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Characteristics Serpentine Aceh; Morphology and Chemical Compositions Studied by SEM-EDS and XRF

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Abstract. Carbon dioxide is a dangerous pollutant that harms the environment and triggers global warming, which causes greenhouse gases. Serpentine is a rock rich in magnesium silicate, which can be used to reduce carbon dioxide pollutants through adsorption technology. Aceh Province has the potential for serpentine rocks, which can be processed to adsorb carbon dioxide pollutants. This research examines the characteristics of serpentine based on its morphology and chemical composition as a carbon dioxide adsorbing material. The serpentine aceh was obtained from Indrapuri Regency, Aceh Province. The thermal activation stage is as follows: serpentine aceh is collected, cleaned, reduced in size, and dried, then serpentine aceh is ground to a size of 50 mesh (297–149 μm), 100 mesh (149–94 μm) and 150 mesh (94–74 μm). The serpentine aceh was thermally activated using a furnace at 750 ºC for 1.5 hours for all particle sizes. The characterization of serpentine aceh, which consists of morphological and chemical structure analysis using scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) and chemical composition using X-ray fluorescence (XRF). The results show that the characteristics of serpentine aceh before being activated have a lizardite and chrysotile structure with a chemical composition of SiO_2 38.83% and MgO 29.95%, and after being thermally activated, serpentine aceh has an antigorite structure with a chemical composition of SiO_2 45.42% and MgO 36.83% at 150 mesh. This research contributes to utilizing one of Aceh's natural mineral resources, serpentine rock, as an alternative CCS material to reduce greenhouse gas emissions.

Introduction

The province of Aceh is one of the areas with the potential for spreading serpentine rocks, especially in the territory of Aceh Besar District. The spread pattern of the rocks in the Aceh Besar Region forms a hill starting from Kuta Malaka district in the west direction towards the east, then in Indrapuri district to Kuta Cot Glie district [1]. The effect of greenhouse gases causes global warming due to CO_2 being a pollutant [2]. Increasing CO_2 concentrations in the atmosphere affect the ability of plants and oceans to assimilate CO_2, resulting in CO_2 being trapped in the earth's atmosphere. This phenomenon causes global warming, indicated by rising sea levels, climate change and increasing global temperatures. Excessive CO_2 releases from industrial activities, power plants and other sources have an essential influence on the global climate and the earth's life cycle [3]. Serpentine rock is a metamorphic rock formed due to changes in basalt rock on the seabed. Serpentine mineral content belongs to a group of silicate minerals called phyllosilicate. Serpentine is used as decoration and raw material in the mineral industry. Serpentine contains the minerals silica and calcite found in rock cavities [4]–[7]. Serpentine is a non-metallic mineral that is applied in engineering, such as adding building materials, carbon adsorption materials, and utilizing pure magnesium or amorphous silica through the extraction method [8], [9]. The characteristics of serpentine are green, brown, red and black; strength between 2.5-5; orthorhombic, monoclinic and hexagonal crystal forms. Density 2,5-2,6. Serpentines are derived from ultra-fine rocks that undergo hydrothermal alterations and alterations of minor elements such as nickel, chromium, cobalt, and scandium. The serpentine base crystal structure is a silicate layer connected with the octahedral layer [$MgO_2(OH)_4$] [10]. The constituent structures of serpentine minerals are antigorite [(Mg, Fe)$_3Si_2O_5(OH)_4$], Chrysotile [$Mg_3Si_2O_5(OH)_4$], Lizardite [$Mg_3Si_2O_5(OH)_4$], Orth chrysotile [$Mg_3Si_2O_5(OH)_4$], Para chrysotile [MgFe)$_3Si_2O_5(OH)_4$], Magnetite[F_3O_4] and Amphibole, pyroxene and talc[$Mg_3Si_4O_{10}(OH)_2$][11]-[13].

Potential large serpentine reserves will be applied in high technology, such as carbon capture and carbon storage processes, as a potential source of raw materials for nickel and magnesium in producing metals. Magnesium carbonate is found in an anhydrous form known as magnesite ($MgCO_3$) and is the most thermodynamically stable and efficient carbon storage medium [14]. When heated, serpentine will undergo several reaction stages, namely dehydration, dihydroxylation, and forsterite crystallization at temperatures above 700 ºC. Rock formation processes greatly influence the mineral content of serpentine. The activation process on serpentine aims to remove SiO_2 found on the serpentine surface, which is a barrier to the reaction between MgO and CO_2 [15]. Thermal and chemical activation of serpentine, where thermal activation uses high temperatures to open the pores of the material by removing chemical bonds or oxidizing elements or compounds found on the surface of the serpentine so that it can affect the adsorption performance of the adsorbent material. Thermal activity is aimed at expanding the volume and diameter of pores and can produce new pores. Several studies have evaluated the impact of particle size during thermal activation, indicating that halved materials provide higher yields. The thermal activation method can be carried out using H_2O, CO_2, O_2, and N_2. The serpentine must be activated first to improve the quality of serpentine as a Carbon Capture and Storage (CCS) material. Several studies show that thermally activated serpentine can increase the Mg content so that the CO_2 adsorption process can occur optimally [16], [17].

SEM-EDS is an instrument for analyzing microstructures and elements of various kinds of materials. SEM-EDS consists of two devices combined into one. SEM works to produce images of samples from surface scanning using an electron microscope at a certain scale that is magnified. The interaction between electrons and atoms in a sample will provide a signal containing information about the surface morphology as well as the topography and composition of the sample. EDS is used for elemental chemical analysis of materials. Characteristics are assessed based on the uniqueness of the atomic structure of an element that allows a series of peak spectrum to occur in electromagnetic emissions. XRF can analyze various elements, from sodium to uranium in various concentrations from percentage content to parts per million (ppm) in samples in solid or liquid. This analysis technique measures the elemental composition of metals, ceramics, and composite materials. This study compares the characterization of serpentine materials based on their morphology and chemical composition before and after activation. This research contributes to utilizing one of Aceh's natural mineral resources, serpentine rock, as an alternative CCS material to reduce greenhouse gas emissions.

Materials

The serpentine aceh (SA) was obtained from Indrapuri District, Aceh Province.

Methods. This research was carried out in two stages, namely, the thermal activation stage, which was performed through the following mechanism: Aceh serpentine was collected, cleaned, reduced in size using a ball mill, and dried using sunlight to reduce the water content. Aceh serpentine was ground to 50 mesh (297–149 µm), 100 mesh (149–94 µm), and 150 mesh (94–74 µm) using a mesh sieve. Aceh serpentine was then thermally activated using a furnace (Thermolyze Fb 1410M-33 capacity, 2.1 liters) at a temperature of 750 ºC for 1.5 hours. In the second stage, Aceh serpentine was characterized using scanning electron microscope-energy dispersive spectroscopy (SEM-EDS JEOL JSM-6510LA) to study its morphology and chemical structure and analyzed for its chemical composition using X-ray fluorescence (XRF Rigaku Supermini200). Characteristic testing was carried out on serpentine before and after activation.

Result and Discussion

The SEM-EDS characterization results of serpentine aceh are shown in Figure 1(a) before activation and Figure 1 (b, c, and d) after activation at sizes 50, 100, and 150 mesh, respectively. In contrast, the EDS results are shown in Figure 2 and Table 1. The results of serpentine morphology analysis before and after thermal activation are very different based on SEM-EDS results. Figure 1 shows the results of SEM analysis of serpentine aceh before and after activation at various particle sizes. From these four images, there has been a change in the morphological structure in images 1. b, 1. c., and 1.d (activated serpentine aceh), where it can be seen that the impurity elements that previously covered the serpentine aceh (Figure 1.a) have decreased. In figure 1.a, there are no visible pores, whereas in figure1.b, 1.c. and 1.d the pores are visible. serpentine aceh that has not been activated has a layered structure, while activated serpentine has structural destruction that is shown by the formation of agglomerates. The morphology of serpentine aceh before activation is a chrysotile structure, whereas after thermal activation at a temperature of 750 °C for 1.5 hours, there is a change in morphology with an antigorite structure. The antigorite structure is a structure that is rich in forsterite compounds (Mg_2SiO_4). The formation of the forsterite reaction occurs at 638 ºC – 700 ºC, and the dihydroxylation of lizardite crystal water occurs gradually under endothermic conditions. Serpentine is heated to temperatures above 700 ºC, producing serpentine rich in magnesium [10]. Other research results state that the three polymorphs of the serpentine group are antigorite, kadalite, and chrysotile. According to a study, lizards are very fine-grainy and flat, chrysotile is fibrous, and antigorite is made up of grooved plates or fibres [18]. According to other studies, chrysotile is a fibrous variant, whereas antigorite and kadalite are thick or fine-grained. In micrographs, serpentine-group minerals are seen as layers and parallel aggregates with a fibrous arrangement of long rods [19], [20]. Activated serpentine has a larger pore volume than serpentine that has not been activated [16].

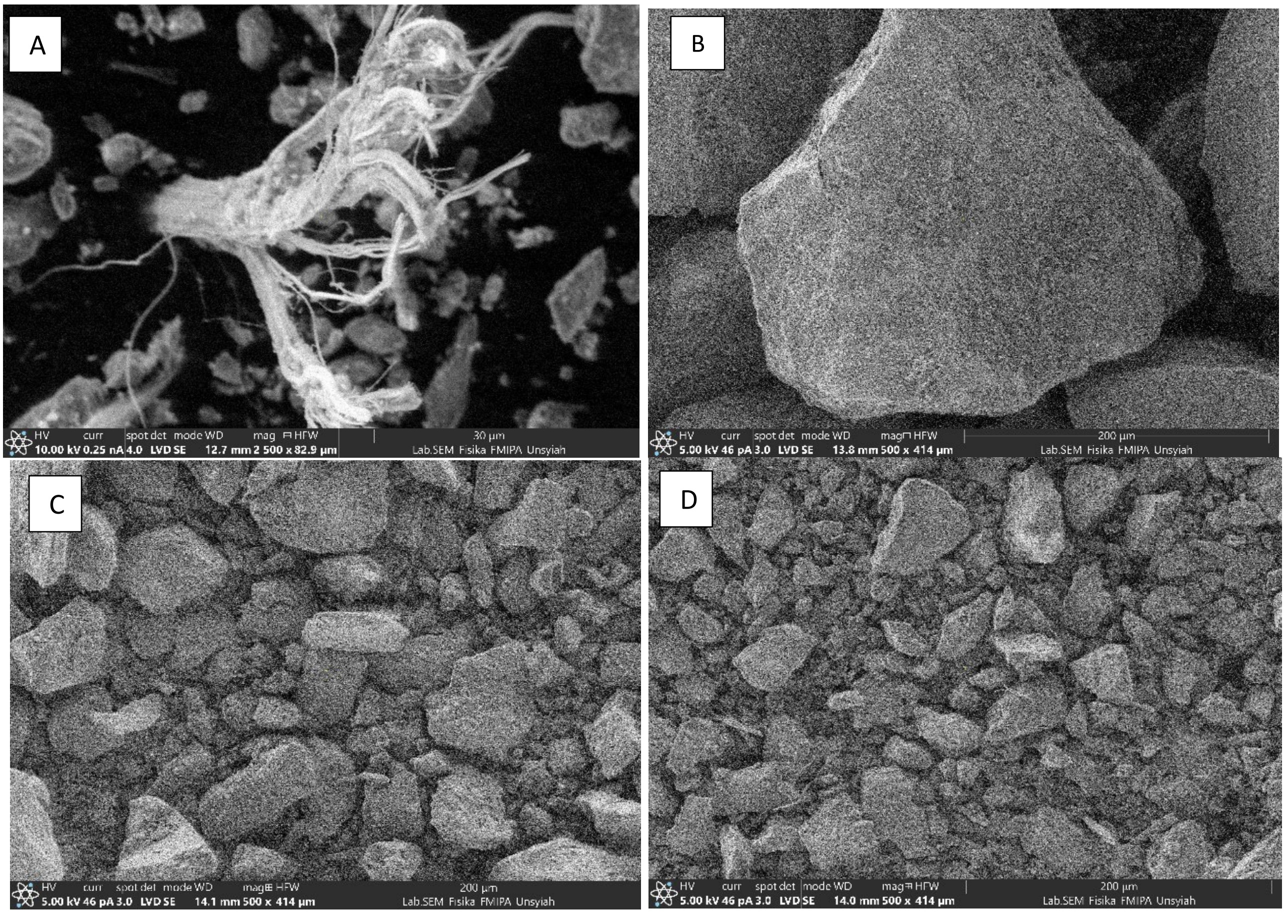

Fig. 1. SEM analysis at (a) SA pre activated. (b) SA activated at particle sizes 50 mesh. (c) SA activated at particle sizes 100 mesh. (d) SA activated at particle sizes 150 mesh

Figure 2 and Table 1 show that the magnesium element in serpentine aceh has changed after activation. Before activation, the magnesium element content was 9%; after activation, at 50 mesh, it was 23.4%; at 100 mesh, it was 25.7%; and at 150 mesh, it was 24.7%. This is because in the thermal activation process, the carbon element, one of the largest elements contained in serpentine aceh, is decomposed to increase the magnesium element in the activated serpentine aceh. Serpentine aceh also contains the elements oxygen, silica, aluminum, and sulfur in different percentages (Table 1).

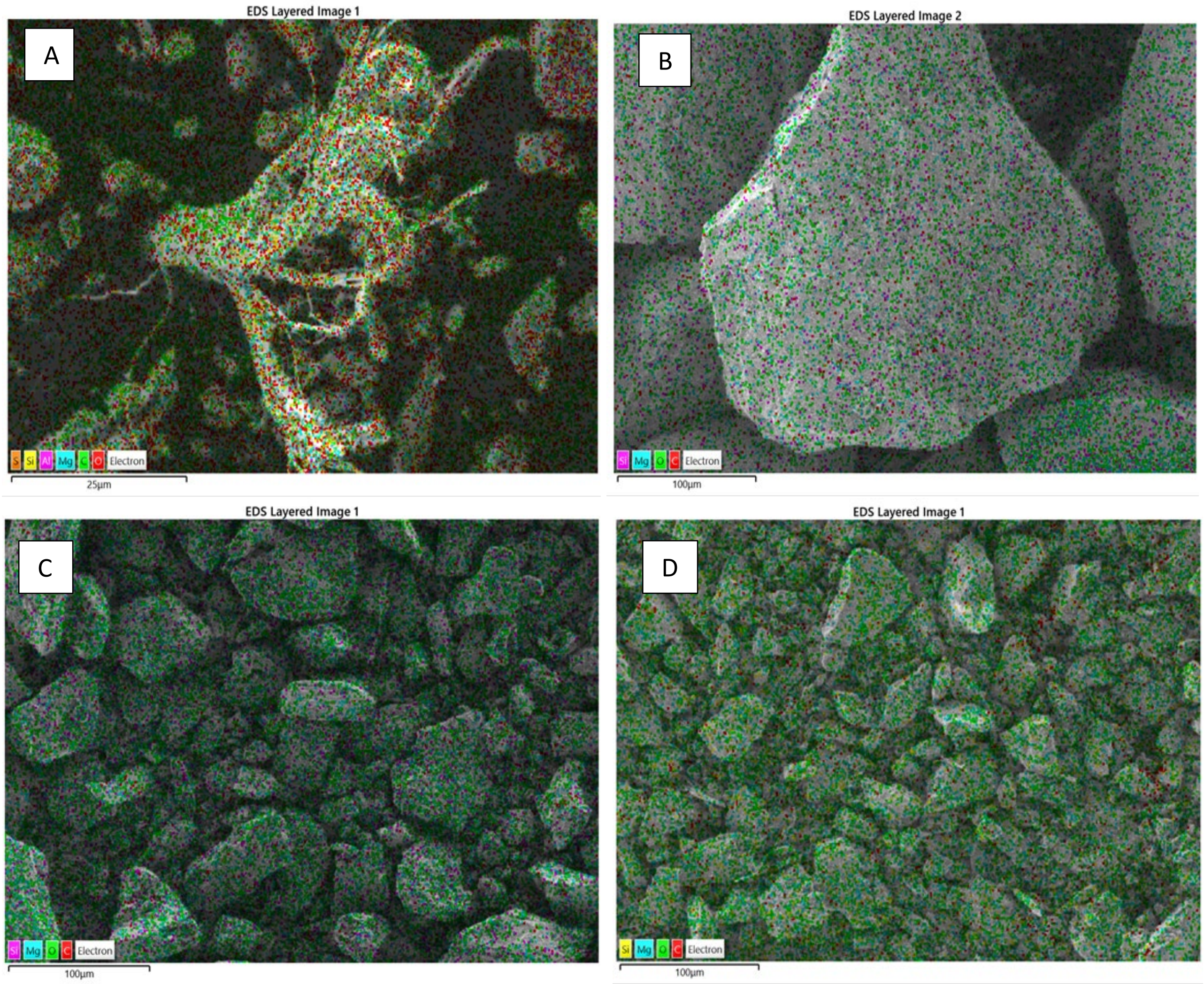

Fig. 2. EDS analysis at (a) SA pre activated (b) SA activated at particle sizes 50 mesh (c) SA activated at particle sizes 100 mesh (d) SA activated at particle sizes 150 mesh

Table 1. SA mineral compound composition pre activated and activated based on EDS analysis

Mineral	SA pre activated (%)	SA activated at particle sizes 50 mesh (%)	SA activated at particle sizes 100 mesh (%)	SA activated at particle sizes 150 mesh (%)
C	43.6	6.8	3.4	6.5
O	38.9	49.1	48.3	48.1
Mg	9.0	23.4	25.7	24.7
Si	7.0	19.7	22.6	20.7
Al	0.8	1.0	0	0
S	0.7	0	0	0

Table 2 shows the mineral composition of serpentine aceh pre activated and activated based on XRF analysis. The composition of serpentine aceh before and after activation shows a very different difference in MgO content. Serpentine aceh contains 29.95% MgO pre activated, and activated the MgO content increases by 36.63%, 36.78% and 36.83% in size particles of 50, 100, and 150 mesh, respectively. The smaller the particle size of serpentine aceh, the higher the MgO content. MgO is the most important compound in a material that adsorbs CO_2 because it can react with CO_2 to make Mg_2CO_3 [21], [22]. The majority of serpentine aceh contain high percentages of MgO and SiO_2. Serpentine aceh also contains other minerals, namely Fe_2O_3, CaO, Al_2O_3, Cr_2O_3, Mn_2O_3, SO_3, MgO, Na_2O, K_2O, TiO_2 and P_2O_5.

Table 2. Aceh serpentine compound composition pre activated and activated based on XRF analysis

Mineral	SA pre activated (%)	SA activated at particle sizes 50 mesh (%)	SA activated at particle sizes 100 mesh (%)	SA activated at particle sizes 150 mesh (%)
SiO_2	38.83	45.41	45.28	45.42
MgO	29.95	36.63	36.78	36.83
Fe_2O_3	7.35	9.06	8.58	8.72
CaO	0.85	1.21	1.05	1.04
Al_2O_3	0.94	1.16	0.99	1.08
Cr_2O_3	0.36	0.43	0.34	0.29
Mn_2O_3	0.06	0.09	0.09	0.10
SO_3	0.05	0.05	0.03	0.06
Na_2O	0.04	0.03	0.01	0.03
K_2O	0.04	0.04	0.04	0.03
TiO_2	0.04	0.03	0.02	0.02
P_2O_5	0.03	0.07	0.05	0.04
*LOI	17.11	1.69	1.85	2.34

** LOI = loss on ignition*

Conclusion

The characteristics of the serpentine aceh have been studied using SEM-EDS and XRF to analyze the morphology and chemical composition of materials. Aceh serpentine has a morphology of chrysotile (pre activated) and antigorite (activated). The three highest compound contents found in aceh serpentine are MgO, SiO_2 and Fe_2O_3. Thermal activation at a temperature of 750 °C and 1.5 hours can increase the MgO content ≥ 36%. The level of MgO content that increases after activation is expected to become an alternative material for the capture and storage of CO_2, a source of emissions that cause the greenhouse effect. Although more research needs to be done on adsorption tests to find out how effective and efficient the Aceh serpentine material is, this will help find the best thermal activation treatment in terms of temperature, pressure, and flow rate.

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