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Effect of Residence Time and Chemical Activation
on Pyrolysis Product from Tires WasteAndi Erwin Eka PUTRA^{*1†}, Novriany AMALIAH^{*1}, Machmud SYAM^{*1}, and Ismail RAHIM^{*2}

(Received March 14, 2019)

In this study, active carbon produced from tires waste using pyrolysis was investigated. As tires were made predominantly from the petroleum product rubber, they have a high heating value, as well as high volatile content and medium sulfur content. Those properties make them excellent candidates for pyrolysis, which can be used to recover energy and by-products. Pyrolysis has been conducted until the temperature of 750 °C and holds in residence time variation of 5, 60, and 120 min after oil and gas completely produced. Char was produced via pyrolysis then activated by chemical activation using NaCl solution for 24 hours followed by drying at the temperature of 100 °C for 1 hour. Activated carbon as pyrolysis product then analyzed using Scanning Electron Microscope, BET analysis and N₂ adsorption analysis. It was observed that the pore size of the resulting carbon generally increases as increasing of residence time. In addition, increasing residence time also resulted in enhanced higher porosity development after chemical activation using NaCl as high as 2.19 nm with BET surface area of 28.19 m²/g. The formation of pores with an average diameter of pore 8 μm was observed on the carbon surface.

Key Words

Pyrolysis, Activated carbon, Tires waste

1. Introduction

Recent developments in the world transportation systems have led to a renewed interest in a major environmental issue. The disposal of tires waste especially in developing countries becomes a primary concern throughout the world. About 2.7 billion units of tires were produced in 2017 and 1 billion units was disposed of¹⁾. This organic solid waste cannot be degraded by a biological process so the one common way of disposal is landfilling. Due to the volume of tires that cannot be compacted, this way is associated to sufficient amount of space and also can become ideal breeding grounds for disease-carrying mosquito as a result of rainwater deposited in the free space of tire wall. Also, landfilling is a potential danger because of the possibility of unintentional fires with high levels of harmful gas emissions²⁾. Another alternative for tires waste

disposal such as retreading, reclaiming, grinding, etc. also have been developing recently.

The waste tire provides products as the oil, releasing gas, char, steel, and residual products. All these products have the potential for reuse. A number of researchers have reported some products produced from tires waste using various methods. Gasification of tires waste using conventional gasification, plasma gasification, and solar-assisted gasification can produce syngas such as methane and hydrogen that can be utilized for fuel cells, gas turbine and gas engine³⁾. Microwave electromagnetic energy can produce a polymer modifier using waste tire rubber by regeneration phenomenon i.e. the combined devulcanization and degradation of the rubber material⁴⁾. Tire waste converted into gasoline, diesel-like fuels, gas, and char using catalytic pyrolysis method⁵⁾. Tire wastes represent an interesting source of raw material due to highly mesoporous activated carbon that can be produced. The activated carbon can be applied to a pollution-cleaning adsorbent and become an effective solution for environmental pollution⁶⁾.

Recently, researchers have shown an increased interest to pyrolysis method of waste tires, since the

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process can be optimized to produce high-quality fuels as products and also one of the most reasonable alternatives in terms of environmental protection. Pyrolysis of tire waste produced oil with main components was naphthalene and anthracene, char, and also fuel gas⁶. Pyrolysis of a solid motorcycle tire waste was determined to produce liquid fuels and chemical. The maximum liquid yield of 49 wt% was obtained at temperature 475 °C with a residence time of 5 s under N₂ atmosphere in a fixed-tube heating reactor system². Activated carbons were produced from tires waste using a chemical activation method impregnated with KOH. It is recommended that a KOH/ratio of 4, a pyrolysis temperature of 700 °C, and a zero holding time are the optimum values of the parameters for producing high surface area carbons⁷. The pyrolysis of tire powder was studied experimentally and the pyrolysis rate increases with increasing temperature in the range of 500-1000 °C. The yield of a gaseous product increases from 5% to 23% (wt) within the temperature range studied with maximum value reached at 900 °C⁸. Among the numerous chemical reagents that can be used to create porosity in carbon, sodium chloride (NaCl) was considered as activator due to its low cost, nontoxic, and environmentally friendly. The previous study of biochar produced from food waste has shown that char yield, pore volume, and average pore diameter increases as the impregnated NaCl content increase⁹.

In this paper, active carbons produced from tires waste in pyrolysis were the main concern. The influence of residence time and chemical activation using NaCl to the porosity development was extensively explored.

2. Experimental

The experimental apparatus of pyrolysis of tire waste is shown in Fig. 1. A motorcycle tires waste were cut into small pieces approximately 3 mm and a total weight of 25 g. The sample was put in the reactor then heated using a

stove. In this process, pyrolysis was carried out by heating the samples at 20 °C/min until the temperature of 750 °C. The pyrolysis of tire waste generated products such as gas, oil, and char. Gas and oil were started to produce at the temperature approximately 40 °C and 410 °C respectively. The residence time was varied after temperature reaches 750 °C.

Pipe was used to distribute and separate oil and gas product of pyrolysis. Water displacement method was conducted to gather gas product. After oil and gas are exhausted, temperature of 750 °C was maintained at residence time of 5, 60, and 120 min. The char produced after pyrolysis was activated by immersed in 35% NaCl solution for 24 hours followed by heating at the temperature of 100 °C for 1 hour. After the activation process, the weight of active carbon was then measured and a Scanning Electron Microscope (SEM) was used to study the surface feature of active carbon produced. The thermal properties characterization was conducted using Thermogravimetric Analysis (TGA) method at the temperature of 600 °C and a gas flow rate of 10 °C/min. Specific surface area, pore volume and pore diameter of activated carbon determined by N₂ adsorption analysis and Brunauer-Emmet-Teller (BET) method (Quantachrome TouchWin v1.2).

3. Results and Discussion

3.1 Thermogravimetric analysis

The thermal stability for the tire waste pyrolysis is shown by the TG and DTG profile in Fig. 2. TG and DTG analyses were conducted in the N₂ gas atmosphere at the temperature of 30-600 °C. TG profile shows the mass percentage reduction versus temperature while DTG profile determines the reduction of mass per time versus temperature. The TG profile reveals tire waste sample lost about 65.8% of mass before it reached 700 °C through three decomposition stages. The first stage occurred at the temperature of 200 °C with a mass decrease of 0.9%

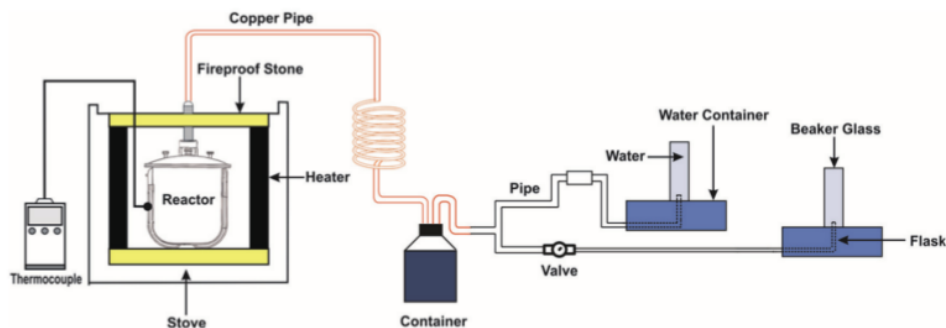


Fig. 1 Experimental design of tire waste pyrolysis

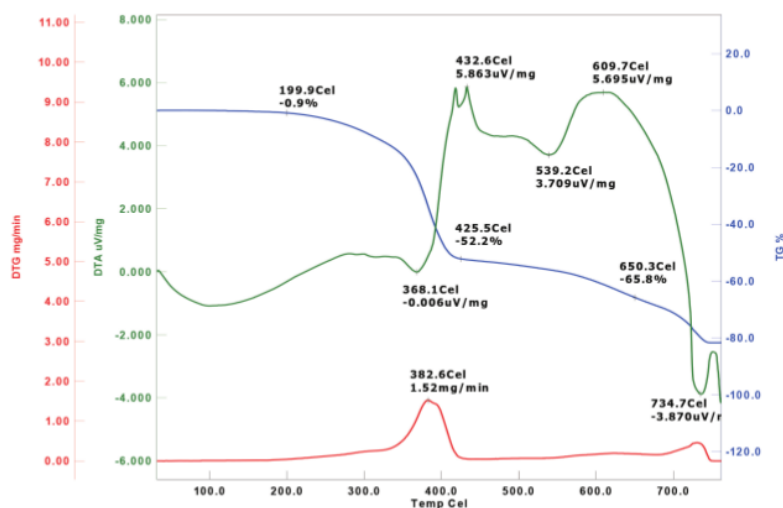


Fig. 2 TG and DTG profile for tire waste pyrolysis

through the evaporation of moisture content. The pyrolysis effect on the sample showed the highest mass loss of 52.2% in the second stage at the temperature of 400-450 °C. This range of temperature of tire waste degradation was in a good agreement with the literature^{10) 11)}. The highest decomposition rate on the DTG profile was 1.52 mg/min as observed at about 382.6 °C indicating a broad peak. This is compatible with what was observed at the second stage of the TG profile so it can be explained that the temperature of 382.6 °C is the thermal resistance temperature for the tire waste.

3.2 Effect of residence time

The carbon yield produced from pyrolysis of tire waste at temperature 750 °C respectively with residence time 5, 60, and 120 min was shown in Table 1. In addition, a yield of carbon was estimated according to

$$\text{Carbon yield (\%)} = [W_{\text{ca}} / W_{\text{inc}}] \times 100\% \quad (1)$$

Where W_{ca} and W_{inc} are the weight of carbon product and the tire precursor, respectively. Previous study reported that when pyrolysis of tire waste was conducted with no residence time, at temperature 700 °C and 800 °C, carbon yield were 16% and 12% respectively¹²⁾.

There was a significant weight loss in the carbon

produced after pyrolysis. This result can be explained by the fact that during the pyrolysis process, non-carbon materials such as oxygen, hydrogen, nitrogen, sulfur, etc. have been largely excreted in the form of gases and oils so that carbon becomes a prominent element of the resulting char.

The SEM images of activated carbon prepared at 5, 60, and 120 min of residence time respectively are shown in Figs. 3 – 5. It can be seen in Fig. 2 that there are many small particles assumed to be amorphous carbon produced after pyrolysis. A SEM pictures of tire waste from pyrolysis treatment also show the same condition^{13) 14)}. The average pore diameter was found to increase with the increasing of residence time. This finding is in agreement with carbon production from waste tires with Potassium Hydroxide (KOH) impregnation followed by pyrolysis. Extending holding time from 1 hr to 3 hr resulted in the removal of carbon atom on pore walls, which widened the micropore¹²⁾. At 5 min residence time, there was no pore detected after pyrolysis process. The only visible pore of 20 μm was the pore of tire that can be seen by naked eye when the tire was cut before pyrolysis treatment. After 60 min, a 2 μm of pore is observed and become 4 μm after 120 min of residence time.

Table 1 Carbon weight with variation of residence time

Residence Time (min)	Initial Weight (gr)	Carbon produced Weight (gr)	Carbon Yield (%)
5	25	10	40
60	25	9	36
120	25	8	32

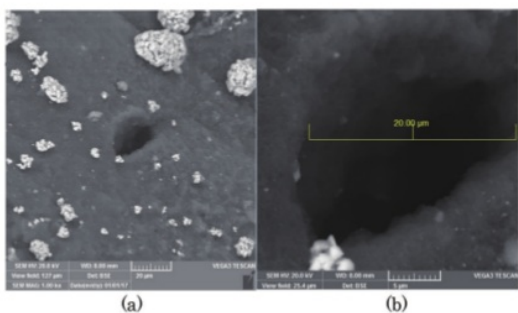


Fig. 3 SEM picture of tire waste carbon prepared at residence time 5 min with magnification of (a) 10000 times, (b) 100000 time

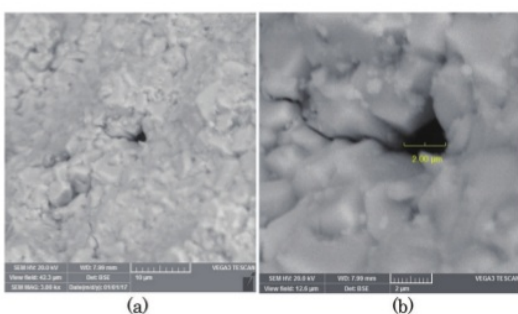


Fig. 4 SEM picture of tire waste carbon prepared at residence time 60 min with magnification of (a) 10000 times, (b) 100000 times

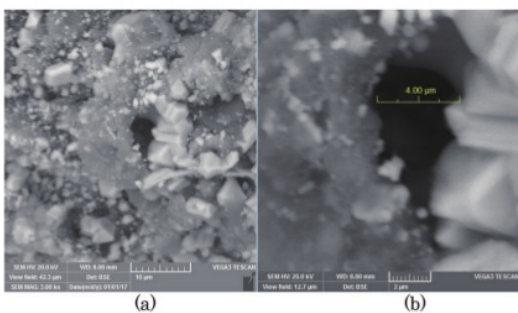


Fig. 5 SEM picture of tire waste carbon prepared at residence time 120 min with magnification of (a) 10000 times, (b) 100000 times

3.3 Carbon activation using NaCl

Pore size distribution and average pore diameter are significantly affected by the pyrolysis parameters and chemical activation. Table 2 shows how the pore size distribution varied with the residence time after NaCl activation. It can be seen that the increase in the value of this parameter results in an increase of pore volume and average pore diameter. An increasing of pyrolysis temperature from 700 to 800 °C can be attributed to the extensive gasification break through pore wall, resulting in an increase in pore volume¹²⁾.

The porosity development of the carbon prepared was investigated by N₂ adsorption. Fig. 6 shows the typical isotherms of N₂ adsorption on the carbons prepared from tire waste at different pyrolysis residence time. It can be seen that the isotherms exhibited loop with similarity to the type-I and IV based IUPAC classification^{15) 16)}. Type-I indicates the presence of abundant micropores, and type-IV demonstrated the appearance of a combination of micro and mesopores. The pore size distribution numerous concentrated on 1.67 nm, 1.78, and 2.19 nm for 5, 60, and 120 min of residence time.

The SEM picture of carbon produced at different residence time after chemical activation by NaCl is shown in Fig. 7. A slightly increasing of pore size is observed after activation. Pores with estimated sizes of 5 µm and 8 µm were formed respectively after 60 min and 120 min of residence time. Chemical agents are used in the activation to conceive the pathways of pores that had been developed by pyrolysis and to prevent the production of tar and enhanced the release of the volatile matters from the carbon. The reaction between the carbon atoms and dehydrating agent is promoted in the extended interlayer of carbons. Upon activation, the salt left in the pores are eliminated by acid wash¹⁷⁾. NaCl as a chemical activator is able to suppress the hydrocarbon impurities in the carbon pore that enlarge pore diameter size. Furthermore, NaCl has a higher boiling point than carbonization temperature thus does not decompose during carbonization to produce hazardous products^{18) 19)}.

Table 2 The surface characteristic of the carbons from pyrolysis of tire waste at different residence time with NaCl activation.

Residence Time (min)	Carbon Yield (%)	BET SA (m ² /g)	Pore Volume (cm ³ /g)	Average Pore Diam. (nm)
5	40	25.55	1.33	1.05
60	36	28.19	6.64	4.71
120	32	27.53	6.33	4.59

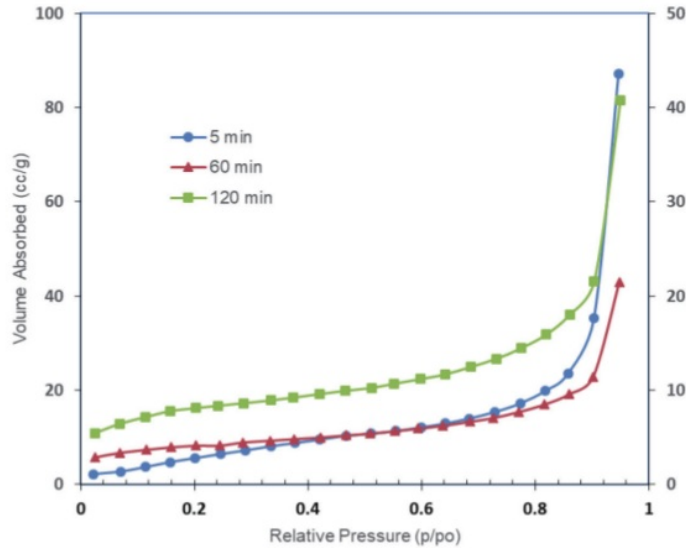


Fig. 6 Adsorption isotherms of N₂ on the carbon prepared from pyrolysis and NaCl activation with residence time variation

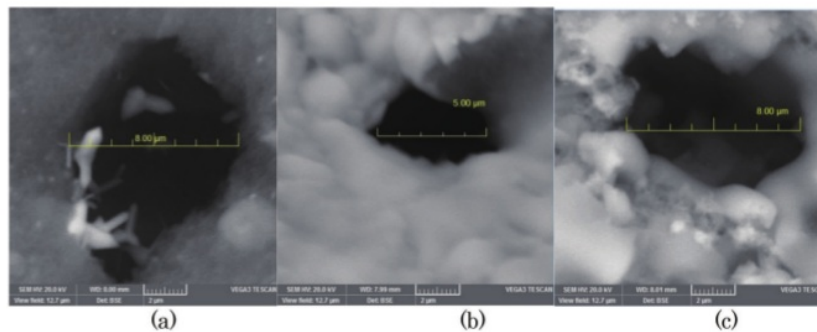


Fig. 7 SEM picture of tire waste carbon activated with NaCl with residence time: (a) 5 min, (b) 60 min, (c) 120 min

4. Conclusion

This study demonstrated that activated carbon can be produced from pyrolysis of tire waste using pyrolysis method and N₂ activation. After pyrolysis conducted at 750 °C, the carbon yield was found to decrease with extending residence time. The N₂ adsorption result shows the porosity development that consists of a combination of micro and mesopores in accordance with BET analysis. The SEM picture shows that the average pore diameter was found to increase with increasing of residence time and after activation using NaCl. Future research will be conducted to investigate a surface area, pore size distribution, and increasing pore diameter.

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