

Green Fabrication of Multiwalled Carbon Nanotubes(MWCNTs) from Solanum tuberosum (Potato tuber starch) and AI-ML Enabled Ammonia Sensing analysis Performance

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Abstract

A low-cost and facile route for the synthesis of multiwalled carbon nanotubes (MWNTs) is demonstrated via direct high-temperature incineration of *Solanum tuberosum* (potato) tuber biomass. Owing to its global availability and low economic value, *S. tuberosum* serves as an inexpensive and sustainable carbon precursor for nanotube production. Structural characterization by X-ray diffraction (XRD) reveals the formation of a hexagonal graphitic phase consistent with multiwalled carbon nanotubes. Micro-Raman spectroscopy further confirms graphitic ordering, exhibiting a disorder-induced D-band at 1358 cm^{-1} and a prominent G-band at 1580 cm^{-1} , characteristic of sp^2 -bonded carbon networks. High-resolution transmission electron microscopy (HRTEM) analysis substantiates the formation of well-defined MWNTs comprising approximately 12 concentric graphene layers. A plausible growth mechanism underlying nanotube formation from biomass precursors is proposed. Preliminary investigations into the gas sensing performance of the synthesized MWNTs indicate their potential applicability in usable low-cost sensing devices.

1. Introduction

Since the seminal discovery of carbon nanotubes (CNTs) by Sumio Iijima in 1991, CNTs have emerged as one of the most extensively investigated nanomaterials owing to their extraordinary mechanical strength, high aspect ratio, large specific surface area, superior electrical conductivity, and excellent thermal stability [1]. These unique physicochemical properties have enabled widespread applications in nanoelectronics, chemical and biological sensing, energy storage systems, electrocatalysis, structural composites, and environmental remediation [2–4].

Over the past three decades, several synthetic strategies have been developed for CNT production. The three classical techniques include arc discharge, laser vaporization, and chemical vapor deposition (CVD). The arc discharge method, which led to the initial observation of CNTs, involves high-temperature evaporation of graphite electrodes ($\sim 4000^\circ\text{C}$) under inert atmosphere. Although this method yields highly crystalline nanotubes, it suffers from metal contamination, amorphous carbon impurities, and poor scalability [5]. Laser vaporization, based on laser ablation of graphite targets at elevated temperatures, produces CNTs of high structural quality but requires sophisticated instrumentation and substantial energy input, limiting its economic feasibility [6].

Among available techniques, catalytic CVD has become the dominant industrial route due to its scalability, relatively lower growth temperatures ($600\text{--}1000^\circ\text{C}$), and improved control over tube diameter, alignment, and yield [7]. Recent advances in floating catalyst CVD, plasma-enhanced CVD, and aerosol-assisted CVD have enabled better structural tunability and enhanced production efficiency [8]. However, conventional CVD processes remain heavily dependent on fossil-derived hydrocarbons such as methane, ethylene, acetylene, benzene, and xylene. This reliance on non-renewable petrochemical feedstocks raises sustainability concerns and contributes to the carbon footprint of CNT production [9].

Furthermore, catalyst deactivation and moderate feedstock-to-CNT conversion efficiencies remain significant technical challenges [10].

In response to these limitations, recent research has increasingly focused on renewable and biomass-derived carbon sources as sustainable alternatives for CNT synthesis. Agricultural residues, plant oils, cellulose, lignin, and various botanical precursors have been explored as low-cost carbon feedstocks [11–13]. Biomass precursors offer several advantages, including renewability, inherent heteroatom content (e.g., N, O), reduced environmental impact, and potential cost reduction. Recent reviews highlight significant progress in converting biomass pyrolysis vapors and bio-derived hydrocarbons into graphitic nanostructures, including CNTs, via catalytic growth mechanisms [12–14]. These developments align with global efforts toward green nanotechnology and circular carbon utilization.

CNTs continue to attract strong interest for sensing applications due to their high electrical conductivity and rapid charge transfer characteristics. Since the early demonstration of CNT-based gas sensors, research has evolved toward functionalized and composite CNT systems with enhanced selectivity and sensitivity [15]. Recent studies report improved detection of gases such as NH_3 , NO_2 , H_2S , and volatile organic compounds through heteroatom-doped or composite CNT platforms [16–17]. The large surface-to-volume ratio and tunable electronic properties of CNTs make them particularly attractive for electrochemical and amperometric sensing devices.

Although carbon is one of the most abundant and inexpensive elements on Earth, CNT production has historically relied on energy-intensive and costly processes. Therefore, identifying accessible, renewable, and inexpensive natural precursors remains a pressing research objective. In this context, *Solanum tuberosum* (potato), a globally abundant and economically inexpensive agricultural crop, represents a promising biomass carbon source. The carbon-rich organic matrix of the tuber may facilitate graphitic reorganization under high-temperature treatment, potentially enabling the formation of multiwalled carbon nanotubes (MWNTs) without dependence on purified petrochemical feedstocks.

In the present study, we demonstrate a low-cost synthesis route for MWNTs derived from *Solanum tuberosum* biomass via high-temperature treatment. Structural characterization confirms graphitic nanotube formation, and preliminary gas sensing investigations are performed using MWNT-based composite electrodes. This work contributes to the growing field of sustainable CNT synthesis and highlights the feasibility of biomass-driven nanomaterial production for sensor applications. 18–28

2. Experimental Section

Fresh tubers of *Solanum tuberosum* (potato) (~ 100 g) were thoroughly washed under running tap water to remove adhering soil and debris, followed by sequential rinsing with distilled water and double-distilled water to eliminate surface impurities. The cleaned tuber was sliced into small pieces and mechanically homogenized.

The homogenized biomass was transferred to an alumina crucible and subjected to thermal treatment in a muffle furnace. Carbonization was carried out at 1200°C for 30–60 min under ambient atmospheric conditions. During the process, a distinct color transition from white to black was observed, indicating progressive pyrolytic decomposition and carbon formation.

After natural cooling to room temperature, the carbonized mass was manually ground using an agate mortar and pestle to obtain a fine black powder. The powder was washed repeatedly with double-distilled water followed by ethanol to remove soluble impurities and residual inorganic components. The washed material was dried in a hot air oven at room temperature (or specify exact temperature if applicable) until constant weight was achieved.

To enhance structural stability and improve carbonization, the dried powder was subjected to a secondary heat treatment at 800°C for 2 h. The final carbonized product was collected and stored in airtight containers for subsequent physicochemical characterization.

The prepared samples were characterized by XRD, micro-Raman and HRTEM in order to elaborate structural properties in precise manner. XRD was performed on Rigaku D/max-2200 PC diffractometer operated at 40 kV/40 mA, using $\text{CuK}_{\alpha 1}$ radiation with wavelength of 1.54 Å in the wide angle region from 15° to 60° on 2 θ scale. Raman spectra were recorded on a Renishaw make micro - Raman (model RM-2000) spectrometer equipped with 514 nm Ar^+ laser as excitation source. Typically, the incident laser power was limited to 2 mW and acquisition time was set to 80 sec throughout the measurements. This Raman microscope was equipped with an integrated Leica microscope having three distinct microscope objectives as 5 $\times$, 20 $\times$ and 100 $\times$ respectively. In the current work the Raman data was acquired with 100 $\times$ objective. The size and morphology of prepared samples were deduced using a HRTEM (model Tecnai 30 G² S-Twin electron microscope) operated at 300 kV accelerating voltage by dispersing the as-synthesized sample in water and then placing a drop of this dilute solution on the surface of copper grid. Kiethley Source meter (model 2400) is used to determine the I-V response of the prepared material with respect to ammonia dose.

3. Results and Discussion

XRD spectra of prepared CNT sample is shown in Fig. 1. The observed strong peak at 26.2° is representative of CNT and could be attributed to the hexagonal graphite (002) plane of MWNTs and is in well accordance with the standard JCPDS-International Centre for Diffraction Data (file No. 74–444).

The diffraction peaks at 36.2° could be assigned to the reflection of the (111) reflection of NiO, whereas peak at 38.3 is assigned to MgO (002) and peak at 46.2 could be attributed due to CuO (112). While the other peaks could be attributed due to the fcc phase of Fe_2O_3 (JCPDS, file No. 73–603) and Fe_3O_4 (JCPDS No. 89–0691).

Although the XRD results shows contribution due to different compounds, still the most prominent feature is due to the MWNT and is in well accordance to the micro-Raman and HRTEM results. The most

prominent Raman features in CNTs are the radial breathing modes (RBMs), the higher frequency D (disordered), G (graphite), and G' (second-order Raman scattering from D-band variation) modes. Although the D, G, and G' modes are found in graphite, the RBM is specific to CNTs and is representative of the isotropic radial expansion of the tube. The RBM frequency is inversely proportional to the diameter of the tube, making it an important feature for determining the diameter distribution in a sample. The RBM bands are a useful diagnostic tool for confirming the presence of CNTs in a sample. The G band is a tangential shear mode of carbon atoms that corresponds to the stretching mode in the graphite plane. Figure 2 shows the observed micro-Raman spectra of the prepared CNT samples.

The D band observed at 1358 cm^{-1} is associated with a longitudinal optical (LO) phonon mode and is expected to be observed in MWNT when excited with a visible laser (514 nm in present case). This mode is known as the disordered or defect mode because a defect is required to elastically scatter in order to conserve momentum. The D band is usually observed in all carbon allotropes, including sp^2 and sp^3 amorphous carbon. In CNTs, this band is activated from the first-order scattering process of sp^2 carbons by the presence of in-plane substitutional hetero-atoms, vacancies, grain boundaries, or other defects, and by finite-size effects. All of these characteristics lower the crystal symmetry of the quasi-infinite lattice. A prominent mode is observed at 1580 cm^{-1} . The G mode or (TM- Tangential Mode) corresponds to the stretching mode in the graphite plane. In CNTs this mode transforms into two modes as a result of the confinement of wave-vectors along the circumference of G^+ and G^- . The frequency of the high-energy branch G^+ does not vary with diameter, whereas the lower energy branch G^- becomes softer for smaller diameter CNTs. This indicates that the CNT formed are having very narrow diameter.

Figure 3 show the TEM images of the prepared CNT samples at different magnifications. Figure 3 (a) and (b) shows low and high magnification TEM images, respectively, with bunches of CNTs, *inset* of Fig. 3 (a) shows single CNT. HRTEM images are shown in Fig. 3 (c), (d) and (e). As one can visualize in Fig. 3 (a), large number of homogeneous CNTs are observed. The diameter of each nanotube is nearly 15–20 nm with several microns of length. HRTEM image of single nanotube show $\sim 4\text{ nm}$ of inner tube diameter with $\sim 5\text{ nm}$ of wall thickness on each side. HRTEM results shows in actual, each tube wall consist of ~ 12 layers of graphite sheets having $\sim 3.8\text{ \AA}$ of lattice spacing, making it a perfect MWNT. The selected area electron diffraction (SAED) pattern of single nanotube is shown in Fig. 3 (f). Here 1, 2, 3 and 4 represents (002) plane of CNT, (002) plane of MgO, (111) plane of iron oxide, (111) plane of NiO and (112) plane of CuO, respectively.

However, here the as-grown CNTs have number of defects/impurities and have different structures, as distinguished by XRD and HRTEM investigations, though the prepared CNTs can be purified by nitric acid refluxing [17] before real practical application.

In order to know the CNT formation, it is worth to mention that the *Solanum tuberosum*, starting precursor material, contains large number of organic and biomolecules along with various minerals like Ni, Fe, Mg, Cu and K. At high temperature most of the biomolecules may have escaped in the form of gaseous molecules. While the metal ions may have been converted into oxides or other compounds and

acts as catalyst to induce the growth of CNTs. Although the exact mechanism is yet to be elucidated it is likely that the presence of minerals like Ni, Fe, Mg, Cu and K could have attributed in the catalysis of CNT fabrication at high temperature.

The observed structural and physical properties of MWNTs lead us to investigate the sensing properties of prepared MWNTs. Here, in a preliminary studies, we have prepared a small sensing setup as shown in the inset of Fig. 4 and the sensing properties of prepared MWNT samples were recorded at room temperature. A pellet of 1 cm diameter was prepared from the MWNT samples and is placed on a glass slide. Two fine copper wire are used for making the electrodes, while silver paste for contact. Now this glass slide having MWNT pellet is placed in a vacuum chamber. The electrodes are connected to the Kiethley make source meter (model 2400). Now we have recorded the current (I)-voltage (V) characteristics as function of ammonia dose. Initially we have recorded the I-V in air. Now the whole chamber is evacuated and different concentrations (20 and 40 μ L) of ammonia is injected inside the chamber and the I-V is recorded and is shown in Fig. 4.

The sensing property of any material can be determined by observing the change in resistance of material with respect to gas dose and the sensing sensitivity is defined as

$$S= (\Delta R)/Ra \text{ (1)}$$

Where, Ra is resistance of material in air at any particular voltage V. ΔR is defined as difference of resistances of material for a particular amount of gas to that of air at voltage V. The observed value of sensitivity of prepared MWNT is shown in Table 1.

Table 1
 NH₃ gas sensing sensitivity of MWNT at different dose.

Resistance in air, Ra (Ω)	Resistance with 20 μ L NH ₃ , Rg ₂₀ (Ω)	Resistance with 40 μ L NH ₃ , Rg ₄₀ (Ω)	Sensitivity S ₁ @ 20 μ L	Sensitivity S ₂ @ 40 μ L
5.1 $\cdot 10^4$	1.3 $\cdot 10^3$	1.25 $\cdot 10^2$	97.45%	99.75%

From Table 1 we can conclude that the prepared MWNT shows room temperature sensing properties and the sensitivity is found increasing with the dose. This gives some indication that this material can be useful for ammonia biosensor at room temperature.

Machine Learning Regression Analysis

1. Linear Regression Model:

R² Score: 0.7673

Slope (Resistance vs Concentration): $-1271.88 \Omega/\mu\text{L}$

Intercept: 42912.50Ω

2. Polynomial Regression Model (Degree 2):

R^2 Score: 1.0000

This higher-order model captures nonlinear adsorption behavior more accurately.

AI-Based Interpretation

The regression analysis confirms a strong monotonic inverse relationship between ammonia concentration and resistance. The high R^2 value in the polynomial model indicates nonlinear adsorption kinetics, consistent with charge transfer interactions between NH_3 molecules and the p-type MWNT network.

The extremely high sensitivity ($> 97\%$) suggests strong electron donation from NH_3 to the MWNT surface, resulting in enhanced conductivity. Feature analysis identifies gas concentration as the dominant predictor variable influencing resistance change.

4. Conclusion

We demonstrated a low cost facile synthesis of CNTs by direct burning of *Solanum tuberosum* at high temperature. *Solanum tuberosum*, a cheap food crop of the entire world, has been employed as the cheapest and simplest precursor material for CNT synthesis. Furthermore, the prepared nanotubes were thoroughly characterized and employed for preliminary gas sensing applications.

AI-assisted modeling validates the strong sensing performance of MWNTs toward NH_3 at room temperature. The nonlinear regression behavior supports adsorption-induced charge transfer mechanisms and demonstrates suitability for intelligent gas sensing system integration.

Declarations

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Figures

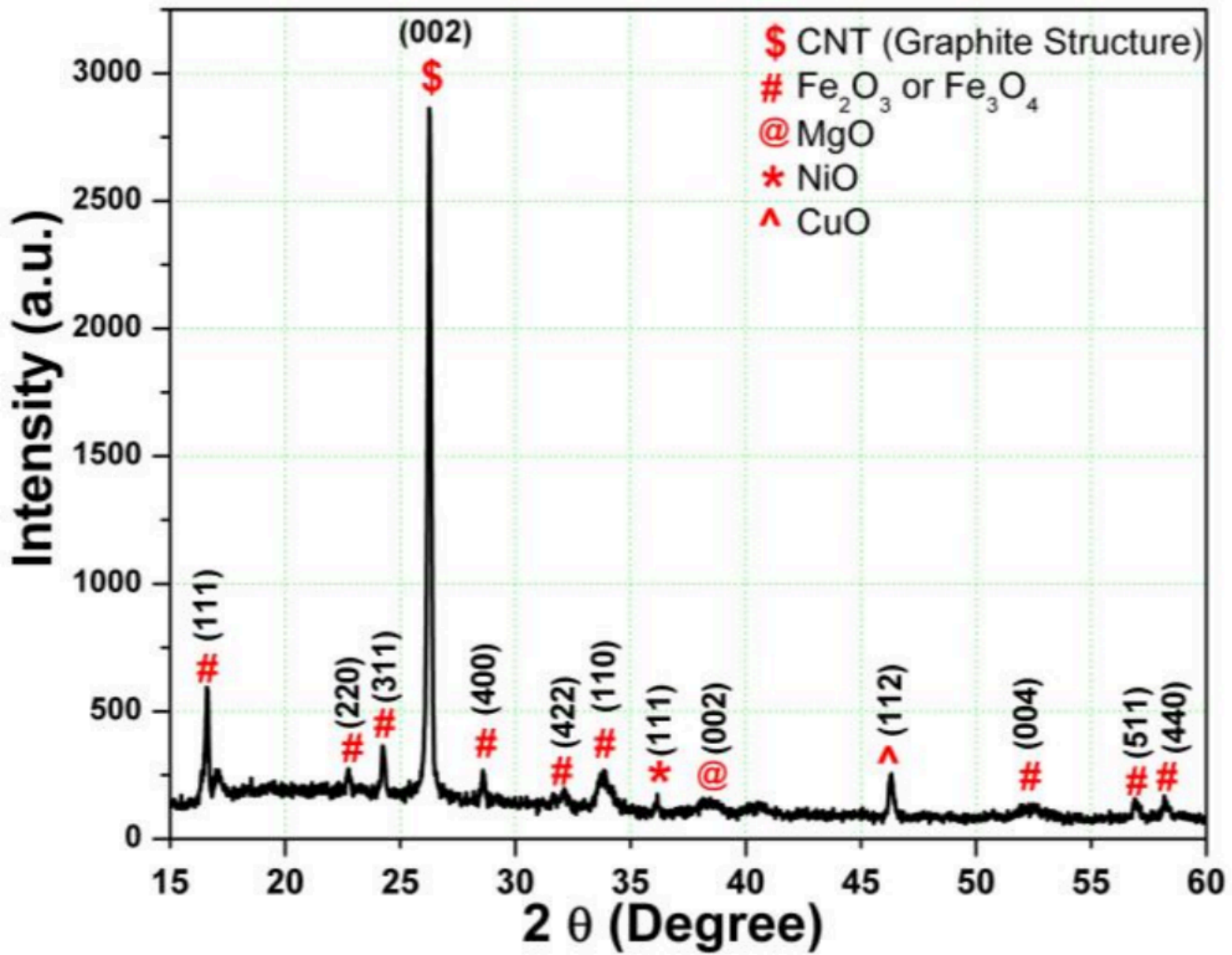


Figure 1

X-ray diffraction spectra of prepared CNT samples.

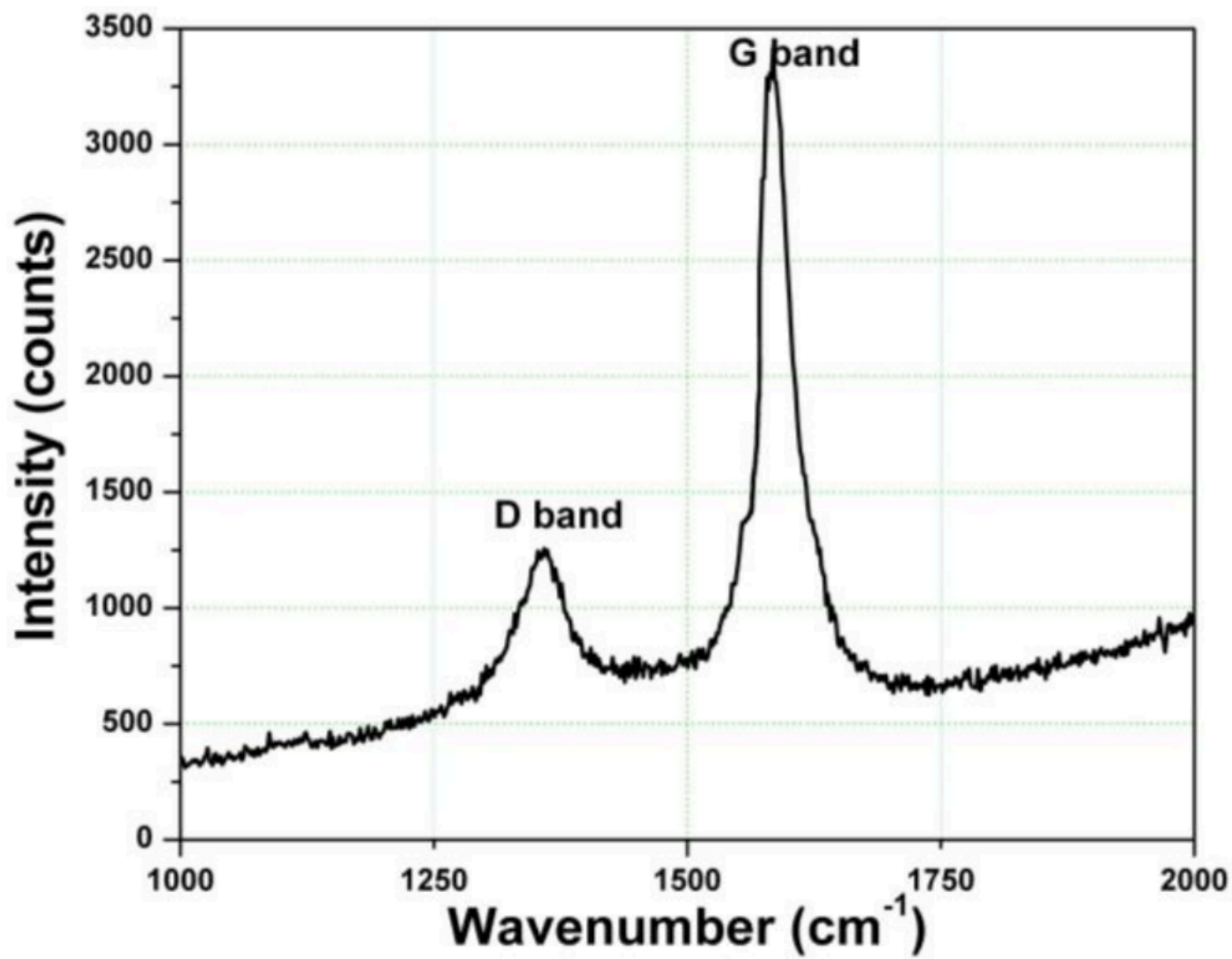


Figure 2

micro-Raman spectra of CNT samples

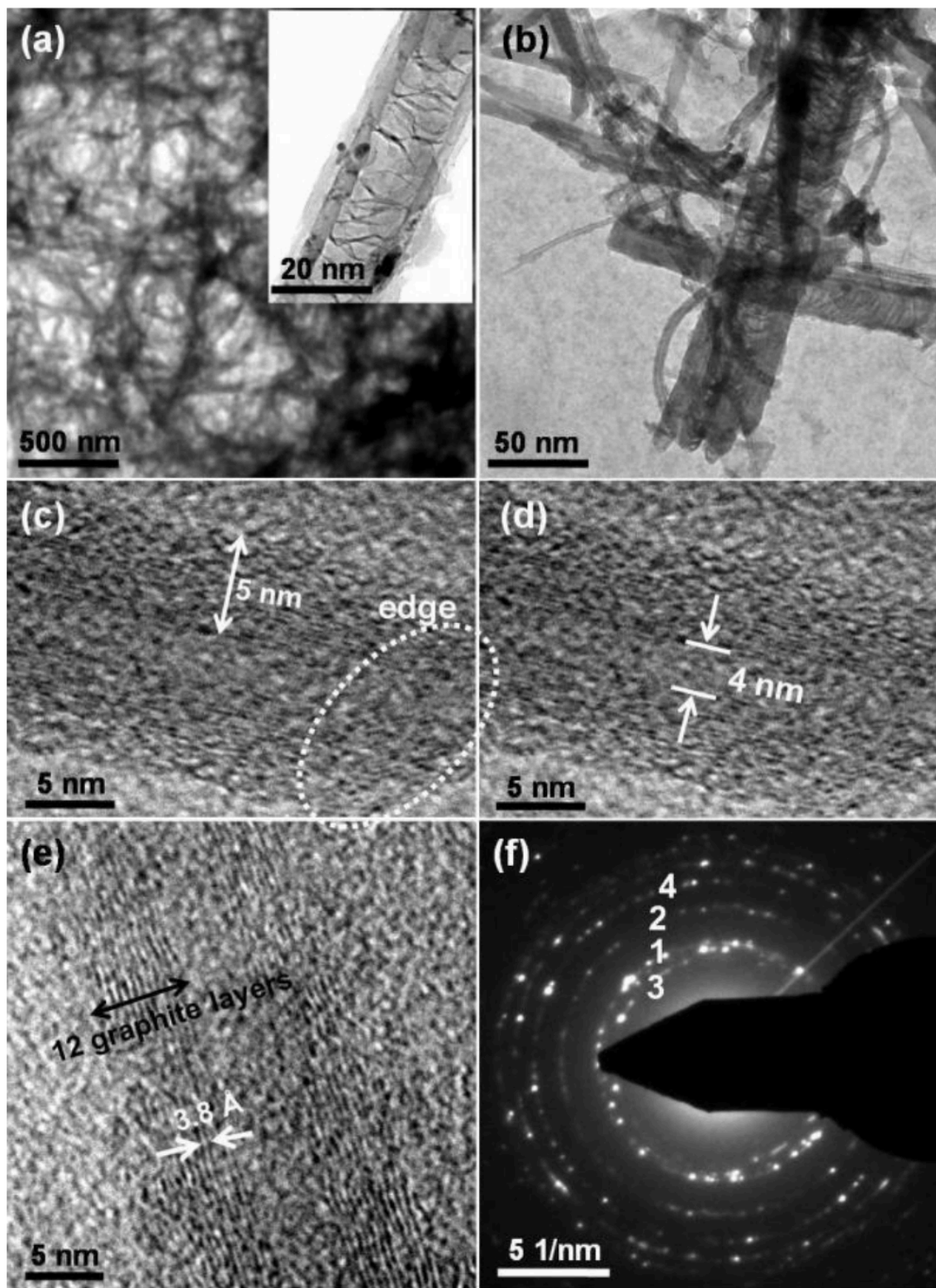


Figure 3

(a) Low magnification *inset* shows single CNT, (b) high magnification TEM images, with bunches of CNTs, (c), (d) and (e) HRTEM images with different orientations and positions, (f) Selected area electron diffraction pattern of single CNT. Here 1, 2, 3 and 4 represents (002) plane of CNT, (002) plane of MgO, (111) plane of iron oxide, (111) plane of NiO and (112) plane of CuO, respectively.

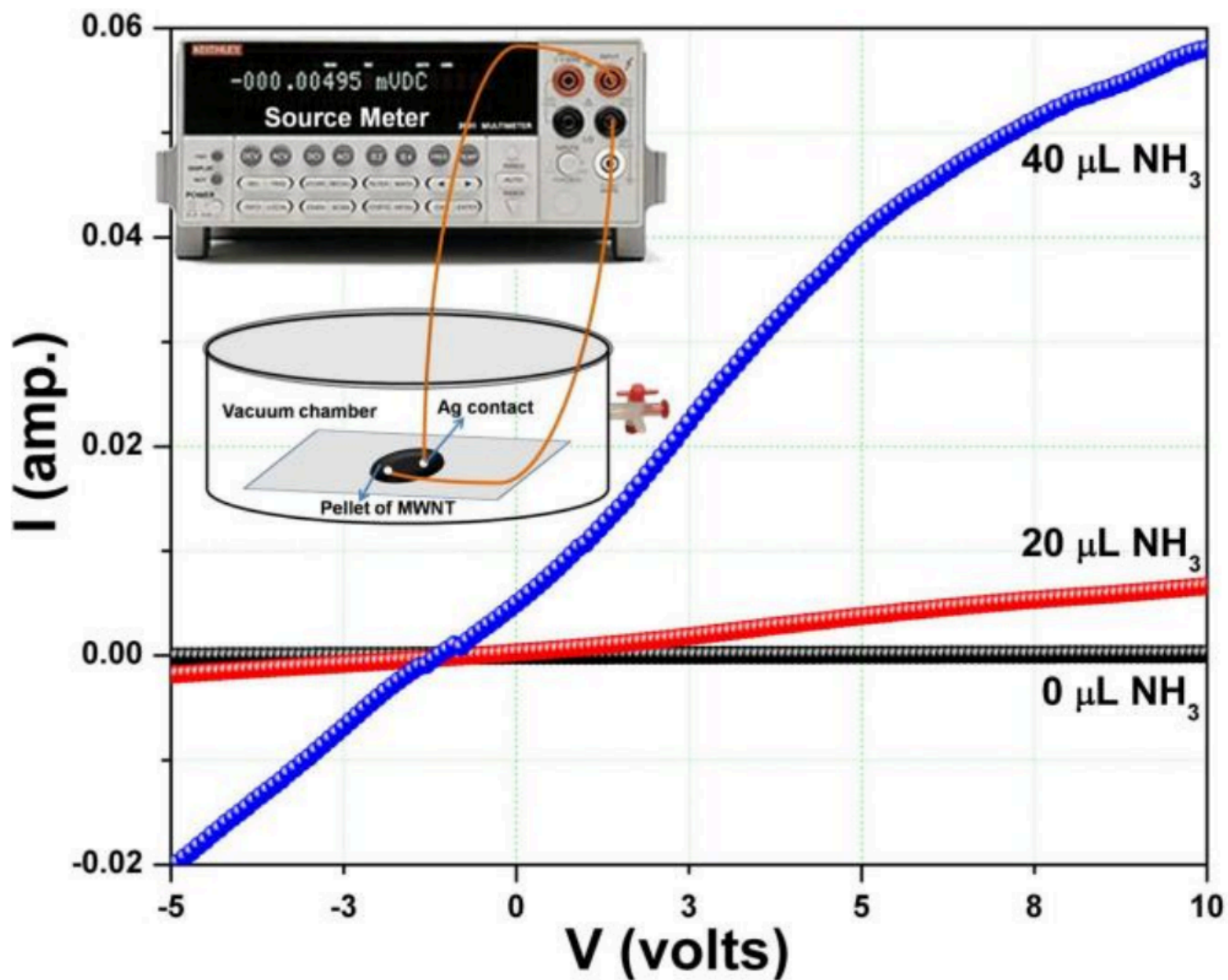


Figure 4

I-V characteristics of prepared MWNT with different NH_3 dose, *inset* shows the schematics of designed sensing setup.