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Effect of Temperature on the Growth Rate of *Carbon Nanotubes* (CNTs) using Electrodeposition Method

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ABSTRACT

Electrodeposition is one of the carbon capture methods used to produce carbon nanotubes with temperature as one of the variable. The research aims to analyze the effect of temperature on the growth rate of carbon nanotubes of 723ºC, 750ºC, 800ºC, 850ºC and 900ºC using X-Ray Diffraction (XRD) testing. Analyzing deposit morphology at the same temperature using Scanning Electron Microscope (SEM) testing. The results of research show that the most optimal growth rate for carbon nanotubes occurred at 750ºC of 7,949 g cm^{-2} $hours^{-1}$. At a temperature of 750ºC, carbon deposits are easier than at 723ºC because that's the melting point of lithium carbonate and has not completely decomposed. The XRD test show that at 750ºC is the highest peak at $2\theta = 26.21^{o}$. The SEM test show that the optimal morphological structure formed occurs at a temperature variation of 750ºC with a fibrous morphology and little impurity at the ends. The results of the CNT percentage using the Material Analysis Using Diffraction (MAUD) method show that the largest quantitative value of the CNT percentage occurs at a temperature of 800ºC of 4.08%.

Keywords: *Electrodeposition, carbon nanotube, temperature, growth Rate*

1. Introduction

High demands for energy and materials have undoubtedly resulted in side effects, the main of which is an increase in the amount of carbon dioxide (CO_2) released and having an adverse effect on atmospheric conditions. There are various approaches to overcome the increase in CO_2 emissions [1]. CO_2 emissions in the power generation sector amounted to 38.80%, transportation amounted to 26.56%, industry amounted to 24.76%, buildings and agriculture amounted to 8.07% and 1.81% [2]. One approach to mitigating CO_2 emissions involves carbon capture and storage (CCS) or carbon capture and conversion (CCC) [1], [3]. The method used for carbon capture and storage (CCS) or conversion (CCC) is electrodeposition. CCS typically involves capturing CO_2 from sources like power plants and storing it underground, while CCC converts CO_2 into useful products. Both methods can be complemented by electrodeposition, a technique that has been utilized for carbon capture and more recently for producing carbon nanotubes (CNTs) [1], [3], [4], [5].

The electrodeposition method has several variables that must be considered to obtain a high growth rate of carbon nanotubes and good carbon nanotubes results [6]. These variables are time, cabon source, external potential, electrode and temperature. These variables have different effects from time to time, in general the longer the electrodeposition time, the more carbon deposits will be formed. However, the optimal carbon growth rate needs to be considered [4]. The carbon source in the electrodeposition process functions as a carbon supplier. Research conducted by Koya Otake in 2014 stated that by increasing the flow rate of carbon sources, the results of carbon deposits also increase. The external potential in the electrodeposition method functions as a way to control CNT growth, carried out in two ways during electrolysis, namely with a constant external potential and a constant current. In general, to produce carbon deposits, the external potential and current used range between 3-6 V [7]. Electrodes are used as a place or container for carbon deposits in the electrodeposition process. Research from Douglas using a Ni electrode produces CNTs, corrosion of the anode to the electrolyte causes a coating on the cathode and becomes a nucleation point for carbon structures through the cathode [8]. The temperature in the electrodeposition process functions to dissolve the electrolyte used. High and low temperatures also influence the reaction speed in the electrodeposition process which also influences the carbon deposit process [1]. An indication of the influence of temperature on the electrodeposition process is that the greater the temperature, the size of the carbon particles will increase [7]. At high temperatures, the coating rate increases, and the corrosion rate decreases. Coating efficiency also increases at higher temperatures.

2. Methodology of Research

The materials used in this research are as follows.

Table 1. Materials in research

Electrode	Nikel
Electrolite	*Lithium Carbonate*
Carbon Source	CO_2

The method used in this research is the electrodeposition method. This research aims to analyze the effect of temperature on the growth rate of carbon nanotubes. The variables used in this research used temperatures of 723ºC, 750ºC, 800ºC, 850ºC, and 900ºC. The voltage used is 5 V, the CO_2 flow rate is 180 ml/minute with an electrodeposition process time of 60 minutes. The result of the electrodeposition process is a deposit that sticks to the electrode. The growth rate of carbon nanotubes can be calculated using the following

$$\text{CNT} = \frac{\text{Total mass of deposit x wt\% CNT}}{\text{Deposition area x time}}$$

Phase observations in the material were carried out using the XRD test. The data produced from the XRD test equipment is in the form of graphs. The material tested is the result of deposits from the electrodeposition process. The test uses XRD to determine the phase formed from the deposit resulting from the electrodeposition process. XRD graphs are analyzed peak identification

Morphological observations on the material were carried out using the SEM test. Data generated from the SEM test equipment to determine the morphology formed from deposits in the electrodeposition process. SEM criteria are used to interpret SEM images as morphology form of CNT.

3. Result and Discussion

Based on the results of calculating the growth rate of carbon nanotubes in Table 2, a temperature of 723ºC produces a growth rate of carbon nanotubes of 0.999 g cm^{-2} $hours^{-1}$. At a temperature of 750ºC, the growth rate of carbon nanotubes is 7,949 $g.cm^{-2}hours^{-1}$. Meanwhile, at a temperature of 800ºC the growth rate of carbon nanotubes is 4,414 $g.cm^{-2}.hours^{-1}$, for a temperature of 850ºC the growth rate of carbon nanotubes is 4,269 $g.cm^{-2}.hours^{-1}$ and the growth rate of carbon nanotubes at a temperature of 900ºC is 0.744 $g.cm^{-2}.hours^{-1}$ at 1 o'clock. The growth rate of carbon nanotubes obtained from deposits using the electrodeposition method was obtained based on the total mass of the deposit, wt% of CNT, the area of the deposit on the electrode and the time of the electrodeposition process.

Table 2. Growth rate from calculation results

Temperature (ºC)	Total mass of deposit	CNT (wt%)	Growth rate (g/cm^2h)
723	3.34	0.47	0.999
750	4.32	2.89	7.949
800	1.7	4.07	4.414
850	2.6	2.58	4.269
900	0.88	1.32	0.744

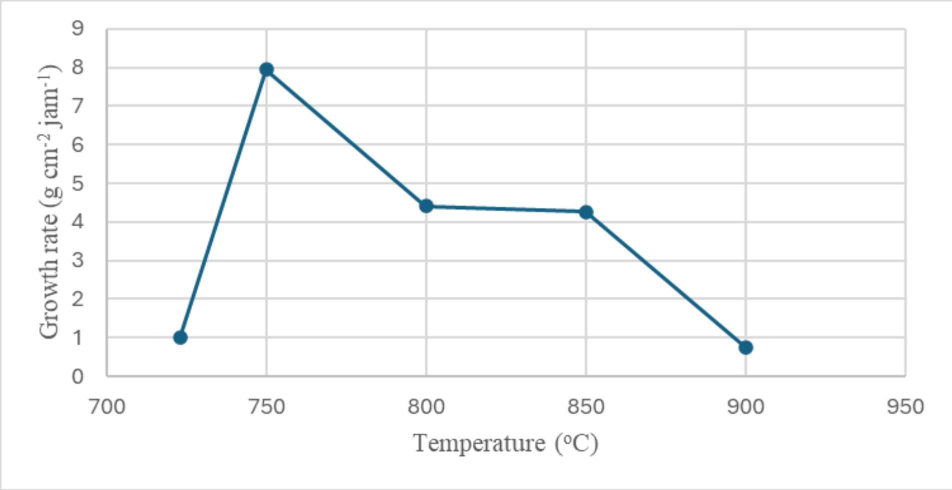

Figure 1. Graph of growth rate results

Based on current efficiency calculations, current efficiency has two variables, namely experimental results and calculation results. An increase in current efficiency means that the experimental results will increase, while the calculation results will decrease [9]. For example, at a temperature of 750°C carbon deposits are easier than at 723°C because the temperature of 723°C is the melting point of lithium carbonate [10]. This can be proven at a temperature of 723°C, the lithium carbonate powder has not decomposed completely even though the lithium carbonate powder has melted [11], [12].

XRD analysis is generally used to measure the average distance between layers and atomic arrangements, determine the orientation of single crystals or grains, determine the crystal structure of a material, and to measure the size, shape and internal stress of crystalline areas [13], [14]. The XRD results are shown in Figure 2.

Based on Figure 2, it can be seen that the XRD graph shows differences in peak height at a temperature variation of 723°C with other temperature variations. At the temperature variation of 723 °C the highest peak is at = and this peak shows the Li_2CO_3 phase [15]. This is because at a temperature of 723 oC the crystal structure of Li2CO3 has not completely decomposed because at this temperature it is the melting point of Li_2CO_3 [10]. Meanwhile, at temperature variations of 750°C 800°C, 850 °C, and 900°C the highest peak is at, where the peak indicates the C-C phase [16].

The morphology of the deposit resulting from the electrodeposition process was observed using SEM. The morphological differences between temperature variations in the electrodeposition process are shown in Figure 3. Based on the SEM test results, it can be observed that at temperature variations of 723°C, 750°C, 800°C, and 850°C the diameter sizes vary from 50-100 nm. At a temperature of 723°C the morphology formed is like fibers. The temperature of 723°C also forms fibers with a little impurity at the ends. Meanwhile, at a temperature of 800°C, the morphology formed is also stringy with slightly clumpy compared to the previous temperature. At a temperature of 850°C the morphology formed is slightly straight with a little impurity at the ends. This is caused by the nucleation of carbon during the electrolysis process which increases [17]. At a temperature of 900°C the morphology is round and has a larger diameter than at lower temperatures. This is because with high temperatures the size of the deposited particles will also increase [7].

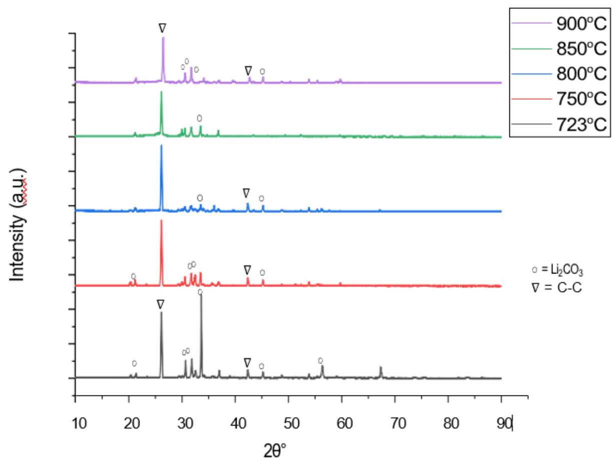

Figure 2. XRD results

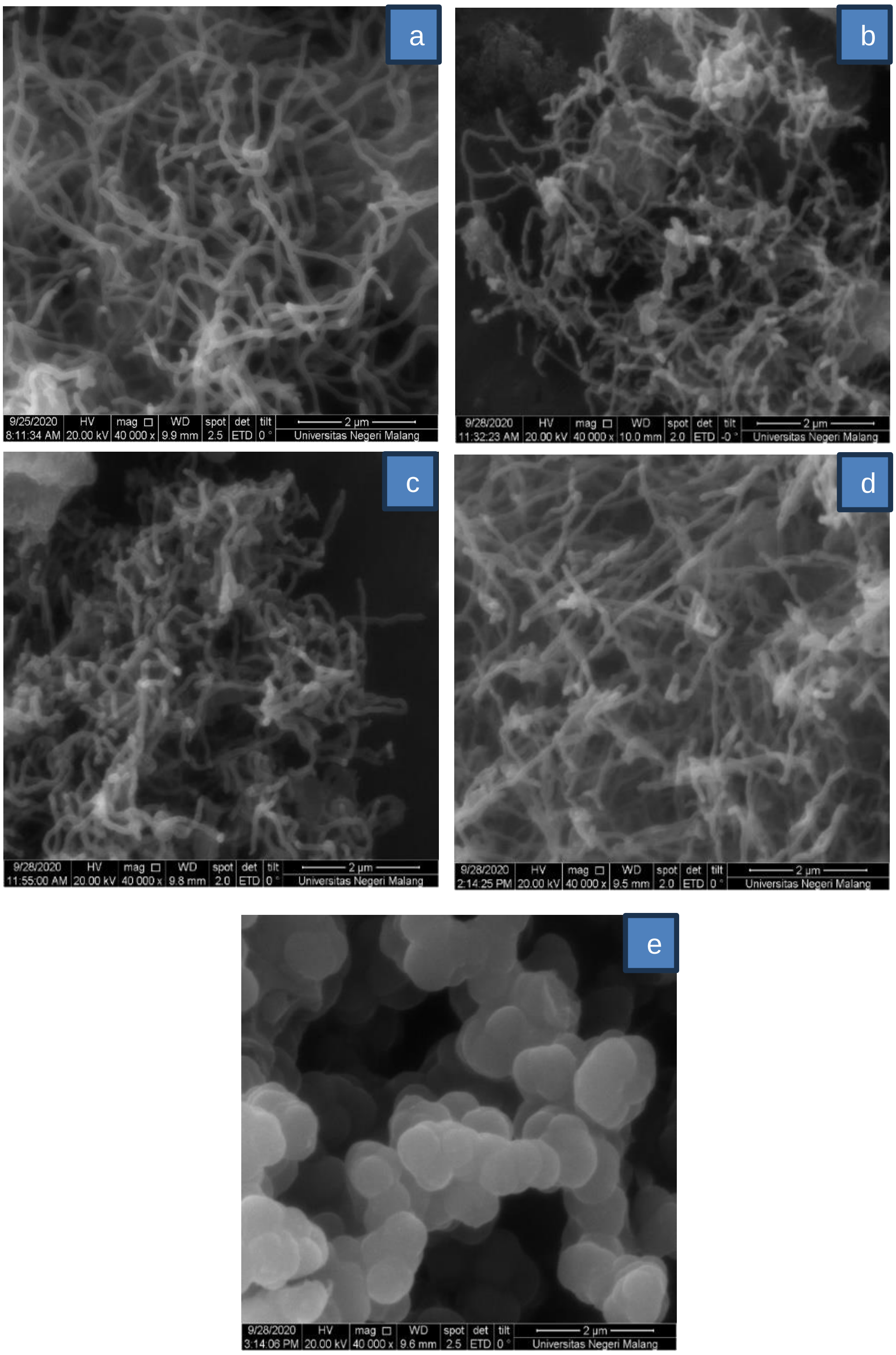

Figure 3. SEM testing results for temperature variation (a) 723ºC (b) 750ºC (c) 800ºC (d) 850ºC (e)

Based on SEM results show that at 723ºC to 850ºC morphology of CNT formed are fibrous with litle impuritis. At highest temperature 900ºC morphology be round lumps. Higher temperatures speed up the rate of chemical reactions on the electrode surface. This can lead to faster growth of films or granular nanotube structures. At lower temperatures, ion diffusion slows down. This can cause slower deposit rates and increase the possibility of uneven or grainy structures forming. Temperatures that are too low can also cause a decrease in the reaction rate, which can result in inconsistent growth of nanotubes that can form fibrous [18], [19].

4. Conclusion

Based on the research that has been carried out, conclusions can be drawn regarding the influence of temperature on the growth rate of carbon nanotubes in the electrodeposition method.

1. The highest growth rate value for carbon

nanotubes occurred at a temperature of 750ºC, amounting to 7,949 g cm^{-2} $hours^{-1}$, while the lowest growth rate value occurred at a temperature of 900ºC, amounting to 0.744 g cm^{-2} $hours^{-1}$

2. The results of the XRD test show that at temperature variations of 723ºC, 750ºC, 800ºC, 850ºC and 900ºC the highest peak is at 2θ= 26.21^0 for temperature variations of 723ºC, the highest peak is at 2θ =33.66^0.

3. The results of SEM testing show that at varying temperatures of 723ºC, 750ºC, 800ºC, and 850ºC the morphology formed is fibrous with little impurities and has a diameter that varies between 50-100 nm. Meanwhile, for the temperature variation of 900ºC, the morphology formed round lumps.

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