one sheet the constrictive motive will be lost or at least reduce. Some of the rays mostly hitting the upper side of tubes, which mostly causing noises without constrictive the signal. The last factor was more critical due to the availability of the periodic arrangement of atoms to enhance the X-ray diffraction in certain directions. Amorphous nature of particle prevent periodicity with a random distribution of atoms and ratios of constructive rays which influence with tubular structure causing noises signals. The two types of CNTs which used in this work were listed in figure 2,3, and 4 include the main peaks without removing any noise.

Silva and Rezende [19] used two type of MWCNTs purchase from Bayer Co. Ltd., Baytubes-C150P code (diameter 13 - 16 nm and length > 1 µm) and CNT-K, from Hanwha (diameter 10-15 nm and lenght10-20 µm) with purities more than 90% for two types. The MWCNTs were used to synthesized composites with epoxy resin, the two types were shown many noises._Yutaka et al. shows [20] that XRD of SWCNTs after and before heating at 350°C did not remove the noise in spectrum. The noncrystalline/crystalline structure of CNTs is obligatory shown distinct X-ray diffraction peaks and huge amount of noises [3,14].The same behavior was reported by Mhamane et al. ,Tang et al. Yeong et al.[20-22] for graphite and graphene when shows noise with graphene and graphene oxide while reduces with graphite but it is less then CNTs. The case of tubular structure particularly with SWCNTs appeared the noise with high density which also reported by Tursun et al. [23]. Launois et al. [24] converted C70 to DWCNTs which shown High temperature value of X-ray diffraction measurements refer to increase the noise for DWCNTs as compare with C70. Guan et al. [25] synthesized MWCNTs by catalytic CVDs by Fe-Co/MgO of methane at 800°C and purified with reflux 3M sulfuric acid for 6h/80°C. The XRD analysis for MWCNTs after and before purification shows increase in noise after purification with intensity more than 25 unite. Hon et al. [26] was synthesized MWCNTs by flame of propane on Ni-surface and analysis by TEM and XRD. The analysis with XRDs shows very sharp shape for the main peaks while the base line was Wide font which refer to noise. The SWCNTs and DWCNTs were synthesized by Mehran et al. [27] from methane at 900°C with catalytic/Fe CVD, the product were analysis by XRD which confirmed existence the noise.Futaba et al. [28] were used XRD/macroscopic method to estimate the number of graphene layer in CNTs. The results based on the (002) peak which decomposed to inter and intra tube to evaluate types of CNTs (SWCNT, DWCNT, FWCNT, and MWCNT) shows that increase the number of sheets reduce the noise of XRD patterns. Yupeng et al. and Dumlich with Reich [29-30] reported that CNTs mostly exists in bundles due to Van Der Waals VDW between tubes of sp2 carbon and that causing enter-VDWs forces in addition to intra-VDWs forces inner the tubes. Chowdhury et al. [31] reported the distance of inter-VDWs was 0.34 nm and the value of intra-VDWs was decided by number of sheets as shown in figure 5.

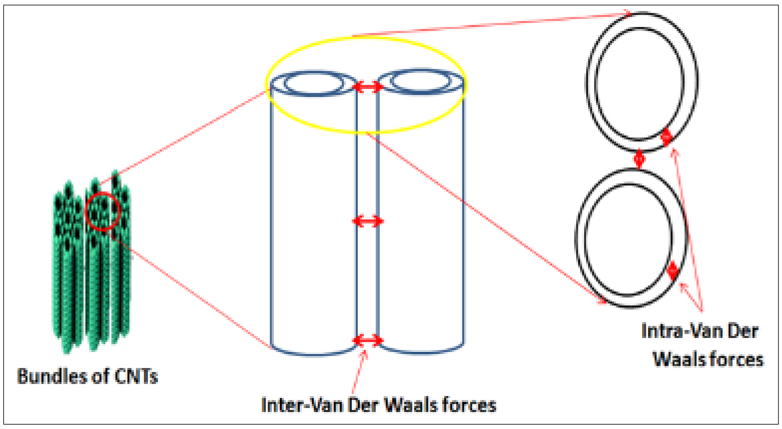

Figure 5 The skim diagram for inter and intra VDWs forces in CNTs

Physics view, for XRD patterns of nanomaterial crystallites, the diffraction of X-ray like mirror reflection, thus absolutely the Nano size of the mirror forming reflected light with low intensity. Figure 5 shows to levels of mirror the first specific for the inner diameters of tubes which is the miner peaks while the ether is the distance between the tubes and that decided by VDWs forces that could be related to movable level. The inner diameter (enter-VDWs forces) which is responsible to main peaks at 2Θ≈ 25° and the distance between the tubes (intra-VDWs forces) which is responsible to huge amount of secondary reflection peaks causing the noise [32]. The two cases inter and intra interaction with each ether causing appearance of huge amount of noise with main peaks and that agreement with many litterateurs [25-32]. Thus increase the number of graphene sheets mostly reduce the noises, while with tubular structure the previous interference causing the noise which is increase as the following arrangement for CNTs types and that agreements with Bragg's law:

SWCNT> DWCNT> FWCNT> MWCNT

Therefore, we emphasize the impotent to producing the structure as it is without any treatment which removes or eliminate important details of the compound.

4. Conclusion

The powder sample was in micron range size then usually 'peak heights' will be large and peaks will be thinner than those observed in XRD scan of Nano sized powder. The advantage for micro size mostly represents with very low ratios of noise as compared with nanomaterials. Remove all the noises in X`pert software after applying the requirements and that causing remove the weak intensity of peaks. The nanomaterials when analysis by X-ray shows the real structure which mostly depend on the number of sheets or layers that form it. Typically the computerized programs very important to plot the results, when keeping the identity of the materials. However the signal that produces from X-ray analysis influence with qualities and quantities of materials, nature, and orientations for the atoms in the compound, in addition to existing the materials in a pure state or with mixtures like composites. Actually, one should not use 'programs' to 'reduce noise'. Noise will be 'constant background' for given set of parameters or settings in X-Ray diffraction experiment.

Compliance with ethical standards

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