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THE EFFECT OF OXIDATIVE HEAT TREATMENT ON THE PREPARATION OF STAINLESS STEEL 304 AND 316 AS THE EFFECTIVE CATALYTIC SUBSTRATE FOR CARBON NANOTUBE GROWTH

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ABSTRACT

Stainless Steel (SS) is the potential substrate in Carbon Nanotube (CNT) synthesis; Iron (Fe) and Nickel (Ni) content make SS function doubly as substrate and catalyst. In this study, SS is prepared with chloride acid, HCl (37.8%) and oxidative heat treatment (OHT) at 850°C for 30 minutes. This study aims to identify the effect of OHT on SS in CNT's formation. The identification is done by using carbon sources of acetylene and camphor. The substrate of SS 304 is varied into foil, plate and wire mesh. The result of using acetylene for 20 minutes in respect of the three variations produces carbon loss of over 90%. This is due to an increase in the Cr percentage which inhibits the formation of the catalyst's nanoparticles. With the help of ferrocene foil substrate, plate, and wire mesh, the CNT produced are 0.0573 gram, 0.0701 gram, and 0.1246 gram along with a reduction in carbon loss to 30%. The use of the substrate of SS 316 with lower Cr content and additional time of synthesis to 60 minutes yields the mass of 0.6325 gram and carbon loss of 2.76%. By using camphor for 60 minutes, the identification results in an increase of CNT mass in SS 304 of 0.831 for foil, 1.856 for plate and 2.6305 for wire mesh. Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDX) is used to identify the carbon form on the surface of the SS while Gas Chromatography Flame Ionization Detector (GC-FID) is used to identify the acetylene decomposition. Based on this experiment, SS 304 and 316 type along with the OHT preparation method can be used easily as an effective substrate to produce CNT.

Keywords: Acetylene; Camphor; Carbon Nanotube; Oxidative heat treatment; Stainless steel

1. INTRODUCTION

Back in 1991, Ijima discovered carbon with a tubular structure was discovered by which from then, until today has been, known as Carbon Nanotube (CNT). Ever since its discovery, the number of research studies on CNT has increased in relation to the method of synthesis, characteristic, and application. Commercial CNT applications include one of the components of nano-tech electronic equipment such as sensors, semiconductors, and fuel cell (Seah et al., 2011).

A quick process of adsorbing and desorbing hydrogen on CNTs is very useful for storing fuel hydrogen (Sudibandriyo et al., 2015). Recently, Stainless Steel (SS) has become widely used as a substrate for CNT growth in different forms like plate (Camilli et al., 2011) and wire mesh (Sano et al., 2012). In a previous study, substrates were used with ultrasonification

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preparation (Alves et al., 2012), and the heat treatment included oxidation and reduction steps (Sano et al., 2012) for better CNT alignment. SS contains Fe and Ni that can function both as substrate and catalyst. In order to function as a catalytic substrate, the SS' surface needs to be prepared to produce a suitable condition for the formation of CNT (Zhuo et al., 2014).

OHT is used as a preparation method combined with acid treatment for chromium reduction in SS 304 and 316. A recent study showed that both SS 304 and 316 could be used as a substrate for CNT. In this experiment, both substrates were used with the same preparation methods in order to identify their effects on the formation of CNT.

OHT is an oxidation preparation with the presence of oxygen used to increase the reactivity of the substrate and the catalytic area for the formation of CNT (Zhuo et al., 2014). Process is initiated by oxygen dissolution on the SS' surface until its saturation point at high temperature. Then, the oxygen is diffused to the alloy by the effect of the concentration gradient. Inside, oxygen reacts with the alloy, yielding an oxide substance that is precipitated on the surface (Wulan & Wijardono, 2017). The purpose of this treatment is to destroy slippery coating layers on the SS so that nanocarbon can grow more easily (Wulan & Cendana, 2016). Another advantage of using SS is that it is obtainable and available with various geometries such as plate, foil, and wiremesh. In addition, the use of SS as a substrate can facilitate the direct interaction between CNT and conductive substrate and increase the level of electrical conductivity.

This study was conducted in order to examine the effect of the OHT preparation method on SS 304 and 316 by using carbon sources of acetylene and camphor. The use of acetylene is based on its high reactivity and its effectiveness. Camphor is a renewable carbon source that can be used as a raw material for synthesizing CNT. Camphor is a substance that can be found on the Cinnamomum camphora tree. Therefore, with xylene as the dominant fraction, camphor is the potential alternative carbon source for the formation of CNT (Wulan & Silaen, 2017).

2. METHODS

In this section, we explain the theoretical basis that informed the present research study, CNT can be produced by using various methods like pyrolysis (Wulan & Wijardono, 2017), plasma enhanced chemical vapor deposition, PECVD, (Teo et al., 2003) chemical vapor deposition, CVD, (Baddour et al., 2008) and floating catalyst chemical vapour deposition, FC-CVD (Wulan and Silaen, 2017). In order to identify the effect of substrate preparation, CNT was produced by using chemical vapour deposition and floating catalyst as the most simply method (Seah et al., 2011). In this study, SS 304 and 316 was used as substrates for the growth of CNT. The SS preparation method included: ultrasonic process with acetone (95%) for 30 minutes etching with HCl (merck) 37.8% for 10 minutes and OHT at 850^{o}C for 30 minutes.

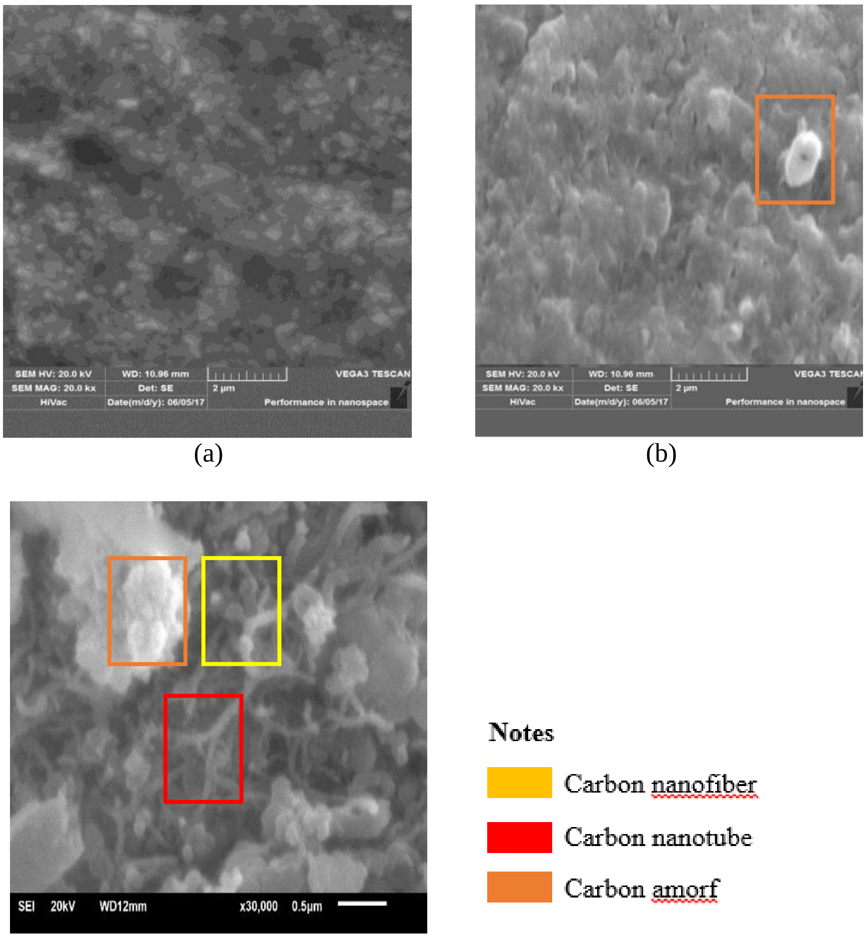
The synthesis process was carried out at 800^{o}C. The synthesis was undertaken by using acetylene with purity 99%, argon (99%) with the flow rate of 450 sccm, acetylene 40 sccm, and hydrogen (99%) 150 sccm, the synthesis lasted for 20 minutes and 60 minutes. The floating catalyst method was carried outby using 100 mg of ferrocene from sigma aldrich. Camphor synthesis was performed by using 10 grams of camphor for 60 minutes with the flow rate of 100 sccm for argon and 50 sccm for hydrogen. The substrate of the synthesis was weighed in order to determine the number of CNT formed which, then, were stored for the characterization process.

Characterization of SEM-EDX (Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy) was used to identify the morphology of the formed CNT. In addition, GC-FID (Gas Chromatography Flame Ionization Detector) was used to identify the composition of output gas of reactor in order to determine the percentage of carbon loss. The characterization, performed in this study, was SEM JEOL JSM-6510LA Analytical Scanning Electron Microscopy and GC-FID (Flame Ionization Detector) Agilent Technologies 6890N by using capillary dimethyl polisiloxane column.

3. RESULTS AND DISCUSSION

3.1. Identification of Substrate SS 304

The identification of substrate SS 304 in the formation of CNT was done by using acetylene for 20 minutes. Acetylene is the highest reactive hydrocarbon due to its bond structure. It can be used to produced CNT in a short time (Kimura et al., 2013) and, for the sake of comparison, the length of synthesis time using substrate 316 was extended to 60 minutes. The study was conducted by varying the three substrates, foil thickness of 0.025 mm (Masarapu & Wei, 2007), plate thickness of 1 mm (Baddour et al., 2008), and wire mesh (Wulan & Cendana, 2016) size of 300. The synthesis process was carried out by using the same size of the substrates; the purpose was to detect the influence of the substrate form in the formation of CNT when using the OHT preparation method.



(a) (b)

(c)

Figure 1 SEM result identification of SS 304: (a) foil; (b) plate; and (c) wire mesh using acetylene in 800°C for 20 minutes

Table 1 EDX result in SS 304 foil, plate and wiremesh in 800°C for 20 minutes

Components	Foil	Plate	Wire mesh
C (% w/w)	7.80	12.30	33.39
Cr (% w/w)	25.40	18.10	15.65
Fe (% w/w)	41.70	42.80	22.23

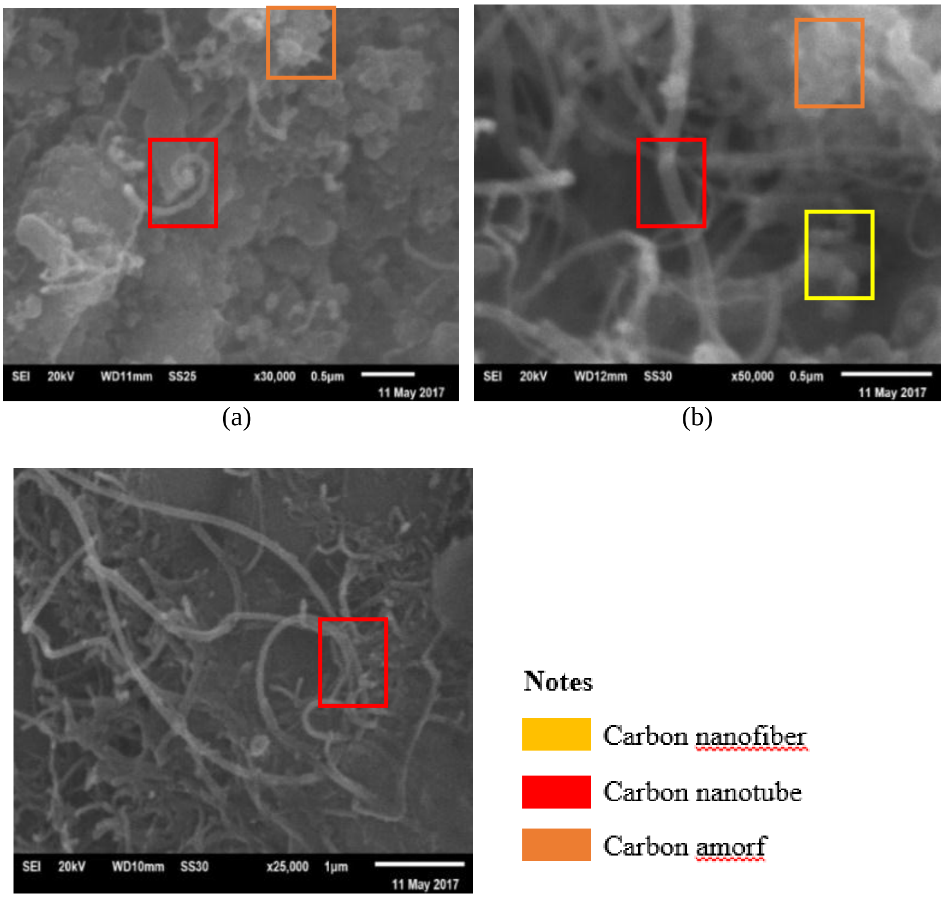
The result shows an increase of mass with an insignificant amount of 0.0006 gram (foil), 0.0009 gram (plate), and 0.0042 gram (wire mesh). Characterization of SEM-EDX was conducted in order to determine the presence of the carbon deposit on the surface of the substrate. Figure 1 shows the characterization result of SS 304 when using the carbon source of acetylene for 20 minutes. The Figure 1 result on SS foil after synthesis shows that, on the surface, only amorphous carbon was produced without the production of carbon filament. Figure 1b shows that the shape of the plate after the synthesis is unseen and that only a clump of catalyst is present without amorphous carbon and carbon filament.

Based on the EDX result in Table 1, the plate substrate has a higher percentage of C than foil. Therefore, the carbon deposit is not characterized in SEM due to errors during handling which caused the carbon to escape from the surface of the plate. In Table 1, the Foil substrate in the EDX result shows the highest percentage of Cr amounts to 22.90 %; when comparing the percentage before synthesis, then, the amount of Cr declines by 4.00%. The absence of CNT on the foil substrate is due to Cr oxide coating the surface of SS so that the nanoparticles of Fe and Ni present in SS cannot be formed; such a case is in accordance with the findings of Sabioni et al's (2005) study.

In Table 1, the change on the amount of Cr and Fe on plate substrate is not very significant. Hence, the non CNT-formation on the plate was not due to the diffusion of the Cr oxide layer with a review of the heat transfer within the reactor. Based on the calculation and by reviewing the layer of plate substrate without direct touch towards the reactor, this causes a reduction in the amount of energy received by substrate of 0.3254 kJ/s. Based on Horvath (2006) findings, the calculation of heat transfer is evaluated by using a tubular shape factor for the isothermal reactor formulas. Thus, based on the calculation, it shows that the CNT on the plate is not formed due to the energy, received by the substrate, being insufficient to produce CNT. As shown in Figure 1c, the result of SEM characterization of wire mesh substrate after synthesis shows the formation of carbon filament on the surface of the substrate. The formed Carbon filament consists of CNT and Carbon Nano Fiber (CNF), in which CNT was formed in small amounts and dominated more by the formation of CNF. CNF is a form of carbon filament with the diameter in the dimension of micron. The identification of the reactor output gas was carried out by using GC-FID. The result of the identification of reactor output gas relates to the 20th minute of each substrate of SS 304. The data relating to the composition of the reactor was processed further in order to calculate the conversion and carbon loss which occurred during the synthesis. The amount of carbon loss was evaluated by calculating the mass balance of carbon atoms at 99.69% for foil, 99.57% for plate and 97.95% for wire mesh. The use of SS substrate without an optimum activation led to a high amount of carbon loss during the synthesis period. Therefore, in order to increase the mass of the produced CNT, the synthesis of SS 304 was performed by using the method of FC-CVD, and ferrocene was used as an additional catalyst.

3.2. Identification of SS 304 using FC-CVD

By using ferrocene on SS 304, the synthesis result was: foil produced increased mass of 0.0573 gram, plate 0.0701 gram, and wire mesh produces 0.1246 gram. High mass of wire mesh substrate occurred due to the shape of wire mesh with widest surface area when compared with the other substrate; this was 155.6348 cm^2. Floating method towards foil substrate still had the highest Cr content. Figures 2a and 2b show the SEM results when using foil substrate and plate. Compared to the previous studies, CNT can grow within foil substrate and plate because the catalyst is obtained from ferrocene decomposition. In Figure 2a, the SEM result on foil substrate shows uneven growth and an increase in the percentage of the least mass of carbon mass due to the high amount of Cr on foil substrate in order to prevent the diffusion of C on the surface of the substrate. The result of SEM characterization on foil shows that the carbon deposit on the surface of the foil substrate is dominated by impurity carbon and vapor growth CNF. Figure 2a shows that the CNT results in foil based on the type of rod buckling. Rod buckling occurs within CNT with a notably high ratio and causes deformation of CNT tubular structure (Buehler et al., 2004). Figure 2b shows the rod buckling CNT formed on the plate substrate. The use of the plate substrate produces CNF which has a much larger diameter than CNT carbide. The SEM result is based on the presence of the catalyst in the body of CNT and by reviewing the catalyst position on CNT which shows the growth mechanism of the type of growth according to the statement (Daenen et al., 2003). Growth tips are due to the weak interaction between the substrate and the catalyst and, therefore, the catalyst is lifted upwards when CNT is formed. The CNT result on the plate substrate shows the effect of buckling.



(a) (b)

(c)

Figure 2 SEM result identification of SS 304: (a) foil; (b) plate; (c) wire mesh using FC-CVD in 800°C for 20 minutes

Table 2 EDX result of SS 304 synthesis using FC-CVD in 800^{o}C for 20 minutes

Components	Foil	Plate	Wire mesh
C (% w/w)	26.38	36.03	42.71
Cr (% w/w)	13.10	12.17	5.29
Fe (% w/w)	39.87	30.38	28.48

Figure 2c shows the SEM result by using the wire mesh substrate. The use of wire mesh substrate in the floating catalyst method produces a longer CNT than the one without using ferrocene. Moreover, the characterization result shows the catalyst content on fewer bodies of CNT when compared to the shape of the plate substrate. The SEM result shows that the CNT growth mechanism occurs, also, in the manner of tips growth. Tips growth mechanism can produce continuous growth, resulting in longer CNT. The catalyst is lifted from the substrate causes CNT growth in order to avoid the difficulty of carbon diffusion and leads to the termination of CNT growth resulting in short CNT. CNT, formed on wire mesh substrate, has a high aspect ratio and direct self-folding on CNT. Based on the result of Buehler et al. (2004) study, (self-folding is a buckling phenomenon that occurs in CNT with a high aspect ratio. Self-folding occurs because of the interaction of Van Der Waals in which CNT binds to the other end and causes CNT to be agglomerated. Impurities of amorphous carbon are formed due to the characteristics of the acetylene source. The result of Kimura et al.’s (2013) study shows that the use of acetylene can produce higher amorphous carbon than saturated chain hydrocarbons.

Table 2 EDX result shows that the use of ferrocene causes a reduction in the percentage of Cr and Fe and an increase in the percentage of C. The result of the use of ferrocene increases the percentage of C more significantly than the one without the use of ferrocene. The use of ferrocene as an additional catalyst increases the carbon deposit on foil plate by 0.0567 gram; the deposit on the plate increases by 0.0692 gram; and the deposit in wire mesh increases by 0.1204 gram. It is shown that the use of ferrocene as an additional catalyst produces a reduced amount of carbon loss when compared to the one without the use of ferrocene. The use of foil substrate on floating catalyst results in a high carbon loss of 72.5% and, therefore, there is no potential to use foil. The use of wire mesh results in a carbon loss of 39.18% with better CNT morphology when compared to foil and plate. The use of plate substrate on floating catalyst results in a carbon loss of 66.52%. It shows that CNT has not been fully formed on plate substrate. The manner, which can be used in order to increase CNT mass, is by increasing the reaction time. Based on previous research, there is no potential to use foil substrate 304 in the formation of CNT due to the high Cr content.

Table 3 Effect of preparation method in SS plate 304 and 316

Components	SS 304	SS 316
Fe (% w/w)	46.80	56.50
Cr (% w/w)	19.60	9.80
Ni (% w/w)	3.70	4.30
O (% w/w)	8.50	27.10
Mn (% w/w)	2.60	2.10
Mo (% w/w)	-	0.10

Optimization needs to be performed by using SS with a lower percentage of Cr, i.e. SS 316. Table 3 shows the comparative influence of the OHT preparation method on SS 304 and 316. SS 304 substrate reduces the percentage of Fe and increases the amount of Cr. While, for SS 316, the amount of Fe reduces only slightly but there is a significant reduction in the Cr percentage. It indicates that only a small amount of CNT was obtained by using the etching method of HCl and OHT. Therefore, in order to increase the mass of CNT, this study increased the reaction time to 60 minutes and used substrate 316.

3.3. Identification of Substrate SS 316

The addition of synthesis time was carried out in order to increase the mass of the formed CNT. The result of using carbon source of acetylene shows that SS 316 is capable of acting as a catalytic substrate for CNT growth with a mass of 0.6325 gram. Increasing the mass of CNT happens due to low percentage of Cr content and, according to Mendoza et al. (2005), the addition of Molybdenum in 316 leads to increasing catalyst activity and CNT yield. The result of GC-FID after 60 minutes shows that the gas remains 100% methane. Methane can be used as a source of carbon in the formation of CNT due to the decomposition reaction (Wulan et al., 2015). The use of substrate 316 reduces the amount of carbon loss from 99.57% (in 304 plate) to 2.76% (in 316 plate). However, as shown in Figure 3, the reduction of carbon loss in plate 316 is not followed by higher quality of CNT.

Figure 3 shows the morphology of the use of SS 316 with a carbon source of acetylene for 60 minutes. It is seen that the surface of the substrate is dominated by carbon impurity and CNF. The resulting CNT is not very long and the catalyst is formed at the end of the CNT. The catalyst position signifies the tips growth mechanism. Seeing that impurity carbon dominates the surface of the substrate, it indicates that CNT has reached a termination stage so that it is unable to undergo further growth. The SEM result shows that the resulting CNT diameter is ± 40-60 nm. The flow rate used is 0.00183 moles of acetylene/min. Based on Kimura et al.'s (2013) curve of carbon source characteristics, the used carbon concentration exceeds the optimum point of acetylene carbon concentration. The use of excess acetylene hydrocarbon concentration leads to a low yield.

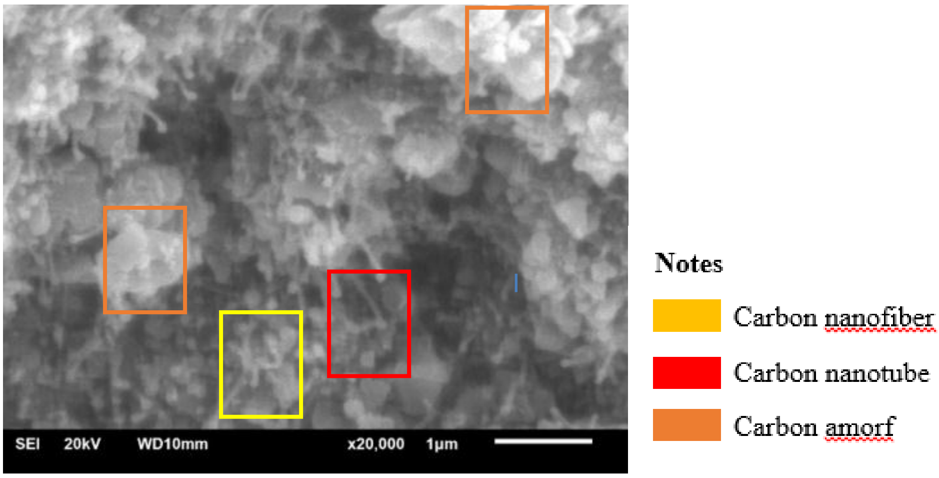
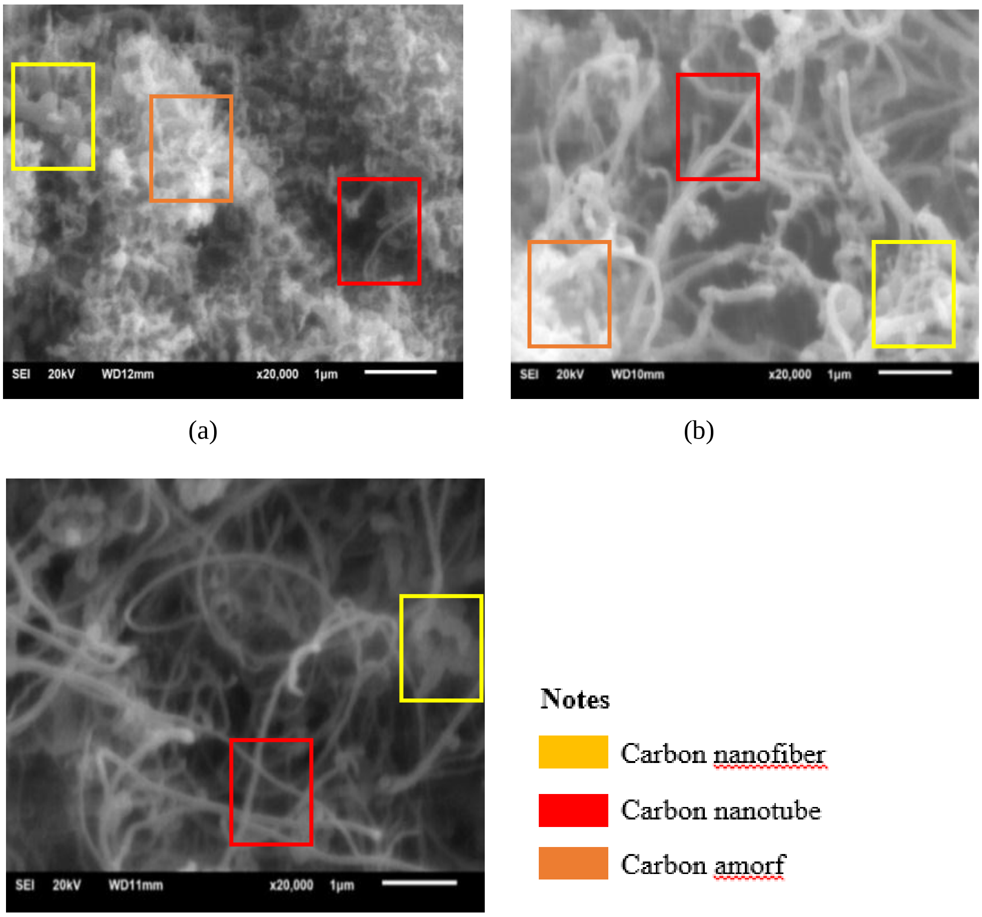


Figure 3 SEM result identification of stainless steel 316 plate using Acetylene in 800ºC for 60 minutes

3.4. Identification the Effect of Camphor Carbon Source

The result of Horvath et al.'s (2006) study suggests that, when compared with benzene, xylene is a more effective carbon source of increasing the CNT yield. In addition, as shown in Kumar and Ando's (2003) findings, the presence of oxygen in the camphor cluster acts, also as an oxidizing agent to reduce the amorphous carbon formed in situ. In addition, Kimura et al.'s (2013) findings show that the xylene characteristic is inversely proportional towards acetylene in that the continuous growth of carbon concentration in xylene increases the yield of the resulting CNT. Therefore, further study was done by using a camphor carbon source. When 10 gram of camphor as a carbon source is used for either foil, plate, or wire mesh, it is able to produce CNT without the aid of an additional catalyst.

The result of characterization using SEM shows, also, that the best CNT morphology is when substrate of SS 304 wire mesh is used. Based on previous research, the use of foil and plate substrates with acetylene are unable to produce CNT without ferrocene as the catalyst. With the same size of foil substrate, the use of camphor as the carbon source can produce CNT with the mass of 0.1662 gram. Figure 4a shows that when the foil substrate is used, the surface of the substrate is dominated by CNF which is agglomerated and covered by impurity carbon.



(a) (b)

(c)

Figure 4 SEM result identification of stainless steel 304: (a) foil; (b) plate; and (c) wiremesh using Camphor in 800ºC for 60 minutes

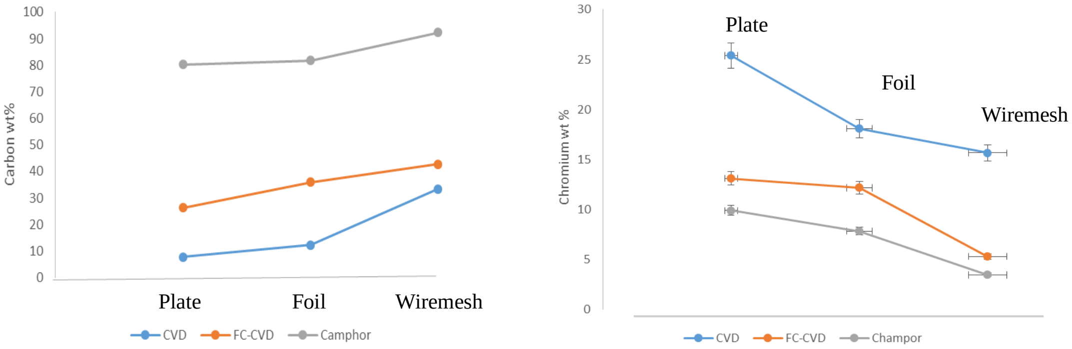
The use of plate substrate with camphor is able to produce CNT with a mass of 0.3712 gram. Figure 4b shows the CNT formed on the plate substrate. Based on the presence of catalyst on CNT, it shows the tips growth mechanism. CNT formed is relatively long and bonded to each other. The interaction between CNT is caused by the Van Der Waals force of each CNT so that it results in CNT undergoing buckling process and being agglomerated. CNT agglomeration causes CNT structure damage which reduces the CNT properties. As shown in Figure 4c, the SEM result of the CNT formed on a wire mesh substrate has the best morphology with catalyst content around the CNT being less than the ones using foil and plate. In addition, the CNF is small and does not dominate. The buckling phenomenon occurs, also, in the CNT of wire mesh substrate. CNT, with a high-aspect ratio, has characteristics which are similar to the structure of wire and is able to bind to each other due to the Van Der Waals force of each CNT.

Table 4 EDX Result of CNT using SS 304 and Camphor within 60 minutes

Components	Foil	Plate	Wire mesh
C (% w/w)	80.24	81.88	92.31
Cr (% w/w)	9.89	7.83	3.46
Fe (% w/w)	3.21	0.80	0.88

Table 4 shows the result of EDX regarding the substrates of foil, plate and wire mesh. Consequently, the substrate of wire mesh has the highest percentage of C with a smaller percentage of catalyst than the substrate of foil and wire mesh. The percentage of carbon is directly proportional towards the produced CNT yield and, therefore, the use of Camphor as a carbon source has been indicated to increase the CNT yield.

Figure 5 shows the relationship of the EDX analysis result in this experiment. Figure 5a shows the carbon percentage result in CVD, FC-CVD and Camphor when using substrate SS 304 plate; foil; and wire mesh. The use of substrates in CVD with OHT resulted in the lowest carbon percentage, then followed by FC-CVD, and the use of Camphor resulted in the highest carbon percentage. Based on substrate forms and when compared to plate and foil, wire mesh resulted in the highest carbon percentage. As can be seen in Figure 5, the increasing carbon percentage results in the chromium percentage declining gradually.



(a) (b)

Figure 5 EDX Analysis result: (a) carbon percentage; (b) chromium percentage in CVD, FC-CVD, and Camphor

Figure 6 shows the effect of OHT on substrates 304 and 316 with an extended synthesis time. Both substrates can be used as substrates for CNT synthesis. The use of substrate 316 with extended synthesis time led to the carbon percentage increasing from 12.3% to 40.18%.

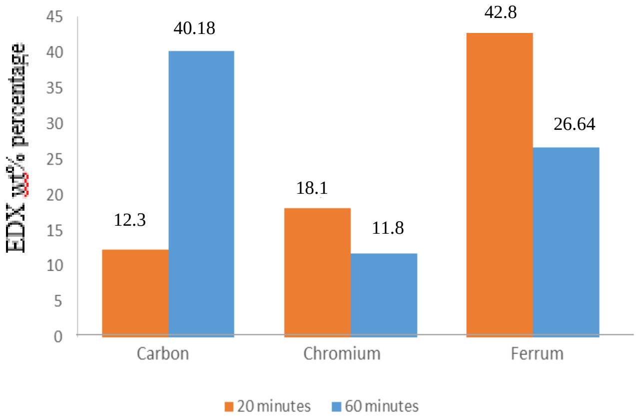


Figure 6 EDX analysis of substrates 304 and 316 with extended time synthesis

The increased mass of CNT production can be achieved by following steps such as: using SS 316 which contains a lower Cr percentage; extended synthesis time; and using a carbon source which has an oxygen bond such as Camphor.

4. CONCLUSION

The results show an OHT loss of over 90% with an increase in the percentage of chromium mass. This was achieved with the help of ferrocene CNT produced with the mass of 0.0573 gram (foil), 0.0701 gram (plate), and 0.1246 gram (wire mesh) along with the reduction in carbon loss of for more than 30%. The use of SS 316 with lower Cr content and additional times of synthesis up to 60 minutes yielded a mass of 0.6325 gram and carbon loss of 2.76%. The use of Camphor as an alternative carbon source resulted within 60 minutes in an increase of CNT mass in SS 304 (0.831 gram for foil, 1.856 gram for plate, and 2.6305 gram for wire mesh). Xylene and oxygen played a significant role in reducing impurity and increasing the mass of CNT. This was because they served as an oxidizer to ensure that the catalytic substrate remained active. Consequently, CNT had better quality when produced by using Camphor as a source of carbon.

5. ACKNOWLEDGEMENT

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