A Preliminary Study of Scanning Electron Microscopy (SEM) for Characterization of the Wood Pellet Process of Sengon Wood (Albizia Chinensis)

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Abstract. Previously, Sengon wood waste from existing sawmills was underutilized. Therefore, one way to utilize this wood waste is to convert it into fuel because the energy demand in this world continues to increase. In doing so, an initial study was conducted to determine the characteristics of wood pellets from Sengon wood using Scanning Electron Microscopy-Energy Dispersive of X-ray Spectroscopy (SEM-EDX), resulting in more porosity in the middle than at the bottom and top because the middle is denser than the inside of the Sengon wood pellet, while the upper part has fewer pores because the outer part of the wood pellet experiences friction with the ring die so that the resulting pores are smoother. Meanwhile, on average, the elements that have been tested have the highest atomic weights, namely C-K elements (40.36%); elements O-K (52.08%); element CuL (1.29%); Elements of AIK (0.85%); SiK element (1.95%); CIK elements (0.42%); KK elements (1.39%) and CaK elements (1.64%).

Keywords: Electron Microscopy; X-Ray; Wood Pellet Process

1 Introduction

Energy needs in Indonesia continue to increase while reserves of fossil energy sources are running low. One of the new and renewable energy sources that have been widely developed is biomass alternative energy because it has great potential to be developed in overcoming the energy crisis in Indonesia. One of these biomass energies is wood pellets, which are the result of the densification of the remaining processed wood waste in the form of sawdust. Biomass resources have been considered as one of the most promising renewable raw materials to replace fossil resources today. potential application of renewable energy sources to replace fossil fuel combustion as the main energy source in various countries, and discusses issues related to biomass combustion in boiler power systems. where biomass includes organic matter produced as a result of photosynthesis as well as municipal, industrial and animal waste materials. A summary of the basic concepts involved in burning biomass fuels is presented. Renewable energy sources (RES) supply 14% of the world's total energy demand. RES is biomass, hydropower, geothermal, solar, wind, and marine energy. Renewable energy is the main source of energy, domestic and clean or inexhaustible. The percentage share of biomass was 62.1% of the total renewable energy sources in 1995. Experimental results for various

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fuels and biomass conditions are presented. Numerical studies are also discussed. Biomass is an attractive renewable fuel in utility boilers, with Elements including K, Na, S, Cl, P, Ca, Mg, Fe, Si involved in the reactions leading to ash fouling and slagging in the biomass combustion chamber. [1], [2],[3]

Biomass energy is one of mankind's earliest energy sources, especially in rural areas where it is often the only accessible and affordable energy source. Biomass worldwide ranks fourth as an energy source, providing about 14% of the world's energy needs. All human and industrial processes generate waste, that is, the normally unused and unwanted products of a particular process. Solid waste generation and recovery varies dramatically from country to country and deserves special mention. The burning speed of pulverized biomass fuel is much higher than that of coal. The use of biomass fuels provides great benefits as far as the environment is concerned. Biomass absorbs carbon dioxide during growth and emits it during combustion. The utilization of biomass as a fuel for electricity production offers the advantages of renewable and CO2-neutral fuel. [4],[5],[6],[7]

Impurities and bottom ash were collected from low power boilers after burning wood pellets and studied using several analytical techniques to characterize and compare samples from different areas and determine the suitability of the analytical techniques used. TGA results show that the fouling contains high organic matter (70%). XRF and SEM-EDS measurements revealed that Ca and K were the main inorganic elements and showed a clear trend in the Cl content which was negligible in the bottom ash and increased as it penetrated the deepest layer of impurities. Calcite, magnesia, and silica appeared as the main crystalline phases in all samples. However, bottom ash is mainly composed of calcium silicate. KCl behaves identically to Cl, preferably appearing in the sample of adherent impurities. [8],[9],[10],[11]

The efficient and profitable combustion of biomass is often limited by operational problems related to ash. Knowledge of ash smelting and sintering is very important, in terms of predicting and mitigating ash-related problems in biomass-fired boilers. The four parts of the Pinus sylvestris tree are the trunk, bark, base of branches, and twigs. A simultaneous thermal analyzer (STA) was used to characterize the smelting behavior of selected biomass fuels in an oxidizing atmosphere. The STA experiment shows that the smelting process of the studied fuel ash starts at a temperature range of 930-965 °C. Scanning electron microscopy (SEM) is equipped with energy dispersive X-ray spectrometry (EDX). The results of the analysis showed that the log ash remained loose structure even after 1000 °C sintering treatment. However, the ash from the upper branches showed signs of sintering at 1000 °C. The results obtained from this work can be considered useful information of industrial interest to predict the melting behavior of forest biomass ash. [12],[13],[14]

Based on some of the literature and the problems above, in this research, it is necessary to conduct an initial study of wood pellets taken from underutilized sawmill waste, to be developed into biomass fuel in the form of pellets. The purpose is that the results of this study can be a reference that can be applied to the need for renewable fuels. The initial study carried out was Scanning Electron Microscopy- Energy Dispersive of X-ray Spectroscopy (SEM-EDX) to determine the density level of the bottom, middle, and top of the wood pellet, in addition to knowing the elements in it so that the characterization of wood is known. Sengon (Albizia chinensis) wood pellets.

2 Methodology

SEM Scanning Electron Microscopy is a high magnification microscope that images the surface of a sample using scanning by electron beams. With the working principle of firing high-energy electrons (1 - 20 kV) through the sample and then detecting secondary electrons and backscattered electrons and x-ray characteristics. Where the electron gun used is a tungsten hairpin gun with a filament in the form of a tungsten coil that functions as a cathode. The voltage applied to the winding causes heating. The anode will then form a force that can attract electrons to move towards the anode. Magnetic lenses are used to focus electrons towards a point on the sample surface from a focused electron beam to scan (scan) the entire sample by being directed by a scanning coil when electrons hit the sample, there will be the scattering of electrons, either Secondary Electron or Back Scattered Electron from the sample surface and will be detected by the detector and displayed in the form of an image on a CRT monitor and why SEM is used because electrons have a higher resolution than light. Light is only capable of reaching 200nm, while electrons can reach resolutions of up to 0.1 – 0.2 nm. By using electrons, several types of reflections will be obtained which are useful for characterization purposes, as shown in Figure 1. SEM block diagram.

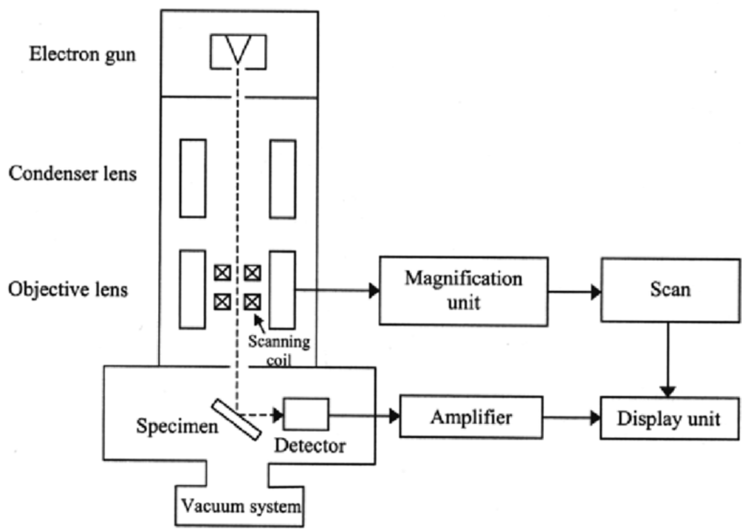

Figure 1. SEM block diagram [15]

Characteristics with Scanning Electron Microscope-Energy Dispersive of X-ray Spectroscopy (SEM-EDX) was carried out to observe the surface morphology, pore size, and elemental content of the lower, middle, and upper Sengon wood pellets with the process as presented in Fig. Figure 2. The interaction of high energy (kV) electrons with (solid) material are shown here. When the electron beam is scanned on the surface of the sample, electrons interact with the atoms on the surface as well as below the sample surface. The electron beam is used to describe the surface shape of the material. analyzed, as a result of this interaction most of the electron beams managed to come out again, these electrons are referred to as Backscattered Electrons, a small portion of the electrons enter the material and then transfer most of the energy to the atomic electrons so that they bounce off the surface of the material, namely Secondary Electrons. The formation of secondary electrons is always followed by the emergence of characteristic X-rays for each element so that it can be used to measure the element content in the wood pellet material being analyzed in this study.

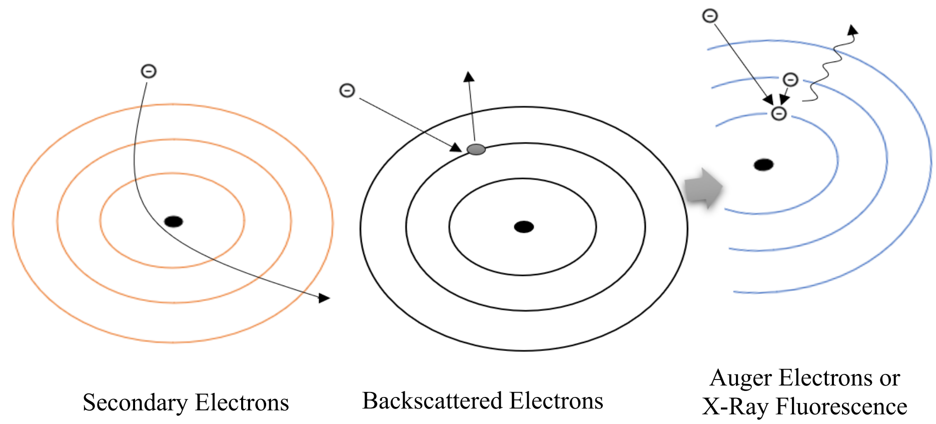

Figure 2. Interaction of high energy (kV) electrons with (solid) material

The process of formation of Backscattered Electrons in Figure 2. occurs in the atoms of the deeper part of the sample surface. This is due to the collision between the electrons from the source and the atomic nucleus, where the mass of the protons that make up the nucleus is greater than the electrons, so each collision will cause to reflect most of the electrons forward, some of which will be reflected back in the direction where they came, namely outside the surface of the material. These backscattered electrons tell us about the atoms they collide with and their bonds in phase. So that the contrast in the image formed from Backscattered Electrons is within certain limits. When the source electron in the process in the material only passes above the electron or orbital of an atom, the electron may transfer some of its kinetic energy to one or more electrons in that orbit. The electron will become unstable and in an excited state so that it leaves its position and exits the surface of the material, then the electron is known as a secondary electron. Because the secondary electrons have low energy, only electrons located or very near the surface of the material can escape. With the help of a special detector, secondary electrons can be utilized to form a good image of the surface morphology of the material. Surface structures such as grain boundaries, edges, porosity, peaks, or valleys will look more detailed with a higher resolution than Backscattered Electrons.

2.1. Sampling Experiment

The sampling of Sengon wood pellets utilized sawing waste of Sengon wood in the Malang area where this Sengon wood waste is widely spread, as Malang has a tropical climate with an average temperature of 23^0 C. The chemical components in this wood can be divided into cellulose, hemicellulose, lignin, and extractives. In the process of making pellets assisted by a wood pellet machine and a rotary kiln with a length of 12000 mm which is used to reduce the moisture content of Sengon wood powder which is on average 80% to 8-11%, then the pellet making process can run, by means of Sengon powder. into the pellet machine continuously through a steel circle with several holes having a size of 10 mm. This compaction process produces a material that is dense and will break when it reaches the desired length. Pellet fuel has a diameter of 10 mm and a length varies between 15–30 mm. In the process of making pellets, it produces heat due to friction of the tool which facilitates the process of binding the material and reducing the water content of the material up to 5-10%. The results of the process of making wood pellets are as shown in Figure 4. Wood pellets of Sengon wood waste using the machine in Figure 3 below.

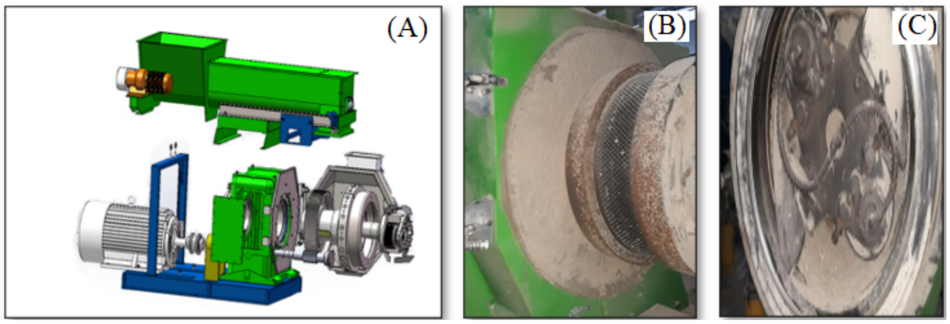

Figure 3. A. Wood pellet machine schematic; B. Ring die; C. Roller

Figure 4. Wood pellets from Sengon wood waste

After the pellet-making process has been completed, testing of the image and composition data of the oxidized sample with the SEM tool is carried out, the sample is placed and affixed to the SEM specimen holder with the cross-section pointing vertically upwards or the objective lens. So that the arrangement of the layers of Sengon wood pellets can be seen clearly. With a double tip made of conductive carbon material on both sides that serves to deliver all electrons that enter the sample out through grounding. The sample chamber is vacuumed to ensure that the SEM column is free of air molecules and for EDX signal acquisition for optimal X-Ray detection.

3 Result and Discussion

SEM-EDX test results for microstructure testing on waste wood pellet samples from Sengon sawmills. The results of the average EDX analysis of the elements contained in Sengon wood pellets can be seen in Table 1.

Table 1. Average composition of Sengon wood pellets

Material	Specimen	Wt (%)							
		CK	**OK**	**CuL**	**AIK**	**SiK**	**CIK**	**KK**	**CaK**
Wood	A	38.04	50.71	02.29	01.02	02.46	01.06	01.93	02.49
Pellet	B	42.00	50.25	01.59	00.69	02.34	00.19	01.30	01.66
Sengon	C	41.06	55.29	00.00	00.85	01.07	00.00	00.96	00.78
Average		40.36	52.08	1.29	0.85	1.95	0.42	1.39	1.64

Table 1. The average elemental composition of sengon wood pellets showed that the elemental composition from the results of EDX analysis, obtained the average element contained in sengon wood pellets, namely C-K elements (40.36%); elements O-K (52.08%); element CuL (1.29%); Elements of AIK (0.85%); SiK element (1.95%); CIK elements (0.42%); KK elements (1.39%) and CaK elements (1.64%) from the average test results, the largest percentage of OK elements was (52.08%) while the percentage of carbon (C) was a determinant of good wood pellet quality or not.

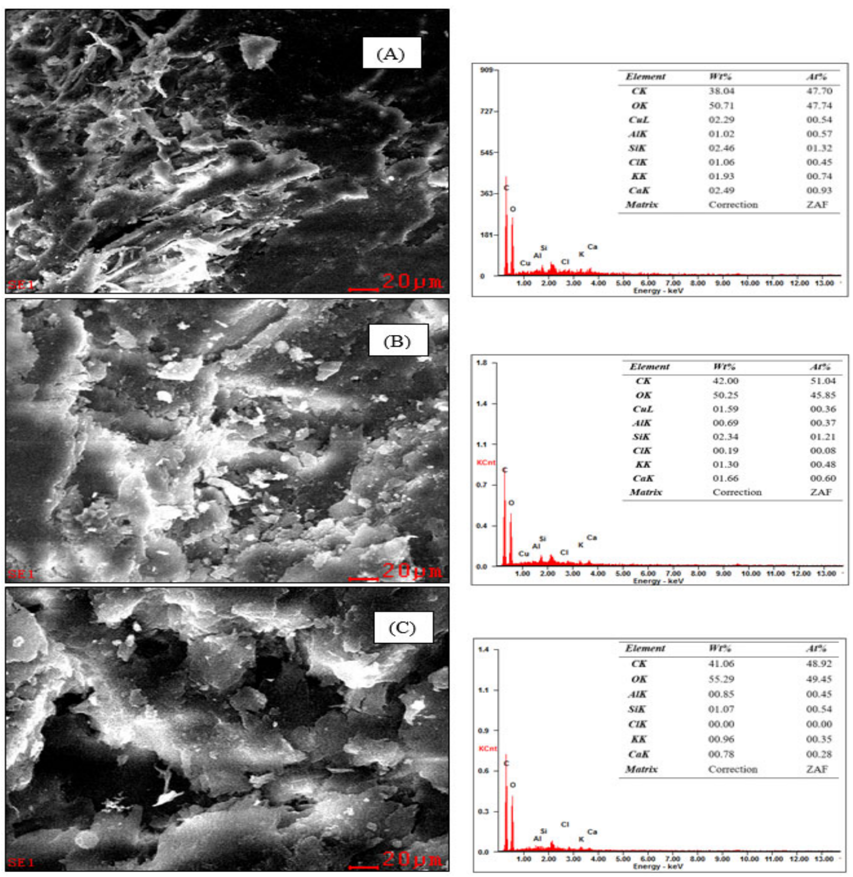

Figure 5. SEM-EDX micrograph on Sengon wood pellet taken three points: A, B, and C

Figure 5 showed the energy emission formed by Sengon wood SEM-EDX wood pellets taken at three points, those are A, B, and C, resulting in the atomic weight of the combined elements in which the highest percentage was OK (50.71%) on the graph of point A with an atomic percentage of 47,74%. The second-highest combined element was C-K (38.04%) with an atomic percentage of 47.70%. While at point B, the highest percentage was O-K (50.25%) followed by C-K (42%). Then at point C, the highest element was OK (55.29%) then followed by CK element (41.06%). In Figure 5, the three largest elements was in the OK element and followed by the most to -2, that was CK elements, then followed by the combined elements of Si-K, Al-K, Cu-L, Ca-K, KK, CL-K appeared with a small percentage. The energy emitted by the atoms in the wood pellet was the highest atom/element of C (carbon), followed by element O. There were radiant energy of several metals (Al, K, Ca), semi-metals (Si), and non-metals (Cl) but only in small quantities. Therefore, the carbon element was the highest composition of wood pellets as shown in Figure 6 as the results of SEM micrographs with different magnification scales.

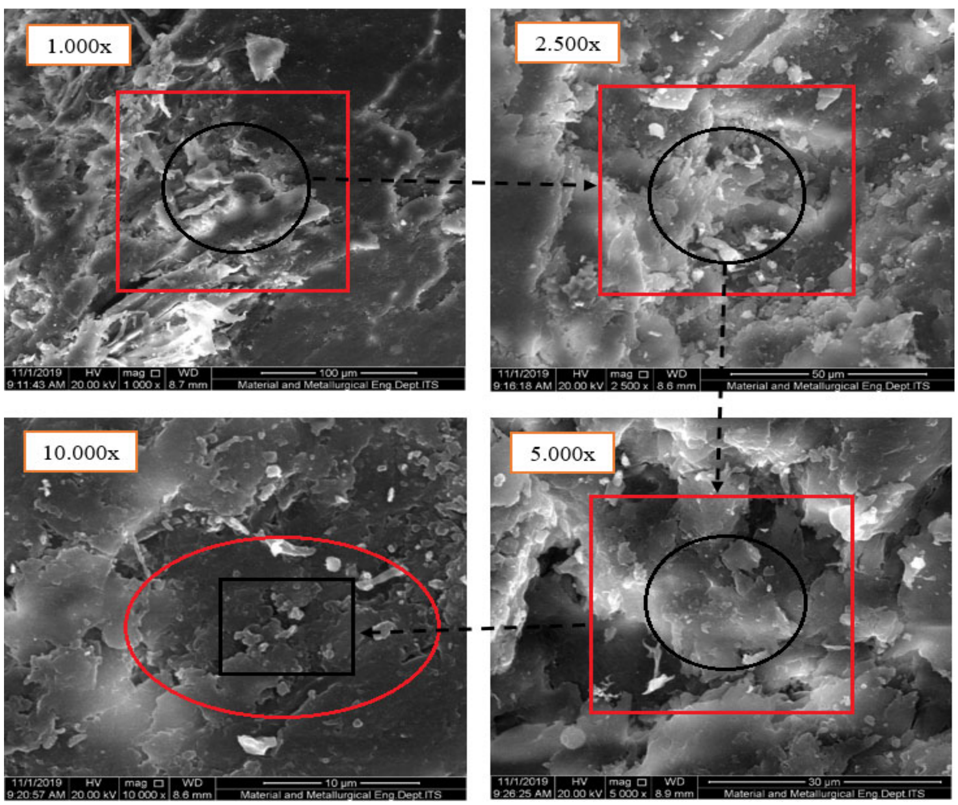

Figure 6. Results of SEM Micrographs with a magnification scale of 1,000X, 2,500x, 10,000 and 5,000x

In the SEM test in Figure 6, it can be seen the surface visualization of the object being tested, from the SEM photo it can be seen the surface of the Sengon wood pellets and their density. The results of the SEM analysis of the wood pellet's surface showed that there were many pores, where this wood pellet had high porosity due to the lack of pressure in the process of making wood pellets. In the process of making wood pellets, the pressure will be made higher so as to produce wood. the better pellets, because the more porosity the easier the compaction process and the better the quality later, where a lot of porosity provides a larger surface area for bonding, as evidenced by looking at the results of SEM Micrographs with a magnification scale of 1,000X, 2,500x, 10,000 and so on. Maximum 5,000x magnification was to see the pores on the top, middle, and bottom. The enlargement of the Sengon wood pellets pores in the middle was more than at the bottom and top because the middle was denser than the inside of Sengon wood pellets. The top had fewer pores as the outer part of the wood pellet had friction with the ring die on the wood pellet machine. on the wood pellet there is a metal element that looks white or bright which is probably a metal element. In addition, it also has an unequal pore size as the pelletizing process occured faster on the outer surface of the pellet, so this layer had much smaller and smoother pores than inside the pellet.

4 Conclusion

Based on the results of the preliminary study analysis using SEM-EDX micrographs on Sengon wood pellets, it was concluded that this wood pellet sample contained C-K elements (40.36%); elements O-K (52.08%); element CuL (1.29%); Elements of AIK (0.85%); SiK element (1.95%); CIK elements (0.42%); KK elements (1.39%) and CaK elements (1.64%). From the results of the average test, the largest percentage of elements was O-K (52.08%) where the percentage of carbon (C) was a determinant of a good quality wood pellet. While the results of SEM micrographs with a magnification scale showed that the pores were at the top, middle and bottom. Pores in the middle were more than at the bottom and top because it was denser than the inside of Sengon wood pellets. The top had fewer pores because the outer part of the wood pellet had frictions with the ring die so that the resulting pores were smoother.

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