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Comparative studies for Synthesis Carbon Nanotubes by Deposition By composition method

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Abstract. This work introduces a concept of deposition by composition method for synthesis carbon nanotubes CNTs from two types of coal where used as sources of carbon to produce multi walls tubes. Qualitative analysis were done by analysis with XRD and EDX. The coal second types of coal C2 was more ability for ignitions which did not succeed to growth CNTs, while first types of coal C1 was succeeded to precipitate CNTs on the surface of the flame method. The variant in abilities for growth CNTs was related to the internal inhibitions when increase with increased the abilities of ignitions which supposed to high concentrations of free radicals.

Keywords: Coal, Calcium, EDX/XRD, CNTs.

1. Introduction

Many attempts were developed for sensitive and selective technology for production carbon nanotube CNTs with different types such single, double or few walled carbon, and multi-walled carbon nanotube SWCNT, DWCNT, FWCNT, MWCNT respectively [1]. The common method for synthesized CNTs were [1] chemical vapor deposition CVD, arc-discharge ARD, laser ablation LA and flame method FL or decomposition method [2]. The sources used for synthesis CNTs mostly hydrocarbons such saturated and unsaturated hydrocarbons [2-3]. Under low temperature CNTs was produced by FLs and the process may include another parameter to judge about the probabilities for forming CNTs. The FLs technique [3-4] required burning materials in gas, liquid or solid phase and forming different fragments or species then precipitation in cold surface or less temperature as compare with ignitions flame. The cheapest and may be typical natural sources material compared to graphite, acetylene, methane and many hydrocarbons is coal [5]. Descript and define coal needed to giving simple and easy understand for coal, thus graphite, can be use by make compare with each other. An allotropic form of pristine carbon with sp2 hybridization refer to graphite while, solid continents of organic and inorganic macromolecular with disorder orientations of carbon and hybridization refer to coal. Ideal investigations required take full advantage of coal for synthesis CNTs such quality and quantities of carbon/impurities, which represent the critical parameter. Coal differs in its ability to combustion depending on the nature of the components [6]. As we reported in our previous work [3] MWCNTs were synthesized without and with catalyst [4] from coal by homemade reactor. Experimentally the variance in possibility for converting random carbon content to regular composition can be done by methods that mentioned previously. The variance was related to combustion or degradation efficiency of coal which represent the best method to extraction the carbon from solid state to gases state then rearrange the carbon atoms by precipitated to CNTs [5-7]. The flame method [7] depends on combustion of source with forming free radicals of carbon atoms were responsible to build tubular structures on the precipitation surface.

Specific heat capacity of coal which responsible to make ignitions were influence with critical parameters such ratios and physical state of carbon, moisture and volatile materials [8-9]. Generally specific heat capacity of coal inversely

proportional with carbon content and directly proportional with volatile matter content [9-10]. Hydrogen, calcium and other hetero-atoms [8] were occurred in high volatile or nonvolatile materials which may inhibit the carbon species that could forming CNTs. Forming high carbon species such free radical or ions may refer to formation of CNTs or may inhibition for each ether and that prevents produce CNTs. In this work the the heat capacity and specific heat which did not listed in this work wear depended to make mechanism or suppose mechanism for forming CNTs. The research also aims to highlight in the types of coal with relationship between physical and chemical properties and the possibility of producing CNTs.

Experimental:

1. **Materials:**

In this work, two kinds of coal were donated C1 and C2 imported from Malaysia, the first was Light in weight, low in density, needs a minute to ignite, and its glow lasts for about five minutes without smoke. The second C2 was more weight and density, it takes three minutes to ignite, with glow continues for about two minutes and a flame accompanied a little smoke, and the two types C1 and C2 were analyzed by EDX analysis .

2. **Method of synthesis by deposition by decomposition method**

The process as represented in figure 1, were don with closed chamber 20 L in volume which supported with two section to inter gases (N2 and O2) in addition to slit which behave as excuse for formed gases during reaction. The reaction was including two steps: the first was burning the coal (500 g) in chamber without N2 for 30 min with oxygen gas (400 cm3/min). The second step was allowed to flow N2 gas (100 cm3/min) for 15 min before stopped the reaction.



Figure 1: *Skim of chamber precipitation CNTs from two types of coal by flame method*

3. **EDXs and XRDs analysis**

The structure and continent for two types of coal was analysis by a JEOL 7500F Field Emission scanning electron microscope, equipped with energy-dispersive X-ray (EDX).The X-ray diffraction (XRD) was performed in a Bruker AXS D4 using, Cu Kα1, and Kα2 radiation at wavelength 1.54056 nm and a nickel filter. The width step was 0.02° per step in the 2θ range from 5 to 90

Results and Discussion:

The elements analysis EDXs for the two samples C1 & C2 were represent in figure 2 and 3, which shows 3.8% Calcium in sample C1 while C2 did not shows any ratios for Calcium. The calcium in coal mostly exist as Ca(CO3)2 which represent the common inhibitor for ignition [13] thus that may be explain the bad ignition for C1. The influence of Calcium as inhibition reduces generation carbon free radical in gases phase or prevent converting carbon to CO2 with keeping stability for CO [14]. The influence of Si or Na did not shows or reported any inhibition effect towards coal ignition.

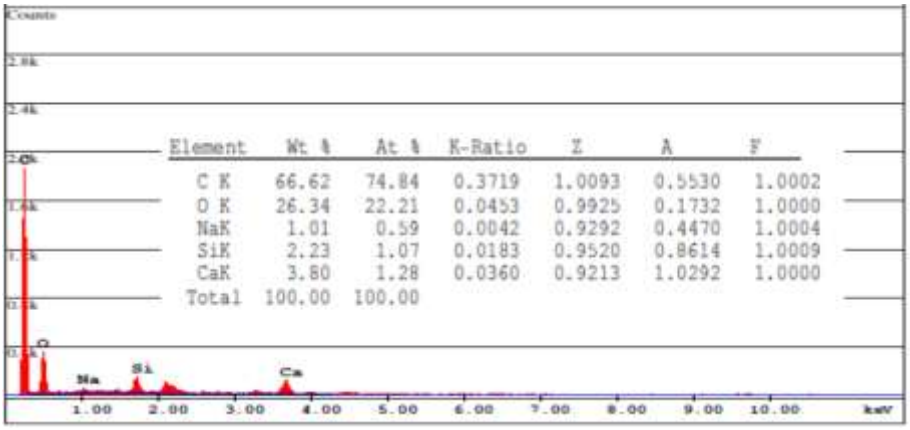


Figure 2: *EDX spectrum for coal C1 which used as source for synthesized CNTs by flame method.*

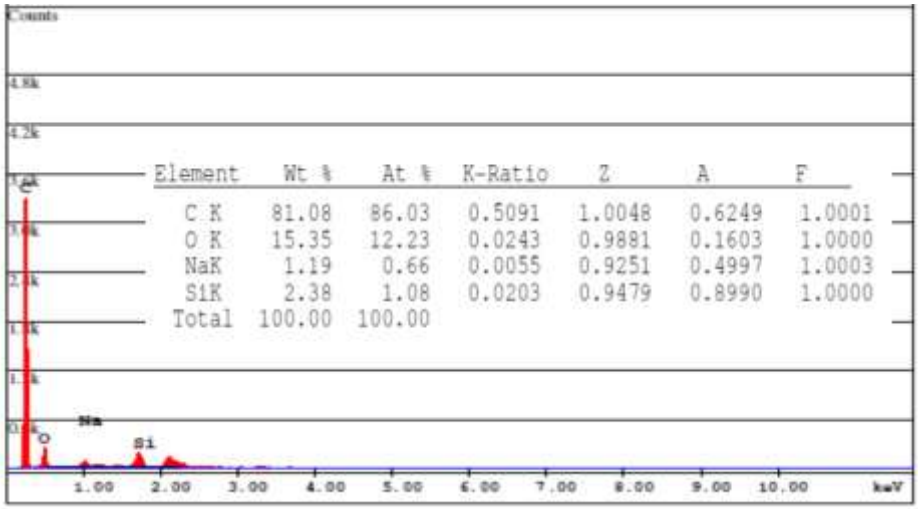


Figure 3: EDX spectrum for coal C2 which used as source for synthesized CNTs by flame method

Compare of XRDs patterns for C1 and C2 was represented in Figure 4, which is taken from 2Θ = 5° to 90°. The XRDs patterns for C2 was more purities with strong peak at 2Θ= 24° and week peak at 2Θ= 44° [15-16] with 001 and 200 planes and that refers to tubular structure. The case was variance with C1 when shows sharp and strong peak at 2Θ= 29° and less strength at 22°, 36°, 40°, 48°, 49° which related to calcium carbide or oxide [14,17] in additions to the two peaks for unconverted coal.

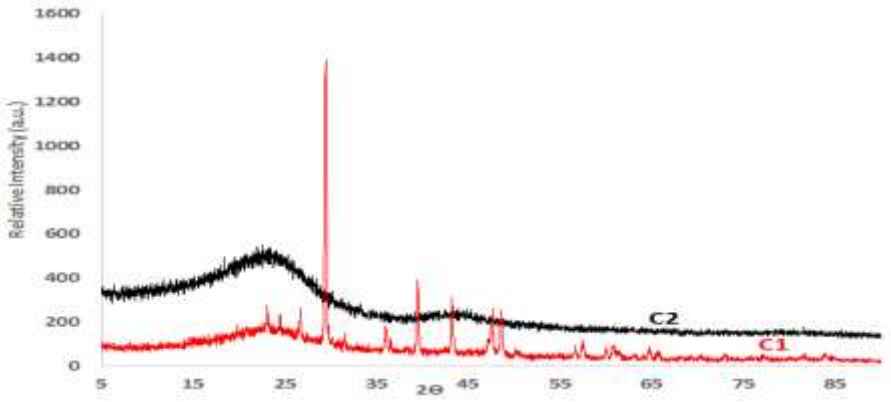


Figure 4: *XRD patterns for coal C1 (bad coal) & C2 (good coal)*

Through own results, studying coal composition which represented by determined XRD and EDX analysis, it could be explain the variance between C2 and C1. The reason behind variance in behaviour C1 and C2 can be related to three reasons: the first higher abilities for C2 to ignitions completely or at least most of it convert to CO2 and very low percent could be forming black carbon [18]. The second: high concentrations of free radicals may cause internal inhibitions for each other's before reach to the surface of condensation or precipitation which prevent forming tubular structures. Third: low ignitions efficiency for C1 reduce the flow of free radicals and increased the probabilities for forming CNTs [14]. When we take the details of flame method the process depends on ignitions the source (C1 or C2) causing flies away for different species towards precipitation surface. The condition produces enough ratios of flies' carbon species with less inhibitions, which needed for control between how many flay and how many inhibitions. When the ratios of flay carbon species after ignitions prevent the inhibitions absolutely rises CNTs growth accrued with C1. While the higher abilities of ignitions enhance forming inhibitions for the flay carbon species which completely prevent growth CNTs.

The SEM images which reported in Fig. 5 and 6 were shown variance in structure when tubular structure with many aggregates due toVan der Waals forces [3-10] while the other image were shown planer structure of carbons materials which represent unconverted carbon.

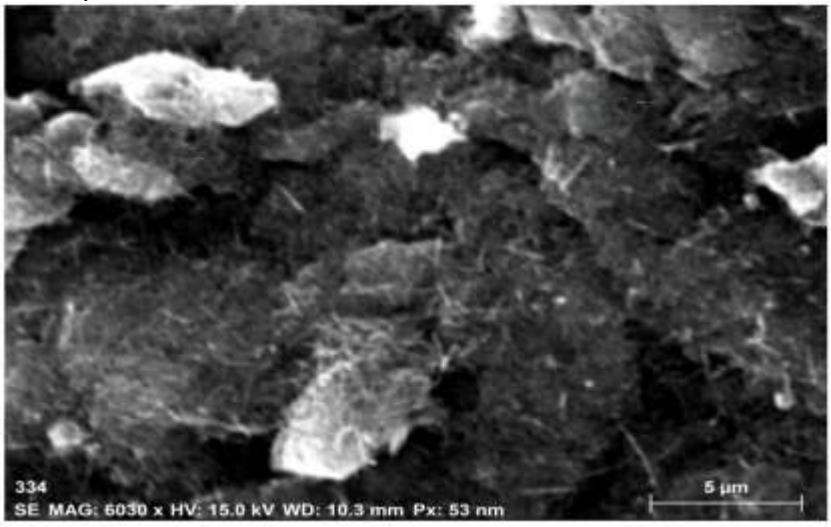


Figure: *SEM image for synthesized MWCNTs from good qualities of coal.*

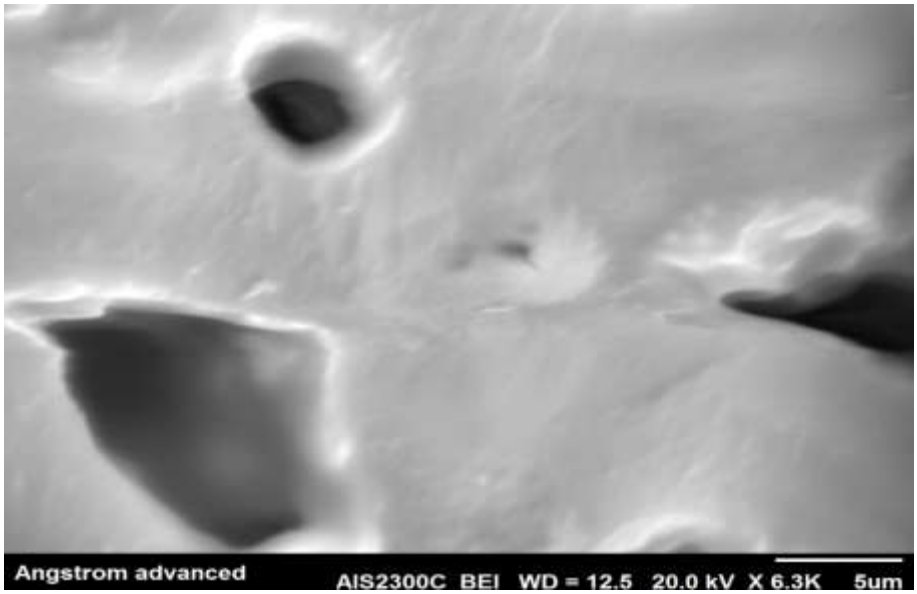


Figure: *SEM image for carbon precipitation on the surface of reactor from bag quality of coal*

Conclusion

Despite extensive effort were deals with used coal as sources of carbon for synthesis CNTs, but the secrets and mysteries of this topic is still needed more and more laboratory work. The mechanism of growth CNTs were shown many variances between chemical vapour deposition and flame from. Because the catalyst and method with condition of preparations it is not the determinants main to prepare CNTs but the nature of physical and chemical properties of precursor form critical parameters. However, the analysis by quantity analysis by XRDs and EDXs enhance the view towards the effect and sensitivity of coal composition for synthesized CNTs.

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