Indonesian Physical Review

Volume 6 Issue 3, September 2023 P-ISSN: 2615-1278, E-ISSN: 2614-7904

Physicochemical Properties Analysis of Local Ceramics with Activated Carbon Additive Based on Rubber Fruit Shells as Thermal Insulators

Chandra Kurniawan1*, Elfi Yuliza1, Irkhos¹ , Dyah Fitriani²

¹Physics Department, Faculty Mathematics and Natural Sciences, University of Bengkulu, Indonesia ²Chemistry Department, Faculty Mathematics and Natural Sciences, University of Bengkulu, Indonesia

Corresponding Authors E-mail: chandra_kurniawan@unib.ac.id

Article Info Abstract

Article info: Received: 14-03-2023 Revised: 22-06-2023 Accepted: 24-06-2023

Keywords:

Insulator; Porous Ceramic; Rubber Fruit Shells, Clay; Activated Carbon

How To Cite:

C. Kurniawan, E. Yuliza, Irkhos, and D. Fitriani "Physicochemical Properties Analysis of Local Ceramics with Activated Carbon Additive Based on Rubber Fruit Shells as Thermal Insulators", Indonesian Physical Review, vol. 6, no. 3, p 274-283, 2023.

DOI:

https://doi.org/10.29303/ip r.v6i3.232

Many building materials that function as insulators to maintain indoor temperature stability during hot weather have been developed. One of them is porous ceramics applied to building walls as heat absorbers. Research related to the manufacture of porous ceramics began to emerge to find the optimal composition when applied as an insulator that can absorb heat well. In general, porous ceramics use activated carbon as a pore agent. Activated carbon can be obtained by carbonating agricultural wastes such as coconut shells, hazelnut shells, rubber fruit shells, rice husks, and cocoa shells. Research continues to develop in finding the best-activated carbon base material according to the required material characteristics. Researchers will take advantage of the abundant potential of local natural resources. The method used to make porous ceramics is to mix clay and activated carbon with a clay-carbon composition ratio of 100:0, 90:10, 80:20, 70:30, 60:40, and 50:50. The sample is made by mixing the two materials and then printed by dry pressing method with a load of 5 tons held for 10 minutes. After the sample is dried at room temperature, it is sintered at 900oC with a holding time of 3 hours. Next, the sample is characterized by determining density, porosity, and water absorption and then tested for thermal properties. After that, the surface morphology and elements in the sample are seen. The optimum composition of clay-carbon mixture is 70:30 grams. Ceramics using activated carbon from rubber fruit shells are suitable as building construction materials as heat insulators because they can absorb heat up to 500oC.

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Introduction

Indonesia is one of the countries with a tropical climate because of its existence on the equator which causes it to have a reasonably hot air temperature. The construction of high-rise buildings in various regions of Indonesia and the reduced amount of green open land also contribute to the increase in air temperature [1]. Urban construction materials also reflect high heat, and radiation from this reflection is partially retained by building walls, roofs, and others.

One way to keep the room temperature in a building stable is to use a material like an absorbent wall. Absorption walls function as heat reducers that reach the plane's surface and block heat from entering the building [2].

One of the building materials that can be used as absorption walls is ceramic. Ceramics is a combination of metal and non-metal materials. The atoms that makeup ceramics have powerful bonding forces in the form of ionic and covalent bonds. As a result, ceramics have a very high melting point. Ceramic raw materials generally consist of kaolin, clay, feldspar, and quartz [3].

Muara Bangkahulu District is located at an altitude of approximately 20 meters above sea level with the condition of a geological unit composed of clay, conglomerates of various materials, breccias, reef limestone, tuffaceous claystone, pumice, quartz sand, and excreted wood which are part of the Bintunan formation [4]. The potential of natural resources in geological conditions is sufficient to support business development in manufacturing building materials in the form of ceramics and bricks.

Several studies have been conducted regarding the manufacture of porous ceramics. The manufacture of porous ceramics with variations of zeolite and the addition of activated coconut shell charcoal was carried out and showed increased porosity and sample absorption [5]. The more rice husk additives, the larger the pore size and porosity of the membrane, but the smaller the surface area [6]. The strength of ceramics itself is affected by the sintering temperature [7][8], regarding the impact of the sintering process of ceramic materials on their mechanical properties, showing the best bending strength at 1250°C.

Rubber fruit shells have not been used optimally, and sometimes they even become waste with no selling value [9]. The addition of activated carbon in the raw material for making ceramics aims as a pore-forming agent [10][5][6]. The resulting porous ceramics will be characterized and tested for their physical and thermal properties to see their feasibility when applied as a thermal insulator.

Experimental Method

Activated Carbon and Clay Preparation

The sample preparation is a mixture of clay and activated carbon of rubber fruit shells in a specific ratio. Preparation is done on clay and rubber fruit shells. The material is washed with water, then dried in the sun. Rubber fruit shells that have dried and then burned. Clay and rubber fruit shells were activated with 10% phosphoric acid solution for 24 hours. After activation, it is rinsed with distilled water until the pH of the rinse water is neutral[11].

The next step is to dry the materials using an oven at 110oC for 2 hours. The dried clay and carbon were crushed using a mortar and sifted through a 100-mesh sieve. Powdered clay and carbon were mixed with a clay-carbon ratio of 100:0, 90:10, 80:20, 70:30, 60:40, and 50:50, drop by drop of distilled water was added and then stirred until homogeneous using a mortar.

Ceramics Manufacture

The material that has been stirred homogeneously is then put into a mould with dimensions of length x width x height 6 cm x 6 cm x 3 cm and then pressed with a hydraulic press at a pressure of 5 tons and held for 10 minutes. The calcination of the sample will be carried out after the manufactured sample is dry at room temperature. The last step of the preparation is to calcine the sample with a sintering temperature of $900\textdegree C$ with a holding time of 3 hours.

Physical Properties and Characterizations

The physical properties of the sample were determined by calculating density, porosity, and water absorption. The density ρ of a substance is expressed by the amount of substance m_k per unit volume V_t .

$$
\rho = \frac{m_k}{v_t} \tag{1}
$$

Porosity is the number of pores in a material that is calculated by finding the percentage (%) based on the material's water absorption and the ratio of the absorbed volume to the total volume of the sample. It is necessary to measure the wet mass m_b of the sample and the dry mass m_k of the sample. The density of water is expressed by ρ_w

$$
\%Porosity = \frac{(m_b - m_k)}{\rho_{air} \times V_t} \times 100\% \tag{2}
$$

Water absorption is the process by which particles are trapped in the material structure and become part of the material. Water absorption is proportional to porosity. The greater the porosity value, the greater the water absorption value. The water absorption test procedure refers to ASTM C-20-00-2005.

$$
\%Water \, abs = \frac{(m_b - m_k)}{m_k} \times 100\% \tag{3}
$$

Differential Thermal Analysis (DTA) is an analytical technique used to measure changes in heat content by continuously recording the temperature difference between the sample tested inert comparison material as a function of temperature change. DTA characterizes a material based on the material's response to temperature [11]. The temperature difference that occurs is recorded during the heating and cooling process. It is then displayed in the form of an enthalpy curve.

Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) is a tool that can form surface images and seeing element content. SEM-EDX uses signals generated by electrons which are then reflected or called secondary beams. The main principle of SEM-EDX is that the electron beam is directed at the surface points of the specimen. The surface structure of a test object can be studied much easier directly.

Result and Discussion

Density

To determine the density of the sample, the mass and volume of the sample were measured after calcination. After the calculation, a graph of the relationship between the sample's density and the clay-carbon composition is obtained as shown in Figure 1. Figure 1 shows a decrease in density as the amount of carbon increases. The highest density is shown by clay-carbon with a ratio of 100:0 which is 2243.73 kg/m³ while the lowest density is shown by clay-carbon with a ratio of 50:50 which is 572.11 kg/m³. Density values that decrease along with the addition of activated carbon are also shown in research conducted [12] where the addition of 30% activated carbon will reduce density compared to the addition of 10% activated carbon.

Figure 1. Relationship between the sample's density and the clay-carbon composition

Porosity

The porosity percentage of the sample was determined from the measurement of the volume and mass of the sample before and after calcination. After calculating the porosity percentage, a graph of the relationship between sample porosity and clay-carbon composition was obtained as shown in Figure 2.

Figure 2. Relationship between the sample's porosity and the claycarbon composition

Figure 2 shows an increase in porosity percentage along with the addition of activated carbon. The lowest porosity is shown by the clay-carbon mixture with a ratio of 100:0, which is 26.59%. The highest porosity is shown by the clay-carbon mixture with a ratio of 50:50, which is 68.91%. This is due to the carbon content that melts during the high-temperature combustion process, leaving pores and increasing oxygen levels in the sample. The same thing also happened in previous research where the composition of 50% activated carbon had the highest porosity [13][14].

Water Absorption

The percentage of water absorption is determined by measuring the wet mass and dry mass of the sample. The percentage of water absorption is directly proportional to the percentage of shrinkage of the sample mass. The higher the shrinkage percentage of the sample mass, the higher the water absorption percentage, and vice versa. The graph of the relationship between water absorption and clay-carbon composition can be seen in Figure 3.

Figure 3. Relationship between the sample's water absorption and the clay-carbon composition

Thermal Properties

Characterization of thermal properties was carried out using TGA-DTA. The temperature increase was set at $5^{\circ}C/m$ inute up to 800oC. The TGA-DTA graph is shown in Figure 4.

Figure 4. TGA-DTA graph of clay-carbon ceramic with 70:30 composition

Figure 4 shows a decrease in the mass percentage of the sample by 2.74% or equivalent to 0.28 mg when heated to 800 \circ C. At temperatures of 60 \circ C to 89 \circ C, there is evaporation of water in the ceramic, then at 200° C there is decomposition of kaolinite and at 510° C there is decomposition of carbonates. This phenomenon also occurred in research conducted [15] on the manufacture of clay-zeolite composites calcined at a maximum temperature of 900 °C. Ceramic samples experience stability in mass shrinkage percentage at temperatures from 290° C to 500° C, this indicates that the local clay-carbon mixture ceramic is suitable for application in building construction walls.

Surface Morphology

Characterization of the surface morphology of the samples was carried out using SEM Hitachi S4800 instrument (Japan). Samples were taken as less than 3 grams. Objects were enlarged at magnifications of 1000 times, 3000 times, 5000 times, and 10,000 times. After the images were obtained, a comparison of clay samples without mixture with clay-carbon mixture with a composition of 70:30 as shown in Figure 5.

 (a) (b) **Figure 5**. SEM image sample (a) pristine clay (b) clay-carbon with 70:30 composition

Figure 5 shows that the pores in the clay-carbon mixture have not formed completely. This could be due to the inhomogeneous mixing of activated carbon. However, it can be seen that the clay without a variety does not form pores.

Elements Contained

Elemental content is carried out using EDX Hitachi S4800 instrument (Japan). In addition to seeing the elemental content, the mapping of elements contained in ceramics can also be shown. Elements contained in the sample are displayed in weight percent (Wt%). Elemental content in ceramics can be seen in Figure 6.

The most elemental content in ceramics is the element C with a weight percent of 37.9% and the element O due to the pores formed in the sample. The least element or almost decomposed during combustion is the element K. Other elements that fulfill the ceramic composition are Si, Al, Fe, and P. Mapping these elements can be seen in Figure 7

Figure 6. Elemental content in 70:30 composition clay-carbon ceramics

Figure 7. Mapping elements contained in 70:30 composition clay-carbon ceramics

The clay-carbon ceramic samples were also characterized using X-ray diffraction. X-ray diffraction is a technique used to identify the presence of crystalline phases in materials and powders, and to analyze the structural properties of each phase. This method uses a diffracted X-ray beam such as a ray reflected from each plane, successively formed by the crystalline atoms of the material. With various angles of incidence, the diffraction patterns formed express the characteristics of the sample. This arrangement is identified by comparison with an international database. Figure 8 shows almost the same diffraction pattern for each claycarbon composition. The difference is the intensity. The 50:50 clay-carbon composition shows the greatest intensity. The carbon content amplifies the diffraction of other elements in the ceramic sample.

Figure 8. X-ray Diffraction Patterns of Clay-Carbon Ceramic Samples of each Composition

Conclusion

The optimum composition of clay-carbon mixture is 70:30 grams. The addition of activated carbon affects the physical properties and the formation of ceramic pores. The more the addition of activated carbon, the higher the percentage of porosity and water absorption, but inversely proportional to the lower density. TGA-DTA analysis shows that the ceramic samples experience a stable percentage of mass shrinkage at temperatures from 290° C to 500°C, indicating that this local clay-carbon mixture ceramic is suitable for application in building construction walls.

Acknowledgment

Authors gratefully acknowledge to LPPM University of Bengkulu for supporting this Internal Funding "PNBP Universitas Bengkulu 2022"with contract number 1780/UN30.15/PP/2022. In addition, the authors thank to Mingchi University of Technology for TGA-DTA, XRD, and SEM-EDX characterizations.

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