JOURNAL OF ENERGY, MATERIALS, AND INSTRUMENTATION TECHNOLOGY

Journal Webpage https://jemit.fmipa.unila.ac.id/

Effect of Magnesium Oxide (MgO) Mass Addition, Temperature and Holding Time on the Characteristics of Composite Ceramics from Coal Bottom Ash, Waste Aluminum Cans and Bittern Water

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Article Information

Article history:
Received March 17, 2024
Received in revised form
July 4, 2024
Accepted August 8, 2024

Keywords: *Ceramics, coal bottom ash, MgO, sol-gel and solid state*

Abstract

The synthesis of silica and alunima from coal bottom ash and aluminum can waste has been carried out as the basic material for making composite ceramics. The purpose of this study is to analyze the chemical characteristics of silica (SiO_2) and alumina (Al_2O_3) produced by coal bottom ash and aluminum can waste as a base material for making composite ceramics, analyze the physical characteristics of composite ceramics produced and analyze the quality of composite ceramics synthesis with composite ceramics factory base material. Coal bottom ash was synthesized using the sol-gel method. The method for making ceramics is solid state with variations in the addition of 0, 10, 15% wt MgO, sintering temperature of 800 – 1.100˚C and varying holding times of 2, 3 and 4 hours. Physical testing included density, porosity and absorption, while chemical testing uses XRF (X Ray Fluorescence) and compressive strength tests use UTM (Universal Testing Mahine). The results of the characterization silica extraction obtained a value of 43.51% and alumina of 31.01%. The results of the physical tests obtained optimum samples, namely density (1.42 g/cm_3), porosity (45.66%), adsorption (32.11%) and compressive strength (2.37 Mpa). The quality of the optimum sample is still below the factory material sample.

Informasi Artikel

Proses artikel:
Diterima 17 Maret 2024
Diterima dan direvisi dari 4 Juli 2024
Accepted 8 Agustus 2024

Kata kunci: *Abu dasar Batubara, keramik, MgO, sol-gel dan solid state*

Abstrak

Telah dilakukan sintesis silika dan alunima dari abu dasar batubara dan limbah kaleng aluminium sebagai bahan dasar pembuatan keramik komposit. Tujuan dari penelitian ini yaitu menganalisis karakteristik kimia dari silika (SiO_2) dan alumina (Al_2O_3) yang dihasilkan abu dasar batubara dan limbah kaleng aluminium sebagai bahan dasar pembuatan keramik komposit, menganalisis karakteristik fisik keramik komposit yang dihasikan dan menganalisis kualitas keramik komposit bahan sintesis dengan keramik komposit bahan dasar pabrik. Abu dasar batubara disintesis dengan metode sol-gel. Metode dalam pembuatan keramik adalah solid state dengan variasi penambahan MgO 0, 10, 15% wt, di-sintering pada suhu 800 – 1.100˚C dan variasi waktu tahan 2, 3 dan 4 jam. Pengujian fisis meliputi densitas, porositas dan absorpsi, sedangkan pengujian kimia menggunakan XRF (X Ray Fluorescence) dan uji kuat tekan menggunakan UTM (Universal Testing Machine). Hasil dari karakterisasi ekstraksi silika diperoleh nilai sebesar 43,51% dan alumina sebesar 31,01%, Hasil analisis fasa pada sampel optimum terbentuk fasa spinel, albite dan corundum. Hasil uji fisis diperloleh sampel optimum yaitu densitas sebesar 1,42 g/cm^3, porositas sebesar 45,66%, adsorpsi sebesar 32,11% dan kuat tekan sebesar 2,37 MPa. Kualitas dari sampel optimum masih di bawah sampel bahan pabrik.

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1. Introduction

Ceramics are inorganic materials composed of compounds such as clay, feldspar, and quartz that undergo high-temperature heat treatment. One type of ceramic known for its excellent thermal resistance is cordierite ceramic. Cordierite is formed from silica (SiO_2), alumina (Al_2O_3), and magnesium oxide (MgO), with the chemical formula $Mg_2Al_4Si_5O_{18}$ or $2MgO·2Al_2O_3·5SiO_2$. Cordierite is rarely found in nature and belongs to a group of silicate minerals with diverse crystal structures (Kingery et al., 1976).

Various methods have been developed to synthesize ceramic composites, including the solid-state reaction method and the sol-gel method. Since cordierite does not occur abundantly in nature, it must be synthesized from raw materials containing MgO, Al_2O_3, and SiO_2. Commercial silica is commonly used as a silica source; however, its relatively high cost encourages the exploration of alternative raw materials. Several studies have reported the utilization of waste materials rich in silica, such as rice husk ash (Sembiring, 2015) and coal fly ash (Retnosari, 2013), as substitutes for commercial silica.

Coal combustion residues, particularly bottom ash, have significant potential as a silica source. Previous studies reported that coal ash contains silica that can be extracted and utilized as a ceramic raw material (Retnosari, 2013). In addition, coal-fired power plants generate large amounts of fly ash and bottom ash. Data from Tarahan Steam Power Plant (PLTU Tarahan) indicate that approximately 58–64 tons of fly ash and bottom ash are produced daily (Aditama et al., 2022). The utilization of bottom ash as a raw material can therefore reduce industrial waste accumulation while increasing its economic value.

Besides silica, alumina is another essential component in cordierite ceramic synthesis. In this study, aluminum can waste was utilized as an alternative alumina source. Aluminum cans are widely used because they are lightweight, non-toxic, odorless, possess good thermal properties, and can be recycled efficiently (Manurung, 2019). Previous studies demonstrated that alumina can be extracted from aluminum can waste through the sol-gel process (Yudhistia et al., 2018). Therefore, aluminum can waste has the potential to serve as a sustainable alternative raw material for ceramic production.

Magnesium oxide (MgO), the third major component in cordierite ceramics, was obtained from bittern water. Bittern water is a concentrated liquid waste generated during salt production and contains various dissolved minerals, including magnesium compounds (Raesta et al., 2017). In this study, MgO derived from bittern water was prepared based on the method reported by Sanjaya (2023).

Several studies have investigated ceramic synthesis using waste-derived materials. For example, Cibro and Mora (2020) produced ceramics using volcanic ash from Mount Sinabung as the primary raw material. In contrast, the present study utilizes three different waste materials, namely coal bottom ash, aluminum can waste, and bittern water, as sources of SiO_2, Al_2O_3, and MgO for the synthesis of composite ceramics.

The composite ceramics were synthesized using the solid-state method by mixing silica, alumina, and MgO extracted from the respective waste materials. The ceramic samples were then sintered at temperatures ranging from 800°C to 1100°C with different holding times. The effects of MgO addition, sintering temperature, and holding time on the characteristics of the resulting composite ceramics were evaluated. Chemical characterization was performed using X-ray fluorescence (XRF) to determine elemental composition, while physical characterization included density, porosity, water absorption, and compressive strength measurements. The results were subsequently compared with those of commercial ceramic materials to evaluate the quality of the synthesized composite ceramics.

2. Research Methods

This study consisted of three main stages: (1) silica extraction from coal bottom ash, (2) alumina extraction from aluminum can waste, and (3) fabrication of composite ceramic samples.

2.1 Materials and Equipment

The equipment used in this study included a mortar and pestle, digital balance, sieve, pellet mold, oven, vernier caliper, glass containers, filter paper, overhead stirrer, hotplate magnetic stirrer, hydraulic press, furnace, pH indicator paper (pH 0–14), spatula, thermometer, Erlenmeyer flask, and funnel. The raw materials used were coal bottom ash, aluminum can waste, magnesium oxide (MgO), distilled water, sodium hydroxide (NaOH, 4 M), hydrochloric acid (HCl, 6 M and 37%), 70% ethanol, and sodium bicarbonate ($NaHCO_3$).

2.2 Extraction of Silica (SiO_2) from Coal Bottom Ash

Silica was extracted from coal bottom ash using the sol-gel method based on the procedure reported by Cibro and Mora (2020). Initially, the bottom ash was sieved using a 100-mesh sieve. A total of 50 g of sieved bottom ash was mixed with 60 mL of 10% NaOH solution and heated at 100°C while stirring using a hotplate magnetic stirrer and overhead stirrer for 120 minutes to produce a silica sol. Subsequently, 250 mL of distilled water was added to form a sodium silicate (Na_2SiO_3) solution. The sodium silicate solution was then titrated with 6 M HCl while continuously stirred at 100°C until the pH reached 7, resulting in the formation of a white silica gel.

The resulting gel was washed three times using 300 mL of distilled water to remove residual impurities. The precipitate was separated by filtration using filter paper and then dried in an oven at 110°C for 2 hours. The dried silica was subsequently ground using a mortar and pestle to obtain fine silica powder.

2.3 Extraction of Alumina (Al_2O_3) from Aluminum Can Waste

Alumina was extracted from aluminum can waste through a precipitation process. To produce approximately 10 g of Al_2O_3, 5.29 g of aluminum, 56.6 mL of 31.45% HCl solution, and 35 g of sodium bicarbonate were used. The aluminum can waste was dissolved in 37% HCl solution to form aluminum chloride solution. The resulting solution was filtered to remove insoluble impurities. Sodium bicarbonate was then added gradually to the filtrate to form a precipitate. The precipitate was washed repeatedly with distilled water and allowed to settle. After separation, additional distilled water was added and the suspension was stirred to obtain a purer precipitate. The precipitate was then filtered and dried in an oven at 120°C for 2 hours to produce alumina solids. Finally, the dried alumina was ground using a mortar and pestle to obtain fine Al_2O_3 powder.

2.4 Preparation of Composite Ceramic Samples

Composite ceramic samples were prepared using the solid-state method following the procedure reported by Cibro and Mora (2020). Silica (SiO_2), alumina (Al_2O_3), and magnesium oxide (MgO) powders were weighed and mixed at a mass ratio of 50:35:15, respectively. The mixed powder was sieved and subsequently dispersed in 60 mL of 70% ethanol. The suspension was stirred using a magnetic stirrer at 120 rpm for 3 hours to ensure homogeneous mixing. After stirring, the mixture was dried in an oven at 100°C for 2 hours and then ground into a fine powder.

In this study, MgO was added at concentrations of 0 wt%, 10 wt%, and 15 wt% relative to the total mass of the composite ceramic. The powder mixture was then placed into a stainless-steel pellet mold and compacted using a hydraulic press under a load of 3 tons to form ceramic pellets. The green pellets were sintered in a furnace at temperatures ranging from 800°C to 1100°C with a heating rate of 5°C/min. Holding times of 2, 3, and 4 hours were applied to evaluate the effects of sintering conditions on the characteristics of the composite ceramics.

3. Results and Discussions

3.1 XRF results of Silica (SiO_2) from Coal Bottom Ash

Coal bottom ash was successfully extracted into SiO_2 by sol gel method. XRF results of silica extraction can be seen in **Table 1**. Based on the results of the XRF analysis, the SiO2 content was 43.51%. From the XRF results, the chemical composition of Cl is 47.36%, SiO2 is 43.51%, SO3 is 4.50%, K_2O is 2.35%, Al_2O_3 is 1.28%, P_2O_5 is 0.50% and CaO is 0.34%. Based on the XRF results, the highest value is the composition of Cl amounting to 47.36%, this occurs because the silica extraction procedure uses 37% HCl for gel formation in the sol gel method.

Table 1. SiO_2 component composition of XRF results.

No	Chemical Composition	Synthesis Results (%)	Factory Material (%)
1.	Cl	47,36	-
2.	SiO_2	43,51	95,81
3.	SO_3	4,50	0,86
4.	K_2O	2,35	-
5.	Al_2O_3	1,28	2,14
6.	P_2O_5	0,50	0,65
7.	CaO	0,34	0,21
8.	TiO_2	-	0,19

3.2 XRF results of Alumina (Al_2O_3) from Waste Aluminum Cans

Waste aluminum cans can be extracted into Al_2O_3 by sol gel process. The XRF results of alumina extraction can be seen in **Table 2.** Based on the results of the XRF analysis, the Alumina (Al_2O_3) content was 31.01%. From the XRF results, the chemical composition of Cl is 65.03%, Al_2O_3 is 31.01%, Fe_2O_3 is 1.17%, SO_3 is 0.95%, CaO is 0.58%, P_2O_5 is 0.50%, MnO is 0.34% and SiO_2 is 0.24% Based on the XRF results, the highest value is the composition of Cl amounting to 65.03%, this happens because the Alumina extraction procedure uses 37% HCl to dissolve aluminum can waste in the sol gel method.

Table 2. Al_2O_3 Component composition of XRF results.

No	Chemical Composition	Synthesis Results (%)	Factory Material (%)
1.	Cl	65,03	-
2.	Al_2O_3	31,01	98,63
3.	Fe_2O_3	1,17	-
4.	SO_3	0,95	-
5.	CaO	0,58	0,29
6.	P_2O_5	0,50	0,98
7.	MnO	0,34	-
8.	SiO_2	0,24	-

3.3 XRF Results of Composite Ceramics

The XRF characterization results of each ceramic sample are presented in **Table 3**. Samples 1, 2, and 3 are the addition of MgO mass with variations of 0%, 10% and 15% of the ceramic mass. Samples 4, 5 and 6 are temperature variations with temperatures of 800°C, 900°C and 1100°C. Samples 7, 8 and 9 are variations in holding time with variations of 2 hours, 3 hours and 4 hours. Sample 10 is a sample using basic materials from the factory Based on **Table 3**, the results of the analysis of the chemical composition contained in composite ceramics, namely, the resulting compounds, namely, SiO_2, Al_2O_3, CaO, Fe_2O_3, K_2O, P_2O_5 and Cl which have a percentage above 1 percent. Based on the results of XRF analysis, none of them reached the desired composition, namely the mass ratio of MgO,

Al_2O_3 and SiO_2 of 14%, 35%, 51%. This occurs due to the low purity level of the raw material, the use of inappropriate comparisons and the process of making samples that are not maximized.

Table 3. XRF analysis results of Composite ceramic samples.

Chemical Composition	Sample (%)									
	1	2	3	4	5	6	7	8	9	10
SiO_2	50,113	47,747	28,115	20,851	20,446	-	44,049	39,058	35,023	54,026
Al_2O_3	28,115	30,425	50,113	9,314	9,227	11,119	33,736	39,653	42,121	31,322
MgO	8,212	7,346	9,142	10,231	9,173	10,221	9,628	9,437	8,912	9,654
CaO	5,095	7,016	4,165	8,194	8,688	41,277	5,411	6,520	6,916	1,484
Fe_2O_3	4,622	3,445	4,622	1,275	1,121	4,578	2,925	2,458	3,748	1,247
K_2O	1,175	1,327	1,175	1,447	1,500	0,603	-	0,445	0,364	0,677
P_2O_5	0,969	1,307	0,969	0,328	-	-	1,062	1,230	1,433	1,056
TiO_2	0,909	0,326	0,909	-	-	0,246	0,853	0,366	0,650	0,293
NiO	0,294	-	0,294	-	-	0,499	-	-	-	-
MnO	0,186	0,298	0,186	0,322	0,262	1,018	0,387	0,350	0,367	-
SO_3	-	0,347	-	3,060	3,010	-	0,515	-	-	-
Cl	-	-	-	44,595	46,237	29,095	-	-	-	-
ZnO	-	-	-	-	-	0,682	0,218	0,214	0,135	-

3.4 Physical Density Test Results of Composite Ceramics

Density testing on composite ceramic samples is carried out with the standard ASTM C373-88. The graph of the test results is presented in **Figure 1**.

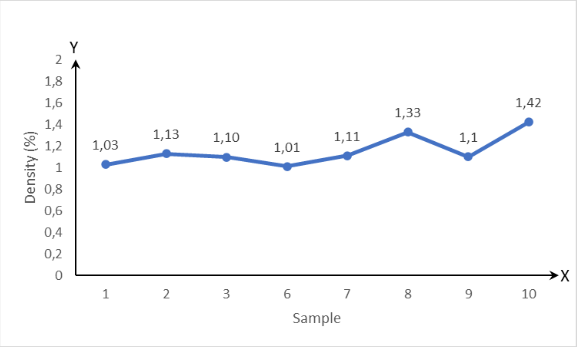

Figure 1. Graph of density values of composite ceramic samples. Sample numbers 1 (0% MgO), 2 (10% MgO), 3 (15% MgO), 6 (1.100˚C), 7 (2 Hours), 8 (3 Hours), 9 (4 Hours) and 10 (factory material sample)

The graph presented in **Figure 1** in the variation of 0% MgO mass addition has a value of 1.03 g/cm^3 then increased the value with the addition of 10% MgO to 1.13 g/cm^3 and at the addition of 15% MgO decreased the density value to 1.10 g/cm^3. In accordance with Meilyana's research (2016), along with the addition of MgO, the density value decreases due to changes in the composition of the ceramic sample. According to Harper (2001), the density value of composite ceramics is 2.3 - 2.5 g/cm3, while the value obtained is still below the standard density of composite ceramics.

Based on the results of the addition of MgO, the highest density is sample 2 of 1.13 g/cm^3. Furthermore, the composition of sample 2 with the addition of 10% MgO is used to perform temperature variations and variations in resistance time.

In the temperature variations carried out with temperatures of 800˚C and 900˚C, the ceramics produced were not in accordance with the standard, so that during the testing process a failure occurred which resulted in the absence of data for testing the density value. The density value obtained in the temperature variation is 1.02 g/cm^3 with a temperature of 1.100˚C.

The graph on the variation of holding time has the highest density value of 1.33 g/cm^3 at a holding time of 3 hours. While the lowest density value is 1.10 g/cm^3 at a holding time of 4 hours.

From the results of the density test on the nine samples made, the highest density value was obtained in sample 8 with a value of 1.33 g/cm^3.

3.5 Physical Porosity Test Results of Composite Ceramic

Porosity testing on Composite ceramic samples is carried out by standard ASTM C373-88. A graph of the test results is presented in **Figure 2.**

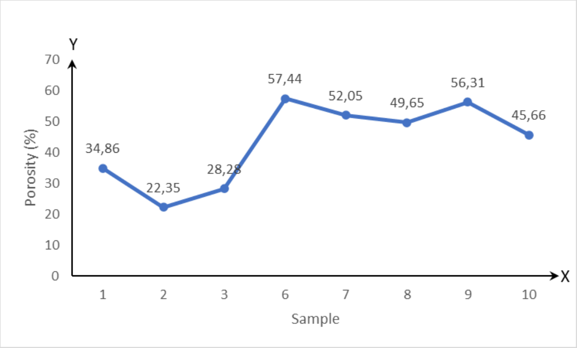

Figure 2. Graph of porosity values of composite ceramic samples. Sample numbers 1 (0% MgO), 2 (10% MgO), 3 (15% MgO), 6 (1.100˚C), 7 (2 Hours), 8 (3 Hours), 9 (4 Hours) and 10 (factory material sample)

The graph presented in **Figure 2** in the variation of 0% MgO mass addition has a value of 34.86% then decreased in value with the addition of 10% MgO to 22.35% and in the addition of 15% MgO increased porosity value to 28.28%. The porosity test results in this study meet the requirements of the porosity value of ceramics on the market, which is 20% to 30% (Kiswanto, 2011). These results are in accordance with the theory, where the greater the density value, the porosity value decreases.

In the temperature variations carried out with temperatures of 800˚C and 900˚C, the ceramics produced were not in accordance with the standard, so that during the testing process there was a failure which resulted in the absence of data for testing density values. The porosity value obtained in the temperature variation is 57.44% with a temperature of 1100˚C.

The graph on the variation of holding time has the lowest porosity value of 49.65% at a holding time of 3 hours. While the highest density value is 56.31% at a holding time of 4 hours.

3.6 Physical Absorption Test Results of Composite Ceramics

Absorption testing on Composite ceramic samples was carried out with the standard ASTM C373-88. The graph of the test results is presented in **Figure 3.**

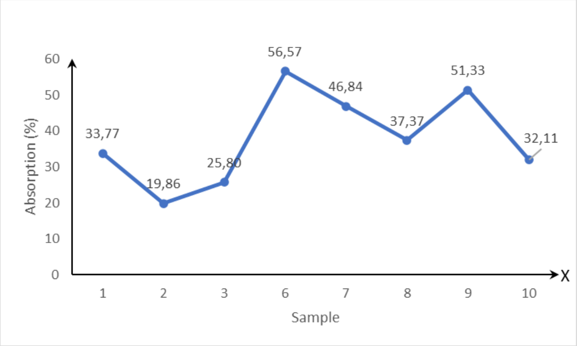

Figure 3. Graph of absorption values of composite ceramic samples. Sample numbers 1 (0% MgO), 2 (10% MgO), 3 (15% MgO), 6 (1.100˚C), 7 (2 Hours), 8 (3 Hours), 9 (4 Hours) and 10 (factory material sample)

The graph presented in **Figure 3** shows that the 0% MgO mass addition variation has a value of 33.77%, then a decrease in value with the addition of 10% MgO to 19.86% and the addition of 15% MgO increased the adsorption value to 25.80%.

In the temperature variations carried out with temperatures of 800˚C and 900˚C, the ceramics produced were not in accordance with the standard, so that during the testing process a failure occurred which resulted in the absence of data for testing the absorption value. The absorption value obtained in the temperature variation is 56.57% with a temperature of 1100˚C.

The graph on the variation of holding time has the lowest absorption value of 37.37% at a holding time of 3 hours. While the highest absorption value is 51.33% at a holding time of 4 hours. The absorption value is proportional to the porosity value and inversely proportional to the density value.

3.7 Mechanical test results of ceramic compressive strength

The compressive strength test was carried out to analyze the quality of composite ceramics synthesized materials with composite ceramics factory base material. Optimum synthesized composite ceramics from the test results of density, porosity and absorption is the composition with the addition of 10% MgO. Furthermore, the optimum synthesized ceramics are compared with composite ceramics factory base material. Based on the results of the compressive strength test on the optimum sample with a compressive force of 1,162.34 N obtained sample compressive strength of 2.37 MPa. While the results obtained from the factory material sample with a compressive force of 2,275.96 N obtained a sample compressive strength of 4.64 MPa. The results of the compressive strength of the factory material sample are better than the optimum sample because the density value of the factory material sample is greater than the density value of the optimum sample.

4. Conclusions

The conclusions obtained in this study are as follows:

1. The results of silica extraction from coal bottom ash obtained a value of 43.51%, alumina from aluminum waste of 31.01%, and magnesium oxide from bittern water of 56.69%. This result is the best value of this research.
2. Physical characteristics of ceramics from variations in the addition of MgO with density, porosity and adsorption tests obtained the best results from sample 2, namely density of 1.13 g/cm^3, porosity of 22.35% and adsorption of 19.86%. Temperature variation obtained the best results from sample 6, namely density of 1.03 g/cm^3, porosity of 57.44% and adsorption of 56.57%. The variation of resistance time obtained the best results from sample 2, namely density of 1.1 g/cm^3, porosity of 56.31% and adsorption of 51.33%. The XRF results of the ceramics did not reach the composite composition.
3. The quality of the optimum sample is still below the factory material sample, namely density of 1.42 g/cm^3, porosity of 45.66%, adsorption of 32.11% and compressive strength of 2.37 MPa. Contains the essence of research written briefly and clearly. Conclusions have answers to problems and their conformity with research objectives.

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