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Effect of Synthesis Temperature on the Growth of Carbon-based Materials from Waste Engine Oil Precursor

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Abstract

Different stuctures of carbon material were succesfully synthesized from waste engine oil (WEO) as carbon source using double-stage thermal chemical vapor deposition method. In this work, 5.33 wt% ferrocene was used as catalyst, precursor temperature at 450°C and the synthesis temperatures were varied from 600-1000°C with 100°C increament. The prepared samples were characterized using field emission scanning electron microscopy and micro-Raman spectroscopy. Below 700°C, amorphous structure of carbon was formed. Well growth carbon spheres were produced at 800℃ while at 900℃, bigger diameter and lower crystallinity of carbon spheres were resulted. At very high temperature, 1000℃, a highly defective structure of carbon was produced. These results show that the structure of carbon materials from WEO precursor was highly affected by synthesis temperature changes.

Keywords: *waste engine oil, carbon-based material, thermal chemical vapor deposition*

INTRODUCTION

Carbon is an abundant element in nature and can be formed into many kind of carbon-based materials such as carbon nanofibers, carbon nanotubes (CNTs), carbon black nanoparticle, graphene, conductive carbon, carbon dye, mesoporous carbon (Poudel and Qiao) and carbon spheres (Zobir, Abdullah et al.). These carbon-based materials have many applications in electrical and optical devices such as solar cell (Poudel and Qiao), field electron emission (Asli, Shamsudin et al.), microcable (Shanov, Cho et al. 2013), transistor (Donev 2009), energy storage, filled composites, nanoprobes and sensors (Ajayan and Zhou 2001; Wilgosz, Chen et al. 2012). They also have been applied in sports equipment, automotive and textile (Nowack, David et al. 2013). Because of the great properties and promising applications, the researchers have been intensely studied about these materials since the last two decades.

Several methods have been used to synthesis carbon-based materials, but the use of thermal chemical vapor deposition (TCVD) method is the most popular method among others due to its simplicity, easy to scale up and still produce the high quality products. The selection of carbon precursor becomes very important in carbon-based materials synthesis. Unfortunately, the use of conventional hydrocarbon such as methane, propane, butane, hexane, acetylene and ethylene as carbon sources to produce carbon-based materials are not cost effective due to the expensiveness. We have recently reported the use of waste materials such as waste cooking palm oil (Suriani, Md Nor et al. 2010) waste chicken fat (Suriani, Dalila et al. 2013) and waste engine oil (WEO) to

produce CNTs (Suriani, Alfarisa et al. 2015; Alfarisa and Suriani 2016).

WEO contains polycyclic aromatic hydrocarbons (PAHs) and metal contaminants that are harmfull for living organisms if not treated properly. We investigated the synthesis temperature changes to the structure, morphology and crystallinity of carbon-based materials from WEO. Previously we reported the effect of catalyst consentrations on the growth of CNTs from WEO (Alfarisa, Safitri et al. 2016). Here, the effect of synthesis temperatures was analyzed on the growth of carbon based materials from WEO precursor.

MATERIALS AND METHOD

The synthesis of carbon-based materials from waste engine oil was done using double stage TCVD furnace with fixed precursor vaporization temperature at 450°C based on thermogravimetric (TGA) analysis in Fig. 1(a). Silicon substrates with 1 cm^2 area were ultrasonically cleaned using acetone, methanol and DI water before being used. The substrates were than placed into synthesis furnace and the temperatures were varied from 600 - 1000°C. Waste engine oil was filtered first before being mixed with 5.33 wt% of ferrocene as catalyst. After stirring for 30 minutes, 3 mL of the mixture was put in alumina boat and then loaded into precursor furnace. Argon (Ar) gas was flushed out during the first 10 minutes before the synthesis process starts. Precursor furnace was turned on after the synthesis furnace reached the set temperature. The synthesis process lasted for 30 minutes under Ar gas flow. The samples were collected and characterized using field emission scanning electron microscopy (FESEM-Hitachi SU8020) and micro-Raman spectroscopy (Renishaw InVia microRaman System).

RESULTS AND DISCUSSION

Another parameter conditions other than synthesis temperature were kept fixed in this experiment. Different structures of carbon-based materials at various synthesis temperature from waste engine oil are shown in Fig. 1(b)-(f). When the precursor furnace temperature reach 185°C, ferrocene molecules start to vaporized (Cheng, Li et al. 1998) and were deposited on the substrate in synthesis area via Ar gas flow. At synthesis temperatures below 700°C, amorphous structures of carbon materials were produced. It was assumed that the temperatures were not high enough to completely decompose the precursor and caused the low catalytic activity of Fe particles. Surprisingly, at 700°C, a material contained copper (Cu) and aluminum (Al) were produced along with carbon microspheres. It was believed that the natural presence of Cu and Al contaminants from WEO during the lubricating process were promoted the growth of this material. When the temperature was increased to 800°C, high density of carbon microspheres were formed. Diameter of the spheres were range from 0.17-2.57 μm. Carbon microspheres with lower crystallinity and bigger size were also produced at 900°C. The spheres diameter range were from 0.57-3.14 μm. Larger agglomeration of Fe particles were formed at higher temperature and caused the bigger diameter of carbon microspheres. The changes and destruction of carbon structure was occurred when the temperature was raised to 1000°C.

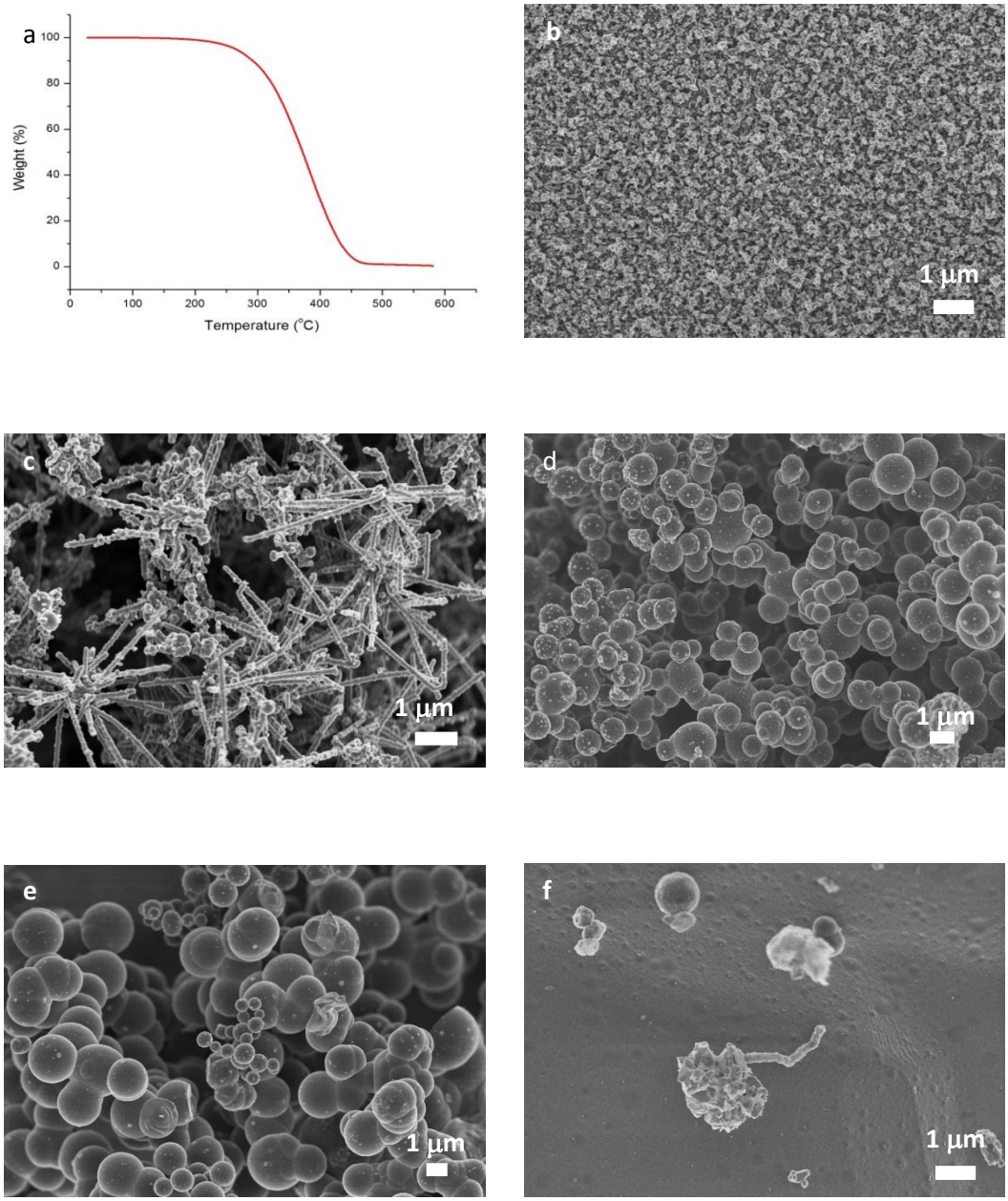

Fig. 1. (a) TGA curve of waste engine oil and FESEM images of CNTs synthesized at different temperature: (b) 600°C, (b) 700°C, (c) 800°C, (d) 900°C and (e) 1000°C

Fig. 2. shows the micro-Raman spectra of carbon-based materials. Two dominant peaks were clearly seen at ~1360 and ~1590 cm^{-1} represented D and G band peaks which indicate the defect and graphitic structure of carbon structure respectively (Lou, Chen et al. 2006). The ratio of D and G bands (I_D/I_G) indicates the disorder of carbon structures. Table 2 shows the D and G peak positions and I_D/I_G ratio of carbon materials at different synthesis temperatures. A moderate I_D/I_G ratio (0.61) at 600°C of synthesis temperature indicate a uniform and low deffect of carbon structure although the a-C was produced. This was also confirmed by the FESEM images in Fig. 1(b). Material synthesized at 700°C has an I_D/I_G ratio of 0.67. Optimized carbon microspheres with diameter of were produced at 800°C synthesis temperature since it has a lower I_D/I_G ratio (0.79) than

carbon microspheres synthesized at 900°C. High I_D/I_G ratio (0.95) at 900°C synthesis temperature shows a lower degree of graphitization of carbon microspheres. The most defective structure, confirmed with FESEM images in Fig. 1(f) has the highest I_D/I_G ratio (0.96) was obtained at high synthesis temperature, 1000°C.

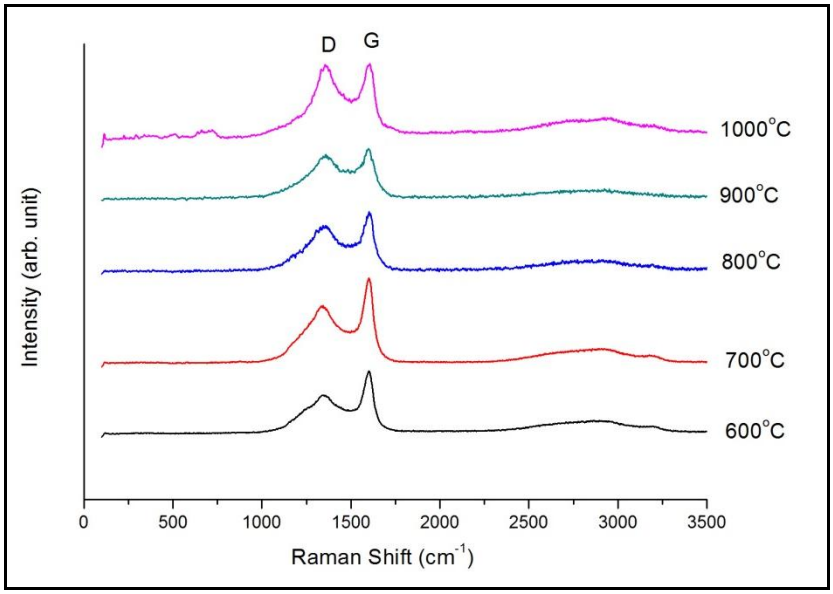

Fig. 2. Micro Raman spectrum of carbon materials synthesized at different temperatures

Table 2: Raman peak position and I_D/I_G ratio of carbon materials synthesized at different temperatures.

Temperature (°C)	**D-peak (cm^{-1})**	**G peak (cm^{-1})**	**I_D/I_G ratio**
600	1359.8	1597.3	0.61
700	1351.8	1596.8	0.67
800	1357.7	1598.2	0.79
900	1368.0	1597.5	0.95
1000	1370.2	1596.8	0.96

CONCLUSIONS

It can be concluded that synthesis temperature has a big role on the growth of carbon materials from WEO especially for their structures. Synthesis temperature also affects the average size of carbon materials. At temperature lower than 700°C, precursor was not completely decomposed and low catalytic activity caused the amorphous structure of carbon material formed. At 700°C, a material contained Cu and Al nanowires together with carbon microspheres were produced.

Well growth carbon microspheres were obtained at 800°C while at 900°C, bigger diameter and lower crystallinity of carbon microspheres were produced. At very high temperature, 1000°C, a high defect of carbon structure was formed.

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