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Synthesis and characterization of carbon nanostructures from green oil using thermal pyrolysis process

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Abstract

The cost of the production of carbon nanotubes (CNTs) is one of the great challenges. Thermal spray pyrolysis is a simple and economic technique for synthesizing CNT's at low temperature. Fabrication and characterization of CNTs based thermal pyrolysis process are reported in this work. The precursors, the catalyst, and the carrier gas are all factors affect the cost of production. Coconut and olive oils are used as precursor, nickel chloride as a catalyst, argon as a carrier gas to produce CNTs on different substrate such as Al alloy, anodized Al and Si wafer operated at different temperatures ranged from 500 up to 700 °C. The concentration of nickel chloride was 5 wt%. The results reveal that the coconut oil producing graphene sheet while olive oils, has been found to be an effective precursor of CNTs than coconut oil. CNTs were prepared by catalytic decomposition of the oil over the metal particles dispersed and supported on the surface by spray pyrolysis method with a reaction time 5 min. The CNTs were characterized by FESEM (A field emission scanning electron microscope), TEM (transmission electron microscopy), XRD(X-ray diffraction) and Raman spectroscopy. Raman spectroscopy reveals that as-grown nanotubes are well graphitized with olive oil than coconut oil.

Keywords:

Thermal pyrolysis technique, Carbon nanotubes (CNTs), Olive oil, Coconut oil, Graphene,

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1. Introduction

Carbon nanotubes (CNTs) are the most conductive, strongest, and lightest materials. CNTs have been discovered in 1993 [1]. CNTs have been extensively studied due to their outstanding physical, chemical, and material properties such as good electrochemical stability, high electrical and thermal conductivities, and high surface area to volume ratio [2]. These unique properties make CNTs to use in the fabrication of the wide range of potential applications like, field emission [3]. Scanning probes sensors [4], nanoelectronics [5], solar cell [6] etc. In spite of its charming aspect, nevertheless synthesis of CNTs is still difficult. Expensive equipment with different kind of gases and carbon sources are being used for the synthesis of single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs).

The cost of production of CNTs is one of the great challenges faced by researchers in the preparation of CNTs. The precursors, the catalyst, the carrier gas and the techniques affect the cost of production. The traditional method of preparation of CNTs is the arc evaporation of graphite [7]. Several other methods have also been used for the synthesis of CNTs; these include spray pyrolysis [8] chemical vapor deposition (CVD) [9], water assisted chemical vapor deposition (CVD) [10], oxygen-assisted CVD [11] point-arc microwave plasma CVD (chemical vapor deposition) [12], molecular-beam synthesis [13], hot-filament CVD [14], and alcohol catalytic CVD (ACCVD) [15].

Fossil fuel precursors are becoming more expensive, as the supply is predicted to run low over the next few decades. There is a trend to use green precursors to avoid pollution. The high carbon content in vegetable oils qualifies them to be a great source of CNTs [16]. Vegetable oils such as turpentine, eucalyptus, coconut, palm oil and waste cooking palm oil as starting materials in CNTs production have been widely reported [16]. Vegetables oil affect in the cost of CNTs as they are easily to be available and they are presented with high percentage in environment. also vegetable oils are less pollution, vegetables oils also are rich in carbon, this means that a more production of carbon nanotubes area than other traditional source such as methanol, ethanol or acetone. Among the new CNT precursors, olive oil is highlighted because it is a natural source which is renewable, environment-friendly and has the potential to be the green alternative for industrial-scale production of CNT [17]. Ferrocene is used as a catalyst in most of production of CNTs, but some researchers looked for less expensive and commercial catalyst. Some researches are done in catalyst effect such as ferric chloride, nickel chloride which gives good types of CNTs [18]. Thermal spray pyrolysis is a simple and economic technique for fabricating CNT's at low temperature. Pyrolysis of the carbon precursor and deposition occur in one step. Recently the spray pyrolysis method has attracted attention due to the possibility of producing carbon nanotubes on a commercial scale [19].

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This present work deals with the effect of vegetable oils (olive and coconut oils) as environmentally, friendly precursors on the growth of CNT film at different operating temperatures (500-700 °C) on different substrates such as Al, Al anodized and Si wafer using thermal pyrolysis technique. The structure and the characteristics of CNTs have been investigated using field emission- scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM), and Raman spectroscopy.

2. Experimental

2.1. The production of CNTs

The production of CNTs is setup in thermal spray pyrolysis oven described by Kamalakaran et al [20]. A schematic diagram of the equipment used is shown in Fig. 1. Quartz tube of 1 meter length and a 27 mm inner diameter was held inside a tube furnace.

A- Materials

Food olive oil 99.99 % pure (Extra Virgin, from Sinai Egypt) and coconut oil 99.99 % pure (Sigma Aldrich) were used as the precursor and nickel chloride with purity 99% (Sigma Aldrich) as a catalyst.

B- Preparation of precursors

A mixed solution of 5% of nickel chloride in olive oil or coconut oil was prepared. Argon gas was used as carrier gas to generate the nickel chloride/precursors mist in the nebulizer.

C- Synthesis of carbon nanotubes

For all experiments, the precursor solution was stored in a syringe and dispensed in a continuous flow at a fixed rate through 5 ml in 5 min. Upon entering the growth region, the gas and reactant mixture were rapidly heated from room temperature (25 °C) up to the operating temperature. When the oven reached the operating temperature, argon fed to quartz tube and the solution was sprayed without any previous heating. During the spraying process, both the coconut and olive oil and the metal compound are decomposed to produce free metal atoms and smaller carbon species, from which CNTs nucleate and grow. Argon gas is applied from the starting temperature till the furnace is cooled. Thereafter, black film made of CNTs formed on the substrates and inner surface of the quartz tube. The product is removed mechanically from the tube with a brush or by immersion in acetone.

2.2. Characterization

A field emission scanning electron microscope (FE-SEM) equipped with energy dispersive X-ray system (EDX) model JEOL, JSM-5410 was used to study the surface morphology of the surface cover with CNTs and the elemental analysis of the deposited layer, respectively.

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Additional study of the obtained CNTs was made by transmission electronic microscopy to corroborate the formation of nanotubes. High resolution transmission electron microscopy (HR-TEM) images were obtained using an FEI (Tecnai G2 20S-Twin, Netherlands) microscope with an accelerating voltage of 200 kV. The sample powders were dispersed in ethanol, under sonication and TEM grids were prepared using a few of drops the dispersion followed by drying in air.

X-ray diffraction (XRD) patterns were obtained over the diffraction angle range (2θ) of 10-80° using an XRD (X'Pert PRO — PANalytical, Netherlands) diffractometer with Cu K α ($\lambda = 1.5404 \text{ \AA}$) radiation at a generator voltage of 45 kV and a generator current of 30 mA, with a step size of 0.02° and a scan speed of 0.05 s⁻¹.

X-ray photoelectron spectroscopy (XPS) thermo scientific K-Alpha was used to evaluate the purity of the prepared samples. The sample was irradiated with Al K α monochromatic X-ray source, and the analyzer passes energy of 200 eV with a step size of 1 eV for high resolution spectra to obtain the chemical state information.

The morphological features of the as-prepared coating film were analyzed by Raman spectroscopy. The Raman spectrum was obtained in (Model Sentera, Bruker, Germany) equipment, at laser wave length 532 nm [doubled Nd:YAG laser (neodymium-doped yttrium aluminum garnet)] and power 10 mW. The equipment is fitted with an Olympus metallurgical microscope and the sample was investigated on a microscope slide with an X 80 lens. The spectrum was obtained at room temperature in the spectral range of 1200–100cm⁻¹.

3. Results and discussion

The impact of reaction parameters like reaction temperature, reaction precursors, and substrate plays a major role in deciding the types of CNTs formation, and its yield. In this work, we used different substrates such as Al, anodized Al and Si wafer, a reaction temperature ranging between 500 to 700 °C, and coconut or olive oils used as environmentally carbon sources. Chemical Treatments for the used substrate was applied before enter the pyrolysis furnace.

3.1. Deposition of CNT from coconut oil on different substrate

Coconut oil is used as a green precursor (source of carbon) to deposit CNT's on Al, anodized Al and Si wafer surfaces. SEM and TEM of the coated CNTs layer formed on these different substrates at different temperatures are illustrated in Figs. 2 and 3. Figure 2 shows the top-view FE-SEM images of a typical sample after thermal pyrolysis process. As can be seen from Fig. 2 the reaction on the different substrates at 500 °C is very low, while at 600 °C long tube-like structures were seen, in addition to these long tube-like structures, it is observed that the shape of the nanotubes changed to irregular shapes. Moreover, at 700

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°C bubble-like or honey structure of tubes was formed on Al substrate and a bundle of tubes was formed on anodized Al and Si wafer surfaces.

Figure 3 illustrates the TEM images of CNTs synthesized on Al, anodized Al and Si wafer substrates by thermal pyrolysis process at 600 and 700 °C using coconut oil as the carbon source. TEM images of the CNTs formed on Al substrate indicate the presence of the shape like graphene structure, while the images of the product on anodized Al reveal the formation of bundles of tubes having the same diameter. The TEM images of the layer produced on Si wafer substrate operated at 600 does not illustrate the formation of tubes. At 700 °C the figure reveals the formation of net shape.

From SEM and TEM images it may be concluded that the operating temperature up to 700 °C is not enough for the formation of sound CNTs on Al and Si wafer substrates with coconut oil.

Raman spectroscopy is commonly used for characterizing CNTs because it is one of the most sensitive tools characterization of these nanostructures [21,22]. The characterization of the coated layer was carried out using Raman scattering spectra for the product on Si wafer surfaces operated at different temperature (500 -700 °C) using coconut oil as carbon source, as shown in Fig. 4. The spectrum of the coated layer on Si wafer shows two prominent peaks from 1331 - 1347 cm^{-1} (D-band) and from 1586 - 1594 cm^{-1} (G-band), respectively. It is known that the G-band can be indexed to the tangential stretching (E_{2g}) mode of the highly oriented pyrolytic graphite and suggests the CNTs to be composed of crystalline graphitic carbon. Higher intensity of G band indicates the higher degree of crystallinity/graphitization. On the other hand, the D – band originates from disorder in the sp^2 - hybridized carbon and indicates lattice distortions in the curved graphene sheets, tube ends, etc. D-band at approximately 1350 cm^{-1} is corresponds to a more disordered structure [23, 24]. In our study the ratio of the I_D/I_G for the product formed on Si wafer at different temperatures amounts to about 1. This can be attributed to the presence of contamination from catalyst and carbon precursors which needs to further purification of the product. G-band of graphene flakes in Raman spectrum of our prepared graphene layer shows a peak located at the range 1586 - 1594 cm^{-1} which confirm that this product is composed of a few layered graphene. Finally the D band at 1331 - 1347 cm^{-1} may be attributed to defects in the graphene nano sheets or their agglomeration.

Based on the result of Raman and TEM of our work, the results reveal that the product is the graphene rather than CNTs.

3.2. Deposition of CNT from olive oil on different substrate

Figure 5 illustrates the morphology of CNTs deposited on the anodized Al and Si wafer substrates at fixed temperature 700 °C. Linear and helical structures were observed at the

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surface. A more uniform distribution of CNTs was observed on anodized Al surface. The diameters of the CNTs of around 40– 50nm were obtained. This result is agreement with finding of Paul and Samdarshi [23]. They reported that the diameters of a random-orientation CNTs of around 80– 90nm were obtained when coconut oil was used as the carbon precursor. In the present work, the surface morphology study showed that the smallest diameter obtained was 40 nm when olive oil was used as the carbon precursor.

The TEM images of a deposited CNT on Al alloy, anodized Al or Si wafer surfaces are shown in Fig. 6. It can be seen that it comprises a hollow core with uniform structure along the carbon nanotube axis. This tube is found to have a diameter of 20-30 nm and it is nearly 1-2 μm long.

The first order Raman spectra of as the grown product CNTs on anodized Al and Si wafer surfaces operated at 700 °C using olive oil as carbon source, are shown in Fig. 7. All spectra illustrate mainly two Raman bands at 1341.8 cm^{-1} (D-band) and 1602 cm^{-1} (G-band), respectively for the product on Si wafer, and at 1343.4 cm^{-1} (D-band) and 1601.5 cm^{-1} (G-band), respectively for the product on Al anodized. In our study, the ratio of the I_D/I_G (~ 0.54) for the product formed on Si wafer and (~ 0.61) for the product formed on anodized Al indicates that the degree of graphitization of the CNT is low. This can be attributed to the presence of contamination from catalyst and carbon precursors which need to further purification is done for CNT. So in order to predict the contamination in the sample, XPS analysis is done. The result that reported from CNT produced by thermal spray pyrolysis of natural precursors I_D/I_G is around 0.3-0.68 [25, 26], and that formed from olive oil is about ~ 0.74 [27].

XPS is one of the surface analytical techniques that inform about the nature of the functional groups and also on the presence of structural defects on the surface. The atomic ratio and binding energy of each element on the coating surfaces are listed in Table 1. They declare the purity of CNTs formed on anodized Al and Si wafer and they are approximately 94.62 and 92.49 %, respectively. The survey spectra in the -200 -1600 eV of binding energy range are investigated. Figures 8 and 9 show the region of XPS carbon spectra a-long with curve-fitting by the Lorentzian method for both Si wafer and anodized Al substrates. The whole spectrum is also shown inset, all of the survey spectra of CNTs formed on anodized Al and Si wafer and used olive oil as a source of carbon (precursors), and operated at 700 °C for 5 min. clear carbon and oxygen peaks at around 285.0 eV and 533.0 eV are appeared, respectively. The ex-tended region shows a broad area in the higher binding energy region. C1s peak is fitted as four peaks shows that there are four different chemical environments of carbon presents in the sample. C=C (sp²) is for 284.22 while C-C (sp³) is for 285.01 Ev and 288.6 eV is for C – OOH bonds. While O1s curve is fitted where the two peaks appeared at 532.8 eV due to the surface oxygen complexes of carbon phase [28].

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Table 1. The atomic ratio and binding energy of each element of WCNTs formed on the Al anodized and Si wafer surfaces

Name	BE eV	Atomic %	
		Al anodized surface	Si wafer
C1s	285.07	94.62	92.49
O1s	533.09	5.38	6.71
Si2p	104.05	-	0.8

XRD is the most popular X-ray scattering route for characterizing the crystallographic structure of the samples. The X-ray diffraction pattern of the CNTs sample according to the standard pattern JCPDS database [ICDD/JCPDS Card Search. In: Jade 7 v7.06: Material Data, Inc.;2004] u. Sharp peak at 25.92 can be seen clearly, corresponding to the (0 0 2) reflection of carbon. The reflection around 43.46 corresponds to the (1 0 0) plane and indicates the presence honeycomb structure formed by sp² hybridized carbons.

The mechanisms of growing carbon nanotubes have been debatable and therefore, a growth mechanism has not been well established. Our suggested mechanism can be outlined as follows: During the spray process, the oil/catalyst (NiCl₂) drops enter to the furnace. The molecules of precursors/catalyst are thermally split. Consequently several reactions occur such as dehydrogenation, condensation of the oil, and Ni atoms agglomeration. The CNTs formation is produced when Ni⁺² is reduced to metallic Ni, which catalyze the hydrogen deprivation of precursor (oil). Thus dehydrogenated precursors begin to bond to other dehydrogenated precursors to form the graphite (carbon) wall of CNTs. Finally, at appropriate conditions, the precursor of CNTs are formed in the gas/vapor phase and consists of Ni particles surrounded by graphite layers as shown in schematic diagram of the mechanism (Fig.12), then these precursors can reach the surface (Si wafer, glass or hard anodized) and start the growing of CNTs. This mechanism is similar to the Geohagan mechanism [29, 30]

4. Conclusion

- This work present, the successes in the production of carbon nanotubes and achieve higher yield by a simple and inexpensive method.
- The synthesis temperature, carbon precursor, and substrates have a great influence on the production of CNTs. Temperature is one of the most important parameters for growth of CNTs. It is shown that, with 5 wt% metal concentration, CNTs are formed selectively at 700 °C, on Si wafer and Al anodized substrates, while graphite shit formed at temperature less than 700 °C. So, with vegetarian hydrocarbon as a carbon precursor, we have been avoided using organic toxic precursors like benzene, toluene and harmful gas as carbon monoxide.

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- Moreover, we demonstrated that coconut oil producing graphene on a commercial scale to offer low costs and high productivity. Also, this process will open the way to many well-established chemical techniques that could be used to produce graphene sheets into various structures.
- Graphene can be produced by thermal spray pyrolysis using vegetable oil, this can be a good well step in production graphene by a simple method.

5. Abbreviation

Name	Abbreviation
Carbon nanotubes	CNTs
XRD	X-ray diffraction
TEM	Transmission electron microscope
FESEM	field emission scanning electron microscope
CVD	Chemical Vapor Deposition
ACCVD	Alcohol catalytic chemical vapor deposition

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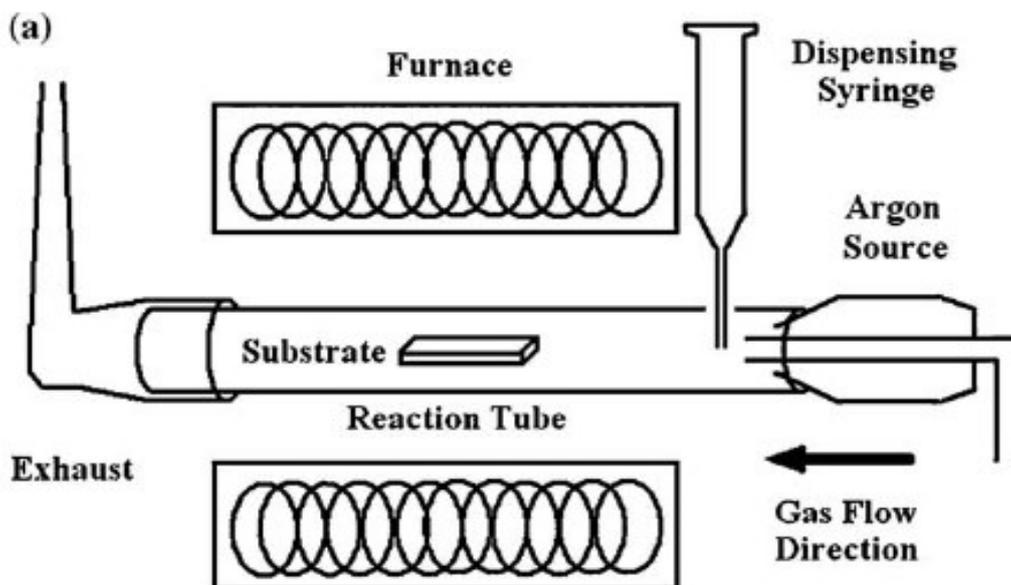
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Fig(1) : A schematic diagram of the CNT tube furnace.

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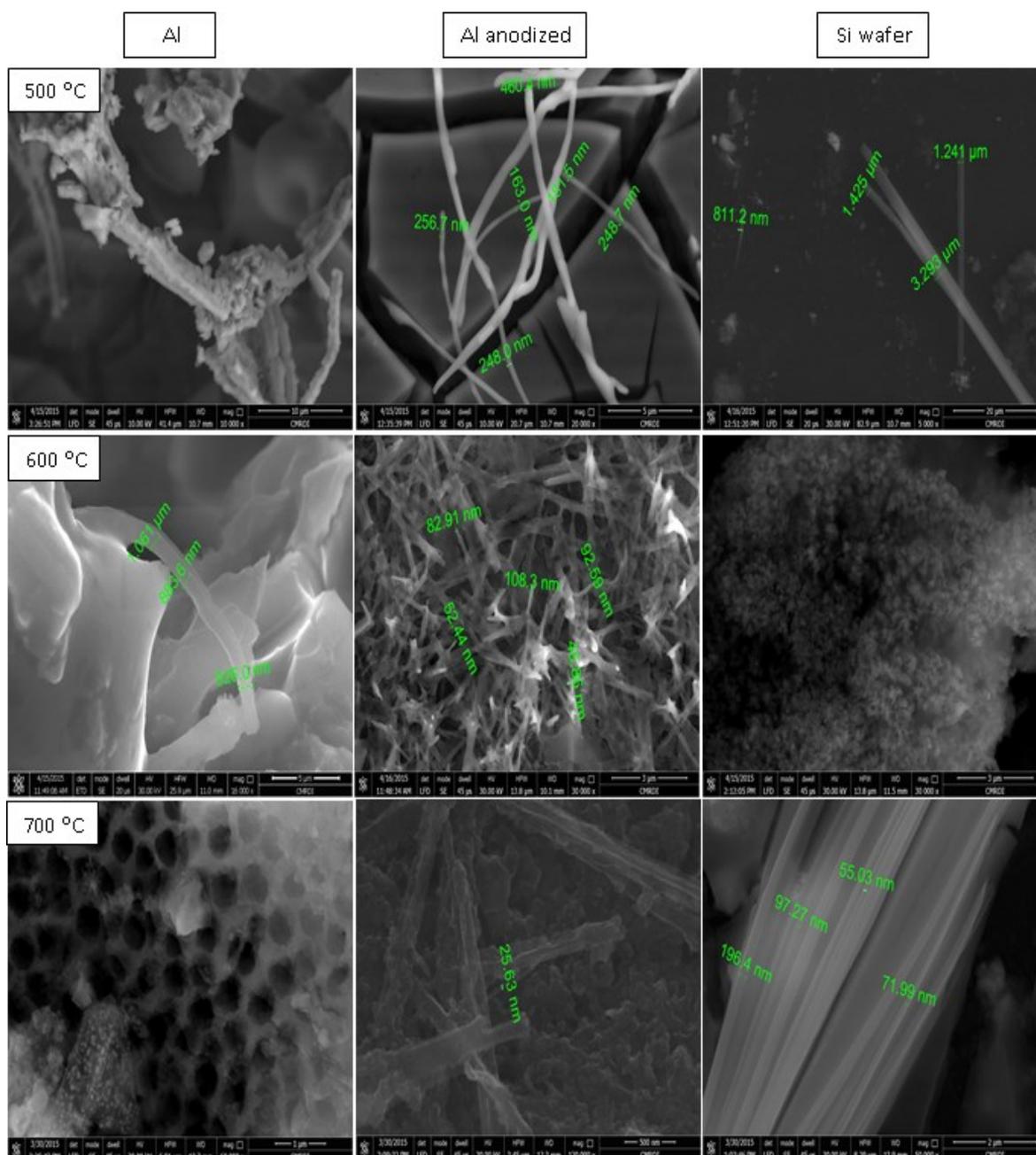


Fig. 2. FE-SEM of CNTs synthesized on Al, Al anodized and Si wafer substrates by thermal pyrolysis process using coconut oil as the carbon source at 500, 600, and 700 °C.

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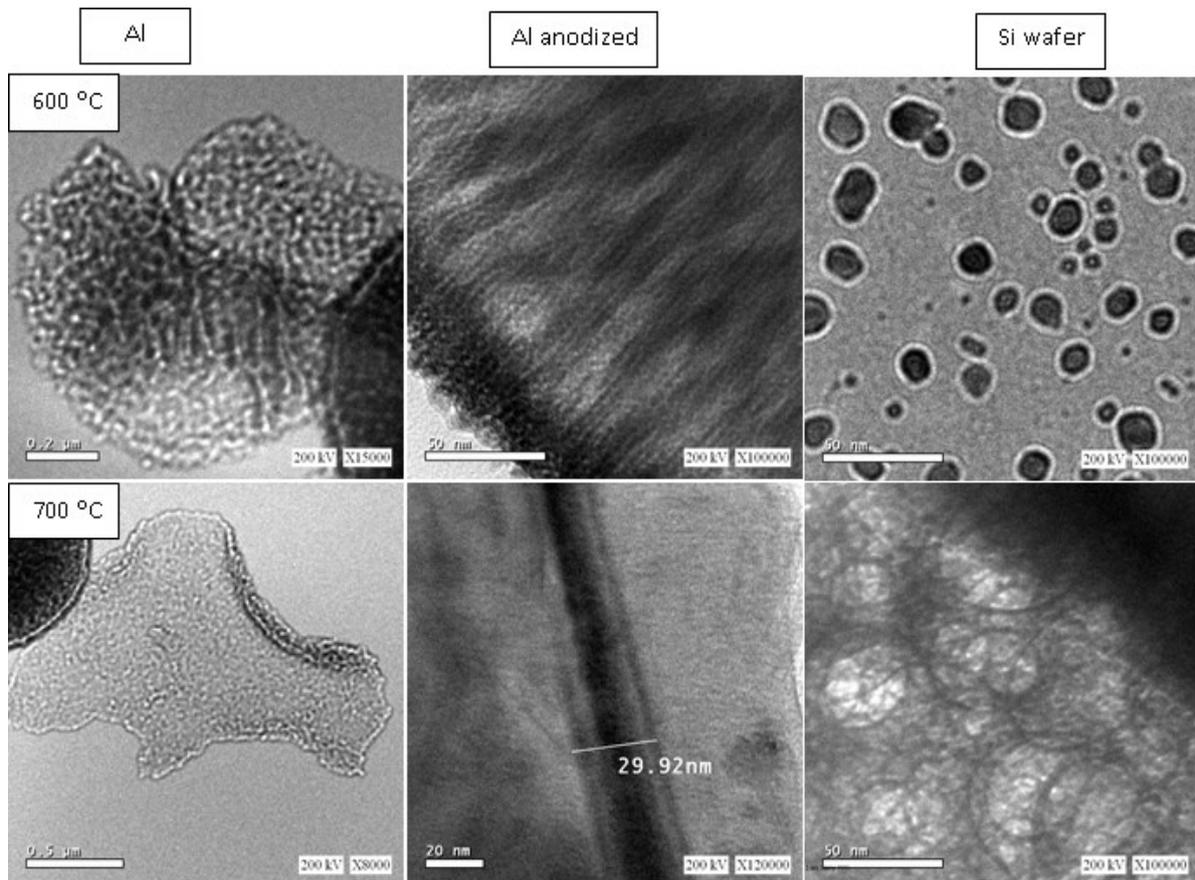


Fig. 3. TEM images of film synthesized on Al alloy, Al anodized and Si wafer substrates by thermal pyrolysis process using coconut oil as the carbon source at 600 and 700 °C.

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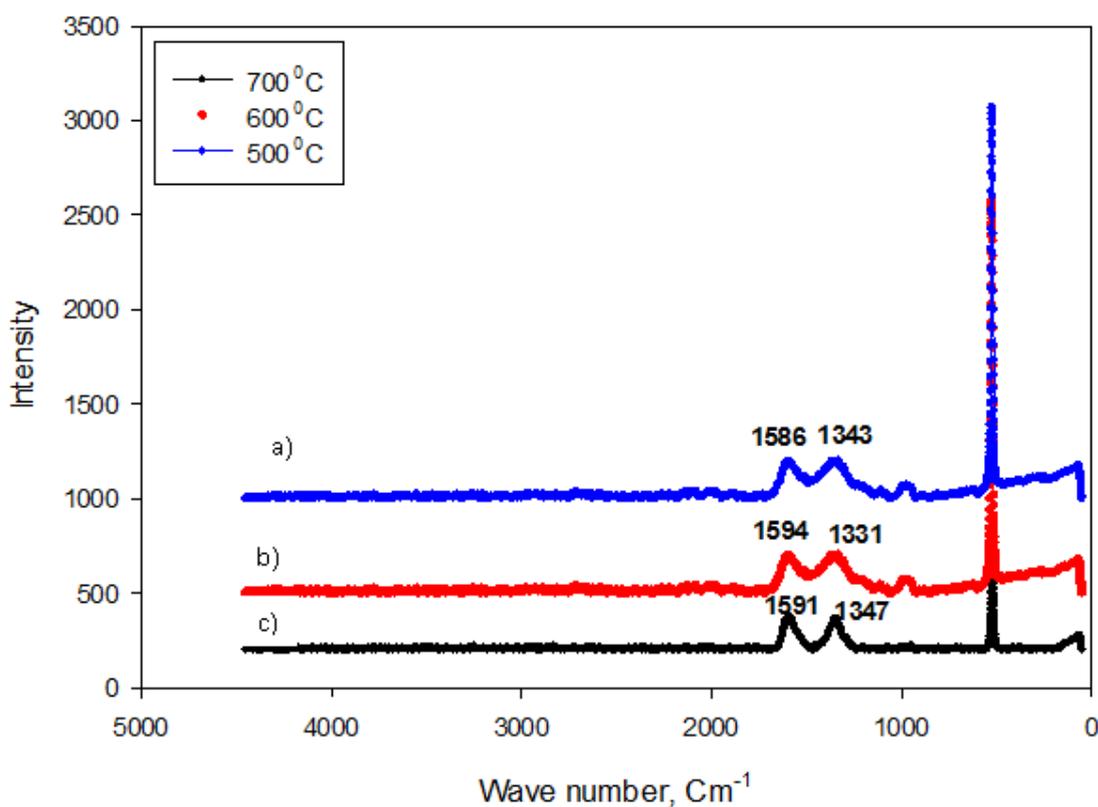


Fig. 4. Raman spectroscopy of the deposited layer using coconut oil as a precursor on Si wafer at different temperatures where, a) at 500 °C , b) at 600 °C and c) 700 °C using Ar gas and Nickel chloride as catalyst.

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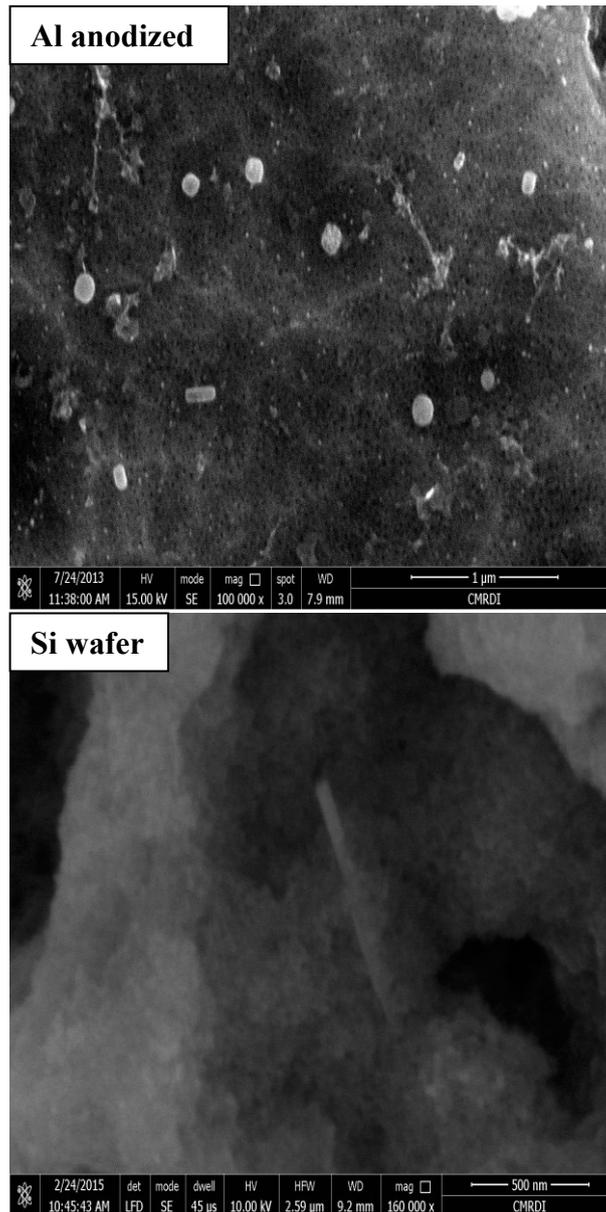


Fig. 5. FE-SEM of CNTs synthesized on Al anodized and Si wafer substrates by thermal pyrolysis process using olive oil as the carbon source at 700 °C.

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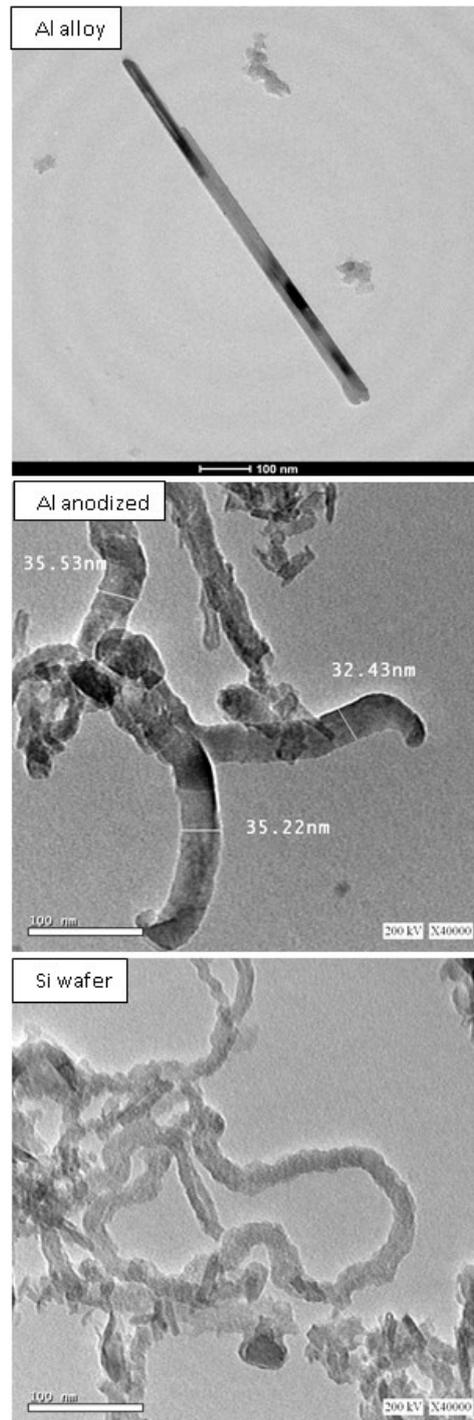


Fig. 6. TEM imgs of CNTs synthesized on Al alloy, Al anodized and Si wafer substrates by thermal pyrolysis process using olive oil as the carbon source at 700 °C.

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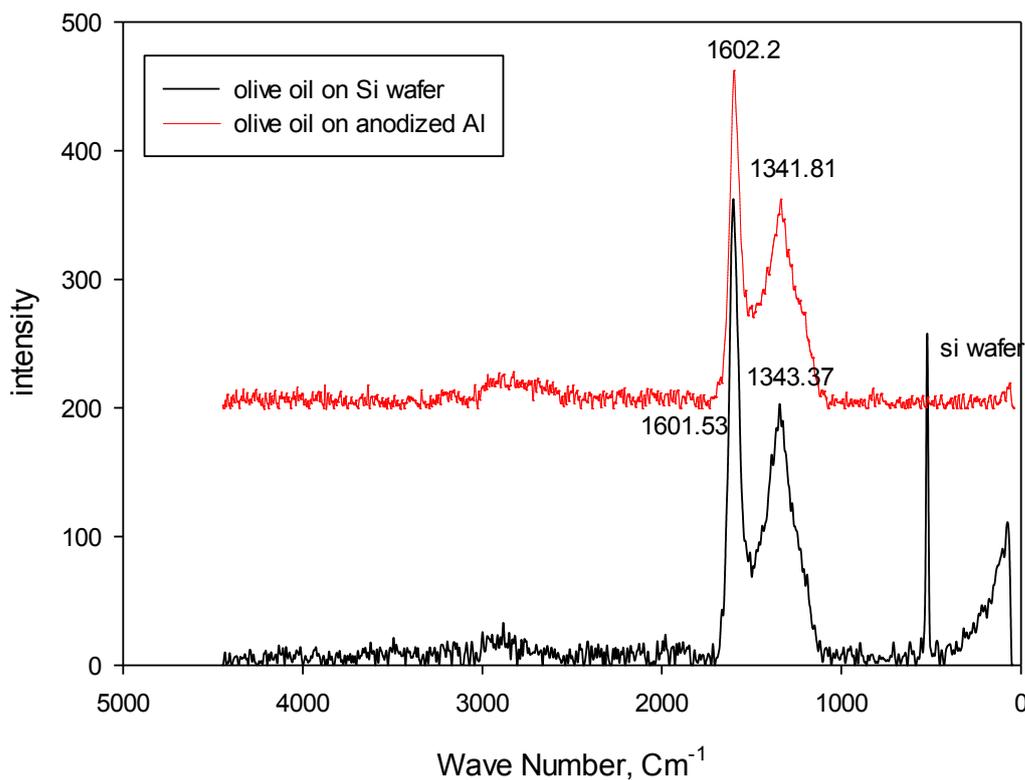


Fig. 7. Raman spectroscopy of olive oil as a precursor on a) Si wafer, b) Hard anodized Aluminum at 700°C using Ar gas and Nickel chloride as catalyst

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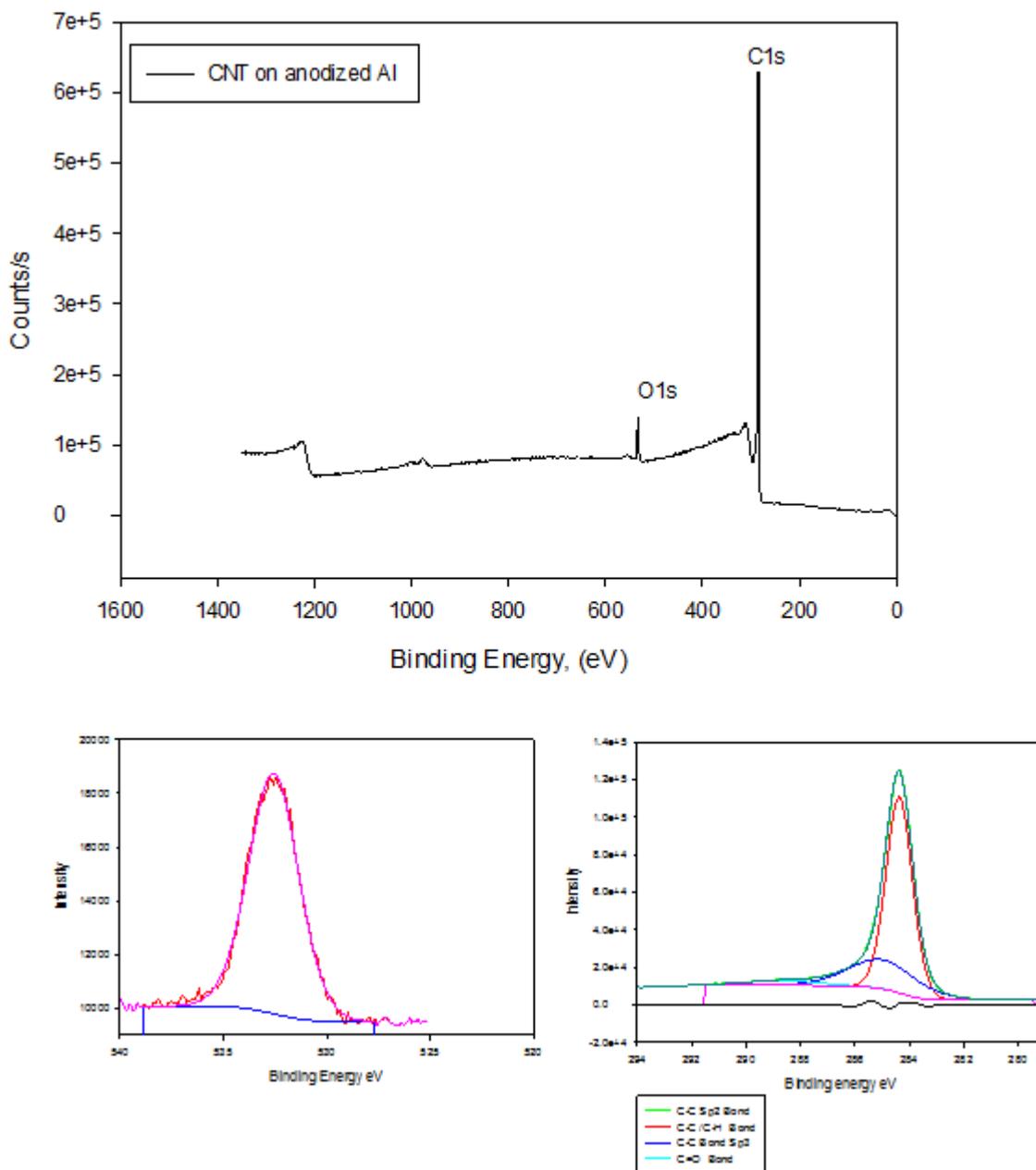


Fig. 8. XPS of CNT on anodized Al using olive oil as precursor in presence of nickel chloride at 700 °C and argon gas.

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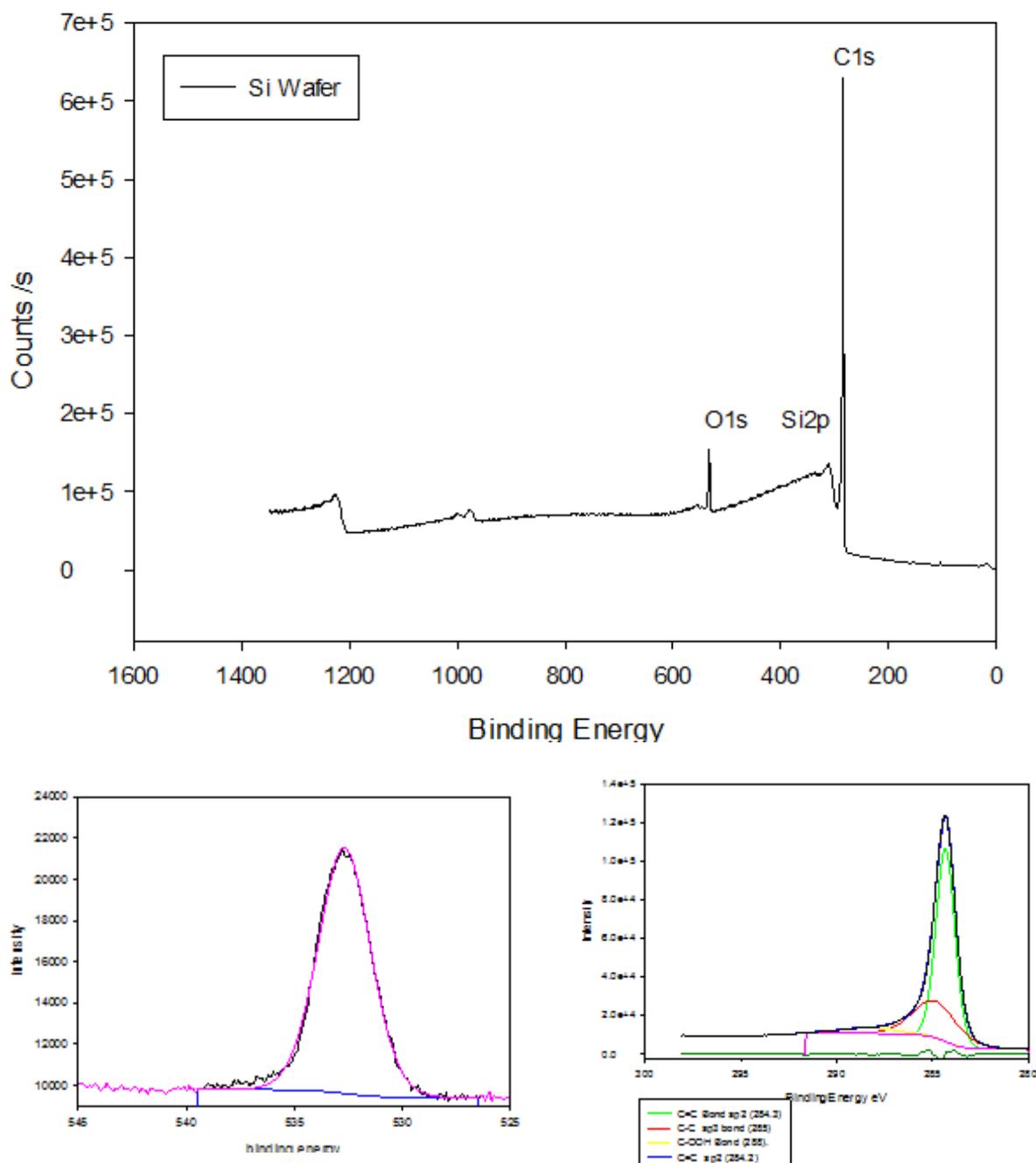


Fig. 9. XPS of CNT on Si wafer using olive oil as precursor in presence of nickel chloride at 700 °C and argon gas.

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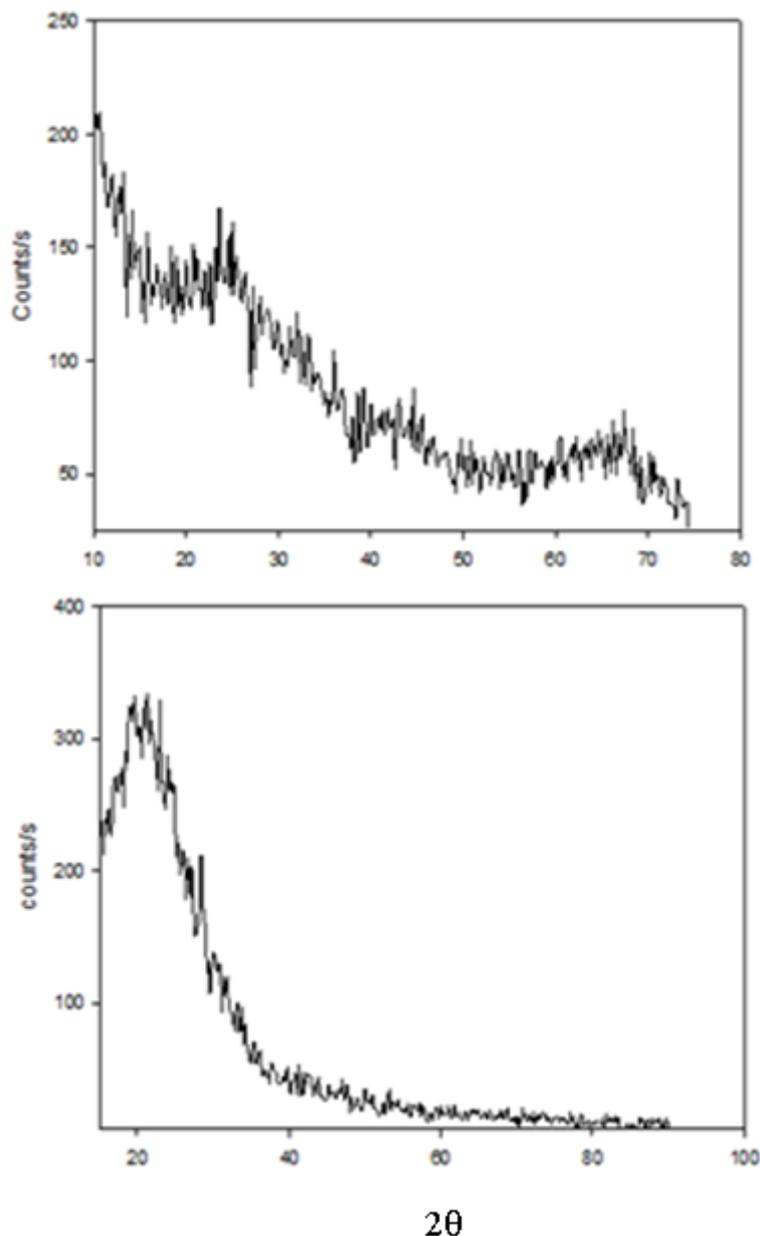


Fig. 10. XRD pattern of CNT Synthesized by pyrolysis at 700°C, from olive oil precursor.

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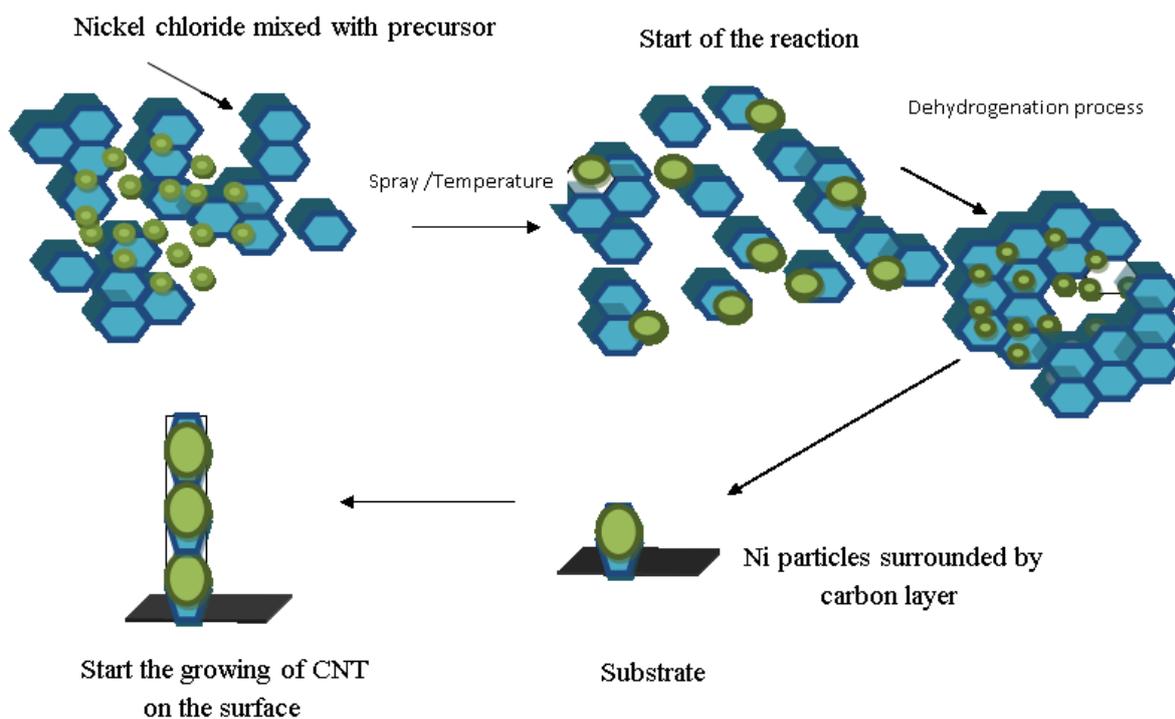


Fig.12. Mechanism of CNTs growing from precursors/catalyst drops.



Precursor

Nickel chloride