

# THE DOCTORAL RESEARCH

## ABSTRACT

Volume: 1, Issue: 1 May 2012



# FIRST ISSUE



INSTITUTE of GRADUATE STUDIES

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IPSis Biannual Publication

**Name :** Suriani Binti Abu Bakar, PhD  
**Title :** Controlled Growth Of Vertically Aligned Carbon Nanotubes From Palm Oil Precursor Using Thermal Chemical Vapour Deposition Method And Its Field Electron Emission Properties

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Vertically aligned carbon nanotubes (VACNT) were synthesized using palm oil as an environmentally friendly starting material. The synthesis was carried out in a thermal chemical vapour deposition reactor. Parametric studies were done to determine the optimum parameters to obtain VACNT with favourable properties at high volume. The parameters included seeded and floated catalyst preparation method, stacking substrate configuration (lower and upper growth), synthesis temperature (700-900°C), palm oil vaporization temperature (300-600°C), synthesis time (5-90 min), different carbon precursor (palm oil and waste cooking palm oil), substrate positioning (position 1-6), ferrocene concentration (0.67-5.33 wt%) and different carrier gas (argon and nitrogen). The carbon nanotubes (CNT) products were then characterized using several analytical techniques which were electron microscopy, energy dispersive x-ray analysis, micro-Raman and Fourier transform infrared (FTIR) spectroscopy, thermogravimetry analysis (TGA) and CHNS-O analysis. Prior to the synthesis process, several analyses such as TGA, gas chromatography-mass spectrometry and FTIR characterizations were done on the carbon precursor namely palm oil and waste cooking palm oil in order to facilitate the optimization procedures of VACNT. For every synthesis parameter, the nanotubes growth rates were measured and the nucleation as well as termination factor were investigated. CNT diameter, degree of alignment, crystallinity and purity were extensively studied as they were found to be greatly affected by the synthesis parameters.

Based on the inspection of the morphology and crystallinity of CNT it was found that the following parameters can be considered as the optimized parameter to produce higher quality of bulk VACNT in our reactor; the floated catalyst and lower growth approach at the synthesis temperature in range of 750-800°C, precursor vaporization temperature in the range of 400-500°C, the synthesis temperature of 15 to 35 mins, sample position at P2 and P3, and ferrocene concentration of 1.33 - 5.33 wt%. Synthesizing VACNT within nitrogen ambient produces higher VACNT growth rate with considerably more bamboo-liked structure as compared to argon ambient. In this study, we have also demonstrated that waste cooking palm oil from domestic frying can be utilized as an efficient, economical and environmentally friendly carbon source for VACNT and bulk CNT synthesis. A mixed bottom-tip growth model has been proposed for the floated catalytic CVD synthesis with the bottom growth mechanism was believed to take place in the early stage of the synthesis. The VACNT growth can also be initiated by the yarmulke growth mechanism. The yarmulke growth has been used to explain the presence of tubes with bamboo-type structure. By examining CNT synthesized under different conditions, it was found that an amorphous carbon coating of roughly 8 nm within 6 min synthesis time was sufficient to completely terminate the growth. However in a controlled condition, the growth was not expected (not) to terminate even though for 1 hour synthesis time. We also assessed the potential of palm oil based VACNT as field emitter by measuring its field electron emission (FEE) properties. Field emission from the VACNT synthesized within nitrogen ambient at 2.5 wt% ferrocene concentration indicated the lowest turn-on field at 2.95 V $\mu\text{m}^{-1}$  which corresponded to the current density of 10  $\mu\text{Acm}^{-2}$ . The threshold field was observed to be about 3.55 V $\mu\text{m}^{-1}$  at 1 m $\text{Acm}^{-2}$ . The maximum current density of 7.30 m $\text{Acm}^{-2}$  measured was obtained for 4.30 V $\mu\text{m}^{-1}$  and good emission stability with low degradation. Electrode separation of 200  $\mu\text{m}$  gave the best FEE performance and smaller or larger electrode separations gave inferior results. It can be concluded that the VACNT from the bio-hydrocarbon precursor palm oil was stable for applications in field emission devices such as flat panel displays and flat lamps.