Effect of activation time the chicken feather activated carbon on surface area of pores: Candidate for hydrogen storage application

T Partuti, A Alhamidi and M Y Ardiansyah
Department of Metallurgy, Faculty of Engineering, University of Sultan Ageng Tirtayasa, Cilegon, Banten, Indonesia

Abstract. The use of renewable energy such as hydrogen gas as fuel is still very lacking due to difficulties of its storage. Hydrogen storage material is focused on developing porous material. Chicken feather is one of the organic wastes that can be used as porous activated carbon. The chicken feather activated carbon made in powder and briquette form. The activation process was carried out physically with variation in time 30, 60 and 90 minutes, temperature 500 °C and flow rate of nitrogen 0.3 l/minute. The carbon content from carbonization of chicken feathers is 62.85% using ultimate analysis. The activated carbon analysis with Brunauer-Emmett-Teller (BET) show that surface area for powder sample before activation is 0 m²/g and with activation time 30, 60 and 90 minutes is 79.28 m²/g, 128.28 m²/g and 332.49 m²/g, respectively. The surface area for briquette sample before activation is 20 m²/g and with activation time 30, 60 and 90 minutes is 741.37 m²/g, 150.55 m²/g and 162.65 m²/g, respectively. The Scanning Electron Microscope (SEM) results show that pores appear on the briquette sample are much more and more visible than the powder sample. The pore surface area of the briquette sample is inversely proportional to the increase of activation time. The longer the activation time the pore surface area of the briquette sample decreases. While for powder samples vice versa.

1. Introduction
The consumption of fuel oil in Indonesia in the last three years has increased. In 2015, fuel consumption reached 44.45 billion litres, increasing to 48.65 billion litres in 2016. And in 2017, it increased to 55.40 billion litres [1]. The increasing consumption of fuel oil and limited petroleum supplies can cause an energy crisis. At present, Indonesia is developing renewable energy as an alternative energy for fuel, but the utilization of renewable energy resources has not been fully utilized. It can be seen at Figure 1 that composition petroleum energy mix is 41.43%, coal 30.48%, natural gas 23.37%, hydropower 2.89%, geothermal energy 1.37% and biofuel 0.165%. In total, the utilization of renewable energy in 2016 amounting to 4.42% [2].

Hydrogen can be an alternative to one of renewable energy because it is an abundant element up to 75% of the total mass element of the universe which has many advantages, such as combustion of hydrogen does not cause pollution, but produces water as shown in equation (1). The energy produced by hydrogen is better than the energy produced by petroleum, because its combustion value has three times higher calorific value, which is 28700 Kcal/kg and 10630 Kcal/kg for petroleum [3].

\[ 2 \text{H}_2(\text{g}) + \text{O}_2(\text{g}) \rightarrow 2 \text{H}_2\text{O}(\ell) + \text{energy} \]  \tag{1}

The use of hydrogen as renewable energy is still constrained by storage. At room temperature and atmospheric pressure, the hydrogen form is gas which has a very low energy ratio to its volume.
Research on methods and types of materials in hydrogen storage is still ongoing. Activated carbon as an adsorbent can reduce pressure in tanks with relatively the same storage capacity, so that the explode properties of hydrogen can be reduced [4]. An adsorbent must meet the criteria of having a large surface per mass unit so that the adsorption capacity will also be greater [5]. Besides that, it must be obtained easily. One of the ingredients being developed for the manufacture of activated carbon is chicken feathers.

![Figure 1. Energy utilization in Indonesia in 2016 [2].](image)

Chicken is protein’s source for human. The more chickens consumed, the more available chicken feathers. Chicken feathers contain keratin with consist of 79.88% crude protein, 3.77% crude fat and 0.32% crude fibre [6]. The chemical composition in chicken feathers is almost 50% carbon, 7% hydrogen, 16% oxygen, 0-3% sulphur [7]. With high carbon content in it, chicken feathers can be used as a carbon source, then activated into activated carbon to produce hydrogen storage. The activated carbon from chicken feathers has a surface area of 430 m$^2$/g so that chicken feathers can be used as an adsorbent [8]. Chicken feather has a large surface area and various pores structure, by carbonization and KOH activation. The higher activated temperature can result a larger surface area. Micro-porous structure is formed during the pyrolysis of chicken feathers in the absence of oxygen at 350 °C and collapse above 500 °C [9].

The purpose of this research is making activated carbon from chicken feathers as an adsorbent physically with activation of time 30, 60 and 90 minutes, at temperature 500 °C, and flowrate of nitrogen is 0.3 l/minutes to get more surface area of pores as candidate of hydrogen storage.

2. Experimental Method
The preparation begins with washing chicken feathers using clean water and then separate between barbs, rachis and calamus, then dried under the sun until dry completely. Temperature of carbonization process for barbs at 300 °C, while rachis at 400 °C for 60 minutes each. The carbon produced from this process then crushed to powder with size of 100 mesh. Briquette sample made from 2 g powder sample added 0.04 g tapioca starch and 2.5 ml water, then printed on a steel cylinder with diameter 5 mm and 10 mm height, pressed at 150 bars for 60 minutes, then sintered at 300 °C for 90 minutes. Carbonized chicken feather analysed with proximate (LECO TGA-601) according to ASTM D7582-12, ultimate (LECO CHN-2000) according to ASTM D5373-13 and FTIR instrument. Activation process for powder and briquette sample is done physically with temperature 500 °C for 30, 60 and 90 minutes, flowrate of nitrogen is 0.3 l/minutes. Activated carbon produced analysed with BET and SEM to determine the difference of surface area and pore between before and after activation.

3. Result and Discussion
3.1. Carbonization of chicken feather
The carbonization result by proximate analysis is moisture content 5.96%, volatile matter 43.93%, ash 2.20% and fixed carbon 47.91%. Besides carbon, fixed carbon also contains hydrogen, oxygen and sulfuric. Ultimate analysis result shows that the contents of carbon 62.90%, hydrogen 4.20%, nitrogen 15.65% and oxygen 17.25%. Figure 2 shows the spectra FTIR of carbonized chicken feather.

![Graph](image)
Figure 2. Spectra FTIR for carbonized chicken feather.

It shows that C-H bond is the dominant. And there are C-C bond, C=C bond, C≡C bond appears at specific wavenumbers as shown at Table 1. The presence of an O-H group in the carbonization process is possible from reaction between water vapor and free compounds on the carbon surface [10].

Table 1. FTIR analysis for bond appears in carbonized chicken feather.

| Bond | Wavenumber (cm\(^{-1}\)) |
|------|--------------------------|
| C-H  | 641-1362, 1508, 2522-2730, 3121 |
| C-C  | 1442, 2352, 2874, 2960 |
| C=C  | 1600-1657, 3076 |
| C≡C  | 2130-2220 |
| O-H  | 3634-3908 |

Figure 3 shows the SEM result for powder and briquette sample of carbonized chicken feather with 1500x magnification. There’s no pores surface area in powder sample, but in the briquette sample there’s slightly pores surface area of 20 m\(^2\)/g.

3.2. Activation of carbonized chicken feather

The activation of chicken feather carbon used two forms of sample, namely briquette and powder. Diameter of briquette is 5 mm with 10 cm length. Figure 4 shows the colour change happened in carbon briquette caused by the activation time process. Figure 4a shows that for 30 minutes activation, the carbon briquette hardly shows any colour changes happened, the colour remains black. For 60 minutes
activation, the colour change from black to partial brown (Figure 4b). For 90 minutes activation, it shows a more intense colour change, namely reddish brown (Figure 4c).

In Figure 5, the physical form changes with the time of activation. The carbon powder sample getting agglomerate when activated. The longer the activation time takes place, the carbon is agglomerated perfectly (Figure 5c). During the activation process, there was rearranging the carbon atoms in the horizontal direction and expanding to a higher vertical direction, so that the crystallites formed became smaller due to the increasing distance between the crystals [11].

3.2.1. Surface area of activated chicken feather

The BET analysis result is shown in Table 2. It shows that the pore surface area of the powder before activation is 0 m²/g and 20 m²/g for briquette. The difference happens because the compacted and sintered process of briquette sample before activated. Compaction makes particles in the briquette are cold welded together and have a very weak bond and the next sintering process makes the particles in the briquette become fused together [12]. The presence of starch as natural binder also helps binding the briquette together and probably reduce the brittleness of the briquette [13]. When sintering happened, the evaporation at briquette sample occurred slowly than powder sample, so that the damage of the carbon matrix would be hampered [14]. The pore surface area for briquette sample decreased significantly, from 741.367 m²/g with 30 minutes activation to 150.546 m²/g with 60 minutes activation and for 90 minutes activation had a pore surface area of 162.654 m²/g. It might be happened because starch as a binder attached to carbon strongly. And with constant activation temperature at 500 °C, although the activation time increases, it has not succeeded in breaking the bond between the binder and carbon so it’s difficult to form the pores in the briquette sample.

| Activation time (minutes) | Pore surface area (m²/g) |
|---------------------------|--------------------------|
|                           | briquette | powder         |
| 0                         | 20.173    | 0              |
| 30                        | 741.367   | 79.285         |
| 60                        | 150.546   | 128.283        |
| 90                        | 162.654   | 332.489        |

But for powder samples, it gave the different result. Activated carbon in powder form with the activation time of 30 minutes produces a pore surface area of 79.285 m²/g, 128.283 m²/g for activation time of 60 minutes and 332.489 m²/g for 90 minutes activation process. The longer the activation time,
the more substances on carbon’s surface are released, so that the surface pores at activated carbon is greater and the ability to absorb also increases.

3.2.2. Morphology of activated chicken feather
The surface morphology of briquette and powder sample from chicken feather activated carbon analysed with SEM which produces 1500x magnification for briquettes (Figure 6) and 2000x for powder sample (Figure 7).

![Figure 6](image1)

**Figure 6.** Surface morphology of briquette sample from chicken feather activated carbon with activation time 30 minutes (a), 60 minutes (b) and 90 minutes (c).

The greater surface area was generated in 30 minutes activation for briquette sample, which is 741.367 m$^2$/g. The 1500x magnification used in the SEM results only shows macro pores, while the micro and meso pores are not visible. The presence of a binder resulted the closure of the adsorbent micropore [14]. Meso and micro pores are smaller than macro pores and form roots around the macro pore. Activation carried out on the briquette carbon produces cracks in the carbon particles indicating the formation of pores in the crack s between the particles. Figure 6a shows many cracks which indicate the formation of pores in it and the estimate of pores were 13.202%.

![Figure 7](image2)

**Figure 7.** Surface morphology of powder sample from chicken feather activated carbon with activation time 30 minutes (a), 60 minutes (b) and 90 minutes (c).

Figure 7 shows that powder sample from chicken feather activated carbon with activation of 30 minutes (a) and 60 minutes (b) has a slight crack on the surface of sample, but for 90 minutes activation the cracks are clearly visible on the surface. The estimated of pores is 6.25% for 90 minutes activation time. The pores structure resulting from SEM characterization shows that during the activation process the crystallite becomes exposed to the activating gas which can encourage hydrocarbon residues. The shift of carbon plates produces new pores and develops initial micropores into macropores and decreases the degree of crystallinity. This research inversely to the research conducted by Senoz et. al. which maintains the fibre structure of chicken feather in carbonization and activation process, so that the fibre structure can be seen, while this research made in briquette and powder sample [8].
4. Conclusion
The pore surface area of the briquette sample is inversely proportional to the increase of activation time. The longer the activation time the pore surface area of the briquette sample decreases. While for powder samples vice versa. The surface area for briquette sample with activation time of 0, 30, 60 and 90 minutes was 20 m$^2$/g, 741.37 m$^2$/g, 150.55 m$^2$/g and 162 m$^2$/g, respectively. While for powder sample with activation time of 0, 30, 60 and 90 minutes was 0 m$^2$/g, 79.28 m$^2$/g, 128.28 m$^2$/g and 332.49 m$^2$/g, respectively.

5. References
[1] Badan Pengatur Hilir Minyak Dan Gas Bumi Nasional 2019 National Fuel Consumption Per Year (Jakarta: BPH Migas)
[2] Kementerian Sumber Daya Alam dan Mineral 2017 Kajian Penyediaan dan Pemanfaatan Pusat Data dan Teknologi Informasi Energi dan Sumber Daya Mineral Jakarta (Jakarta: Pusat Data dan Teknologi Informasi ESDM)
[3] Ali J 2012 Pengembangan Adsorben Hydrogen Storage Untuk Aplikasi Fuel Cell Dalam Bentuk Padatan Partikel Nano Karbon Aktif Dengan Bahan Pengikat Liquida Linoselulosa (Jakarta: Universitas Indonesia Press)
[4] Awasthi K E, Kamalakaran R, Singh A K and Srivastava O N 2002 Int. J. Hydrogen Storage 27 425
[5] Hendra R 2008 Pembuatan Karbon Aktif Berbahan Dasar Batubara Indonesia Dengan Metode Aktivasi Fisika Dan Karakteristiknya (Jakarta: Universitas Indonesia Press)
[6] Ketaren N BR 2008 Pemanfaat Limbah Bulu Ayam Sebagai Sumber Protein Ayam Pedaging Dalam Pengelolaan Lingkungan Hidup (Medan: Universitas Sumatera Utara Press)
[7] Poedjiadi A 1994 Dasar-dasar Biokimia (Jakarta: Universitas Indonesia Press)
[8] Senoz E and Wool R P. 2012 Physical And Chemical Changes In Feather Keratin During Pyrolysis Polymer Degradation And Stability 97 297
[9] Zhang F S, Nriagu J O and Itoh H 2005 Mercury Removal From Water Using Activated Carbons Derived From Organic Sewage Sludge Water Research 39 389
[10] Nasution Z A and Rambe S M 2013 Characterization And Identification Of Functional Group Of Palm Shell Carbon By Using Methano-Pyrolysis Method J. Dinamika Penelitian Industri 24 108
[11] Jimenez et. al. 1999 Effect Of The Increase In Temperature On The Evolution Of The Physical And Chemical Structure Of Vitrinite J. Analytic and Applied Pyrolisis 50 117
[12] Neely and Bertone 2003 Particle Metallurgy And Materials of Industry 6th edition (New Jersey: Prentice Hall. Inc)
[13] Ikelle I I, Nworie F S, Ogah A O and Ilochi N O 2017 Study on the Combustion Properties of Bio-Coal Briquette Blends of Cassava Stalk J. Chem Search 8 29
[14] Putra I, Alhamidi A A, Kustiningsih I and Sutowo C 2016 Rekayasa Karbon Aktif Dari Bulu Ayam Untuk Bahan Hydrogen Storage J. Furnace 1