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GASIFICATION OF CHARCOAL IN FLUIDIZED BED FOR MANUFACTURING ACTIVE CARBON

(Gasifikasi arang dalam fluidized bed untuk membuat karbon aktif)

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Ringkasan

Percobaan gasifikasi arang dalam fluidized bed untuk pembuatan arang aktif ini, dilakukan dalam rangka kerjasama penelitian ATA 251 Indonesia - Belgia yang diselenggarakan oleh Pusat Penelitian dan Pengembangan Hasil Hutan, Bogor.

Aspek yang dibahas dalam penelitian ini adalah kondisi pengolahan dengan maksud untuk mempelajari pengaruh suhu dan waktu reaksi aktivasi terhadap rendemen dan kualitas karbon aktif yang dihasilkan. Kondisi perlakuan yang diberikan pada percobaan ini adalah temperatur aktivasi tiga tingkat (731°C, 767°C, 827°C) dengan masing-masing waktu reaksi lima tingkat (10, 20, 30, 40, 50 menit). Kondisi gasifikasi untuk aktivasi dibuat tetap yaitu laju alir uap 0,41 kg/jam dan nitrogen sekitar 1,70 kg/jam.

Hasil penelitian menunjukkan bahwa semua kondisi perlakuan yang diberikan dapat menghasilkan karbon aktif kualitas baik yang ditetapkan oleh daya serap terhadap jodium yaitu 510 sampai 1068 mg/g. Kualitas tersebut termasuk dalam standar perdagangan yang dibuat oleh "American Water Work Association" (AWWA) yaitu sebesar tidak kurang dari 500 mg/g.

Kondisi optimum proses dicapai pada suhu 827°C dengan waktu reaksi 10 menit. Pada kondisi ini memberikan rendemen karbon aktif 61,7% dengan daya serap jodium 687 mg/g. Selanjutnya waktu reaksi optimum terhadap daya serap karbon aktif hasil untuk masing-masing perlakuan suhu reaksi kelihatannya sama yaitu sekitar 40 menit.

Konsumsi uap optimum dalam percobaan ini adalah 3,7 kg untuk tiap kg karbon aktif hasil. Konsumsi uap yang diperlukan tersebut lebih rendah jika dibandingkan dengan kebutuhan uap untuk produksi karbon aktif skala komersial dengan cara kiln berputar di Jerman yang dilaporkan oleh Hormat (1952) yaitu 8 kg uap untuk 1 kg karbon aktif.

I. INTRODUCTION

In terms of commodity, activated carbon is clasified as carbon adsorbent i.e. a form of carbon having a high adsorptive capacity with surface area greater than 400 m^2/g, and belongs to the standard quality of commercial activated carbon.

It is used for adsorbing various kind on dissolved pollutant or impurities from gas phase and liquid phase.

Principally, activated carbon can be manufactured from biomass or any carbonaceous material but commercial brands of activated carbon may be produced from coke, anthracite, wood, coconut shells, palm shells and bones. Activated carbon from biomass is also called activated charcoal and is usually produced from wood or coconut shell char. The production of activated carbon from wood mostly performed by gasification with thermal activation of its char with a high level of heat treatment ranging from 750°C to 1100°C in the presence of limited steam or gas. For this purpose a rotary kiln or fluidized bed oven may be used for thermal activation of carbon.

Indonesia has a great deal of biomass in the form of wood generated from forest logging activities which is available as raw material for producing activated carbon. Logging activities wood processing, and land clearing for transmigration are now producing a lot of wood waste which presently unutilized.

On the other hand, the Indonesian government places great emphasis on the country's industrialisation. Various key industries such as sugar, food, oil, chemicals, drug, refining fossil oil, and water treatment have been encouraged and developed. These industries will need activated carbon as an auxilary material for purification of their products.

Thus the accessibility of raw materials and industrial development will significantly place the active carbon as an important commodity in the industry. In other words, this endeavor will be able to support the establishment of activated carbon industry in Indonesia in the near future.

On the contrary, comparing with other industries, the production cost of producing activated carbon is still reasonably high. Factors contributing to the high component cost such as raw material, transportation, operation, and investment should be avoided from the high economical cost.

In order to find cheaper alternatives to commercially develop activated carbon, a research programme was set up at P3HH to prepare activated carbon from locally available waste materials.

II. EXPERIMENTAL

A. Raw material

The charcoal used for activated carbon was the mixed hardwood charcoal from sawmill wastes. The samples of charcoal taken were classified as waste charcoal with small size of about 15 mm. As feedstock, charcoal received are chopped into small pieces. From these chopped charcoal a fraction of 2-4.75 mm is separated by sieving and used during the process of the experiment.

B. Manufacture

The activating gasification process is carried out in a bach fluidized bed reactor (Figure 1), at the Free University of Brussel. The fluidizing gas is a mixture of steam and nitrogen. The nitrogen is supplied by pressurized gas bottles (1). The flow rate is measured by a rotameter (2). The steam is produced from distilled water (3), which is fed to an evaporater (4) by a metering pump (5). The mixture of steam and nitrogen is preheated in a tubular furnace (6) and fed to the reactor with heating blanket (7) The reactor is made of inconel 600 alloy and insulated with kaowool ceramic fiber. The reactor zone (8) has a diameter of 5 cm and a height of 35 cm. The freeboard zone (9) has a diameter of 10 cm and a height of 30 cm. The total height of the reactor is 80 cm. The feedstock charcoal of 30 gram is fed in to batchwise through a sluice system (10) with two ballvalves. The reactor is filled with 450 gram of silica sand with average particle size of 0.35 mm. The temperature of the reactor is regulated with an external electrically heating blanket. The reactor is followed by a cyclone seperator (11). The pressure drop over the bed is measured by an U-tube manometer (12) and the temperatures are measured with thermocouples of various height in the reactor. These activation experiments are performed at isothermal temperature, i.e. 731°C, 767°C, and 827°C with reaction time of 10, 20, 30, 40, 50 minutes. The steam and nitrogen flow rates are set at the desired values of 0.410 kg/hour and 1.7 kg/hours, respectively. During each period, samples of the produced gas are taken and their composition is determined with a gas chromatograph. The reaction is stopped by switching off the heating of the reactor and the steam flow rate regulator. The reactor content is then left to cool down to room temperature. After one night the reactor content is already cold. The contents are removed and the activated carbon of 2 mm diameter and up is separated from the sand by sieving.

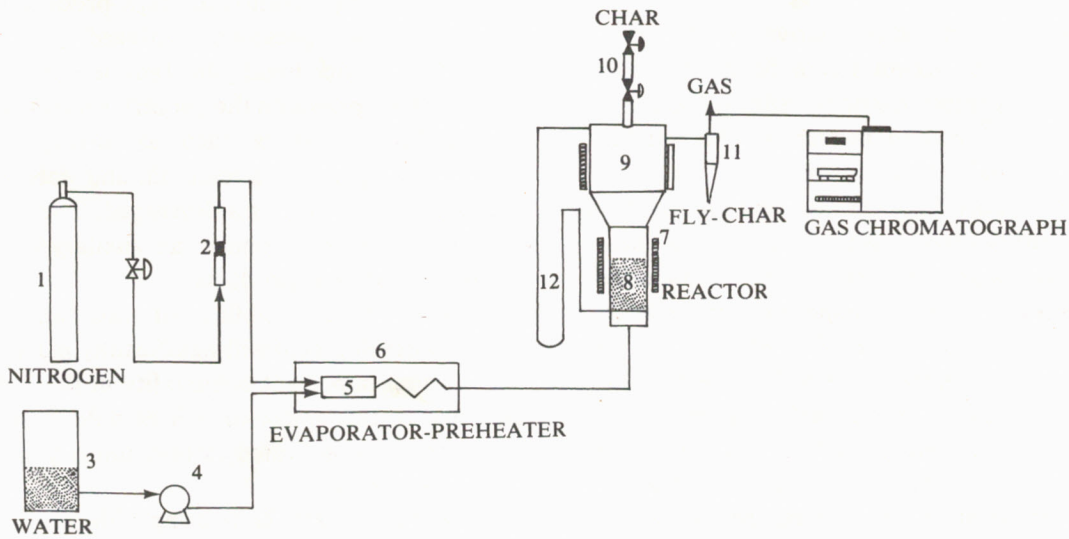

Figure 1. Fluidized bed activation unit.
Gambar 1. Unit "fluidized bed".

1. Gas bottle
2. Rotameter
3. Water supply
4. Pump
5. Evaporator
6. Furnace
7. Heating blanket
8. Sand bed
9. Freeboard zone
10. Sluice
11. Cyclone
12. U-tube manometer

C. *Evaluation of raw material and product*

The quality of charcoal used as raw material was evaluated by proximate analysis by using the ASTM standard for coal (ASTM D-5) as reference. This analysis method is widely used for practical evaluation of the charcoal quality as raw material in industrial applications.

The quality of activated carbon produced was determined by measuring adsorptive capacity of iodine number (AWWA B 600-78 Colorado). The iodine index gives an indication of the amount of pores larger than 10 Å. It is also proportional to the amount of internal surface area and may be used to show whether a carbon is activated or not.

III. RESULTS AND DISCUSSION

1. Properties of charcoal

The results of proximate analysis of charcoal received is given in Table 1.

Table 1. Charcoal properties.
Tabel 1. Sifat arang kayu.

Properties (*Sifat*)	Value (*Nilai*), %	
	Based on sample received (*Atas dasar contoh*)	Based on oven dry basis (*Atas dasar kering tanur*)
Moisture contents (*Kadar air*)	5.6	5.9
Ash content (*Kadar abu*)	3.9	4.1
Volatile matter (*Kadar zat mudah terbang*)	17.1	18.1
Fixed carbon (*Karbon sisa*)	73.4	77.8

From Table 1 it can be seen that the fixed carbon is relatively high. The high fixed carbon will contribute to the high yield and quality of the active carbon produced.

A comparison of results with survey data shows that the quality of the charcoal is comparable with that of the charcoal from wood as raw material for commercial activated char.

2. Influence of the temperature and reaction time on the activated carbon yield

The graphs in the Figure 2 show the effect between the isotherm temperature and reaction time on the activated carbon yield.

It is shown that as the temperature of activation increases, the yield of the activated carbon decreases. This can be explained by the kinetic theory in general, as the temperature increase, the rate of reaction between carbon and steam increases as well, therefore, the amount of carbon reacted within a unit time increases, then the carbon left deacreases.

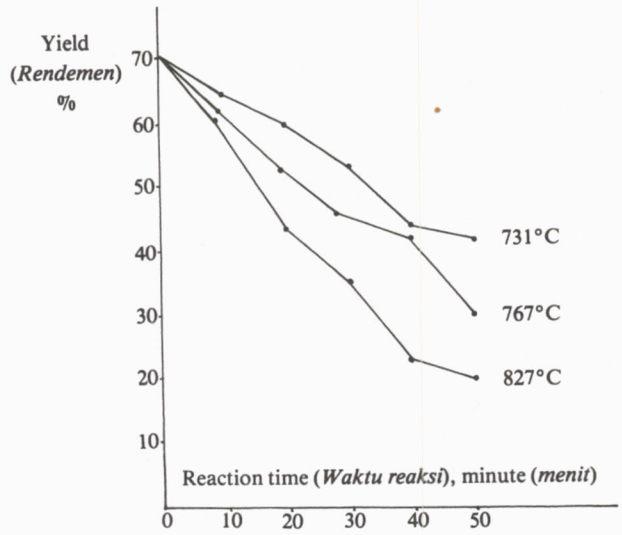

Figure 2. Influence of temperature and reaction time on the activated carbon yield.
Gambar 2. Pengaruh temperatur dan waktu reaksi terhadap karbon aktif hasil.

The influence of residence (reaction) time on activated carbon yield shows that long residence time is significantly influencing the decreases in the activated carbon yield. This can be explained by logical fact that as residence time increases, the amount of carbon reacted increases as well, and the amount of carbon left decreases.

The activated carbon yield obtained shows the results with wide variation, ranging from 20 to 66.7% (See Appendix). The highest yield was obtained at 731°C, whithin a short residence time of 10 minutes, while the lowest yield is at 827°C within 50 minutes. All these yields obtained are some what lower than the actual product of activated carbon. The reasons are that in reality, some carbon will be lost by attrition of particles and intrainment with the fluidizing bed. In addition to that the other carbon with very small size particle (< 2 mm) was seperated by sieving and neglected.

3. Influence of temperature and reaction time on the adsorptive capacity of the activated carbon produced

This relationship between the adsorptive capacity of activated carbon at each isotherm and reaction time condition is limeted by the experimental data obtained it can drawn by the graph (Figure 3).

It has been observed that all indices were initially rise with increasing reaction time until approximately 40 minutes. These indices tend to decrease afterwards at

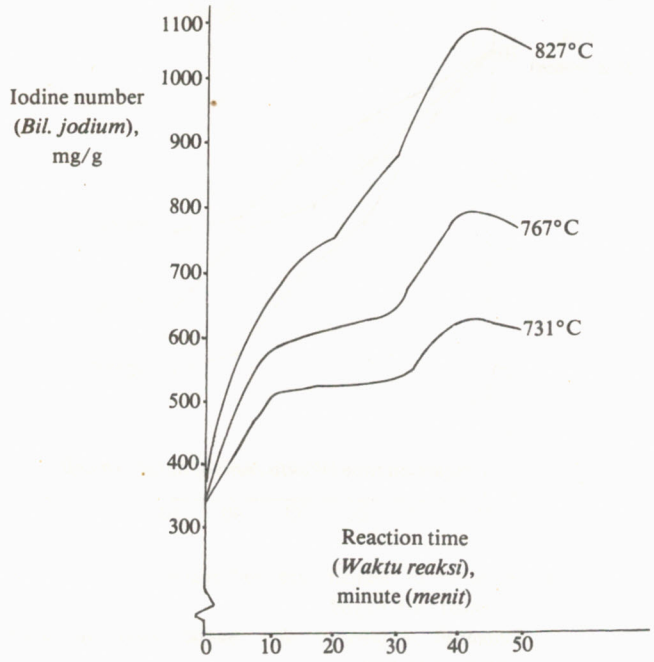

Figure 3. Influence of the temperature and reaction time on adsorptive capacity of iodine of activated carbon.

Gambar 3. Pengaruh temperatur dan waktu reaksi terhadap daya serap jodium karbon aktif.

the reaction time of 50 minutes. The trends can be explained by the theory of creating pores-structure of active carbon resulting in the increases of the internal surface area. Until approximately 40 minutes the increasing the temperature of activation and carbon conversion simultaneously create pore structure followed by the increasing of the internal surface area.

The long reaction time increases carbon conversion (carbon reacted) but until a certain residence time the wall of the pores is initially destroyed. In consequence of this proces, the internal surface area of active carbon decrease. Then the decreasing amount of internal surface area is proportional to the deacreasing of the iodine number. It can be seen that an activated carbon produced at each treatment condition gives iodine number of more than 500 mg/g and the highest iodine number of 1068 mg/g. These figures show that all products are above the specified for minimum iodine number as determined by AWWA which is not less than 500 mg/g.

4. *Optimum activation condition and optimum reaction time*

Optimum condition for activation from these experiments is defined as a treatment condition which can give the best results of the activated carbon product based on both factors of yield and its adsorptive capacity of iodine number. The best results can be determined by multiplying the yield of activated carbon (in percent) with its adsorptive capacity of iodine number. In these experiments, the results of multiplying the two factors aboved is called "Total Iodine Index Yield".

From the table in the appendix, it can be concluded that the optimum temperature of activating condition is at 827°C within a short reaction time of 10 minutes. This condition gives the total iodine index yield of 423 mg/g, which is obtained from 61.7% yield and 687 mg/g iodine number.

The optimum reaction time at each isotherm activation (731°C; 767°C and 827°C) seems to be the same time, which is approximately 40 minutes. Based on the three curves in the figure aboved displayed the same trend.

5. *Properties of activated carbon*

Ideally, the optimum method of determining the quality of activated carbon is by measuring its performance in the removal of the specific impurities present in the gas phase or liquid phase to be treated. However, there are typically several different impurities at varying concentration that are to be adsorbed in a particular treatment system. Consequently, determination of the activated carbon quality in connection with its effectiveness by evaluating its performance in adsorbing the actual impurities present in the gas or liquid phases to be treated may not be practical.

Evaluating the quality of activated carbon produced in these experiments is determined by adsorptive capacity of iodine (number) with AWWA standard. The results comparison study with adsorptive capacity iodine number of several kinds of commercial activated carbon are summarized in Table 2.

Table 2. Iodine number adsorptive capacity of activated carbon.

Tabel 2. Daya serap karbon aktif bilangan jodium.

Kinds of activated carbon (*Jenis karbon aktif*)	Iodine number (*Bil. jodium*), mg/g	Remarks (*Keterangan*)
Obtained from these experiment (*Dari percobaan*)	510—1068	
G A C 40 (CeCa)	1148	Commercial brand
F 400 (Calgon)	1118	,,
HD 4000 (ATLAS)	558	,,
F 300	973	,,
Commercial powdered activated carbon (*Arang aktif komersial*)	500— 600	,,

As follows from Table 2 the activated carbon obtained from these experiments shows good adsorption. This quality is comparable with activated carbon of the commercial brand.

IV. CONCLUSION

The results of the experimental studies on gasification of charcoal for manufacturing activated carbon can be summarized as follows:

1. Wood charcoal from sawmill waste is suitable as raw material for activated carbon. The quality of charcoal obtained by this process is comparable with charcoal from wood or other biomass used as raw material for commercial activated carbon.
2. Activating process by means of steam gasification in fluidized bed at 731°C, 767°C and 827°C can produce good quality activated carbon as determined by adsorptive capacity of iodine index. Iodine number of activated carbon produced ranges from 510 to 1068 mg/g. This quality belongs to the required standard of commercial activated carbon which is not less than 500 mg/g.
3. Activated carbon yield obtained during these experiments ranged from 20.0% to 66.7%. The maximum yield is attained at 731°C within 10 minutes of reaction time, while the minimum yield was at 827°C and 50 minutes.
4. Increasing temperature of activation decreased activated carbon yield but increased adsorptive capacity as expressed by its iodine number. Until approximately 40 minutes of residence time (reaction time) the iodine index of activated carbon increased with the increase of residence time. Long residence time significantly decrease activated carbon yield, however.
5. The optimum activating condition determined during this experiment was 827°C within a short residence time of 10 minutes. From this condition the yield of activated carbon was 61.7% with its iodine number of 687 mg/g.
6. The optimum residence time observed with respect to adsorptive capacity as expressed by the iodine number of the activated carbon obtained, at every isotherm condition namely 731°C, 767°C and 827°C showed the same reaction time, which is 40 minutes. At these isotherm conditions the highest iodine number attained were 624, 791 and 1068 mg/g, respectively.

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Appendix. Yields and properties of activated carbon produced at varying temperature and reaction time.
Lampiran. Rendemen dan sifat karbon aktif pada berbagai temperatur dan waktu reaksi.

Number of sample (*Nomor contoh*)	Activation temperature (*Temperatur aktivasi*), °C	Reaction time (*Waktu reaksi*), minute (*menit*)	Yield (*Rendemen*), %	Adsorption capacity iodine number (*Daya serap bil. jodium*), mg/g	Total iodine index yield (*Hasil bil. jodium total*)
0	731	0	70.0	349	244.30
1		10	67.7	510	345.27
2		20	60.0	515	309.00
3		30	51.7	526	271.94
4		40	46.7	624 (max)	292.34
5		50	43.3	602	260.67
6	767	0	70.0	348	243.60
7		10	63.3	582	368.41
8		20	53.3	615	327.80
9		30	48.3	645	261.23
10		40	40.6	791 (max)	321.15
11		50	30.0	762	228.60
12	827	0	70.0	346	239.40
13		10	61.7	685	422.65 (opt)
14		20	43.3	753	326.05
15		30	36.7	880	322.96
16		40	23.3	1068 (max)	248.84
17		50	20.0	1038	217.60