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https://doi.org/10.5109/7183444

出版情報:Evergreen. 11 (2), pp.1333-1340, 2024-06. 九州大学グリーンテクノロジー研究教育セン ター バージョン:

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Effect of Sodium Tripolyphosphate on The Morphology and Thermal Properties of Anorthite-Based Ceramic Foam Filters for Aluminum Alloy Castings

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(Received October 22, 2023; Revised January 31, 2024; Accepted March 05, 2024).

Abstract: Aluminum is one of the most widely used materials for daily life and has a wide range of applications. However, the continuous use of aluminum makes Indonesia unable to meet its needs for aluminum, thus forcing Indonesia to import aluminum. Therefore, the use of secondary aluminum as a substitute for primary aluminum raw materials is one solution that can be offered. Despite the low cost of secondary aluminum as the raw material, there are many impurities contained in secondary aluminum that can reduce the properties of the resulting aluminum products. In this study, porous anorthite ceramic filters were fabricated to improve the purity of cast products with secondary aluminum. This filter is made by using polyurethane as a support that is dipped into a slurry consisting of a mixture of silica sand and limestone, accompanied by the addition of sodium tripolyphosphate (STPP) deflocculant with variations in composition (5, 10, and 15 grams). Based on the results of testing and characterization, it is found that the filter sample with the addition of 15 grams of STPP provides optimum performance, with an average pore area of 0.329 mm², viscosity of 1.54 cP, porosity level of 68%, density of 0.55 g/cm³, water absorption of 126%, permanent linear change (PLC) of -4.8%, and thermal expansion of 1.06%.

Keywords: Aluminum, Anorthite-Based Porous Ceramic Filter, Replica Method, STPP

1. Introduction

Aluminum is in high demand as one of the export commodities in the future because it has many advantages such as lightweight among other metals, environmentally friendly, good electrical and thermal conductivity, as well as corrosion resistance^{1,2)}. In the global market, demand for aluminum is predicted to continue to grow until 2035, especially in Asia and South America³⁾. Several countries, such as China, Germany, and the United States, were the largest aluminum exporting countries in the world throughout 2016. Meanwhile, Indonesia was ranked 51st as an aluminum exporting country. Aluminum is made from a mixture of 66% bauxite ore and 33% clay. National sources of bauxite in Indonesia can be found in the Riau Islands, Bangka Belitung, West Kalimantan, South Sulawesi, Maluku, and Papua. According to the World Bureau of Metal Statistics, aluminum production in Indonesia is around 220.6 thousand tons in 2021⁴⁾. This production has decreased compared to 2011, which produced around 246.3 thousand tons of aluminum.

In general, the availability of aluminum in the world is very limited. Therefore, aluminum is recycled using scrap aluminum to replace primary aluminum⁵⁾. In the aluminum recycling industry, aluminum is closely related to inclusion⁶⁾. Types of inclusions that can generally be found in aluminum scrap are oxides (Al₂O₃ and MgO), carbides (Al₃C₄, TiC), spinels, borides, and intermetallic^{7,8)}. The inclusions produced by aluminum scrap will be filtered to produce pure aluminum with high levels. The screening can use various tools such as Fluidity Inclusion Meter (APIF), PoDFA, and Prefill^{9,10)}. These tools have a similar function, namely to filter inclusions in aluminum scrap to obtain pure molten aluminum and provide information regarding the concentration and composition of the inclusions contained therein.

Ceramics are a mixture of metals and non-metals, which are made of inorganic materials through a combustion process at high¹¹). Among several types of materials, such as alumina, mullite, cordierite, anorthite, silica, zirconia, and carbon-bound, are used in forming porous ceramics^{12,13}. This study uses anorthite because it has several advantages, such as a very low coefficient of thermal expansion, low dielectric constant and loss, good thermal shock resistance, and high creep resistance at high temperatures. Anorthite raw materials consist of materials that are generally cheap and easy to find in several regions of Indonesia, such as Bangka Belitung. These raw materials include kaolin, calcite, quartz, and CaO from limestone (CaCO₃)^{14–17}.

In this study, a simple ceramic filter fabrication using the replica method is introduced, where polyurethane foam is dipped into a container containing a slurry containing a mixture of silica sand and limestone^{18,19}. In addition, this study will analyze the effect of adding sodium tripolyphosphate (STPP) as a deflocculant on the morphology, physical properties, and thermal properties produced after porous ceramics are formed from heating at 1200°C.

The porous ceramics formed will be subjected to various tests such as microstructural testing with a Scanning Electron Microscope (SEM), composition testing with X-ray Diffraction (XRD), thermal expansion testing, permanent linear change testing, viscosity testing, and porosity testing. This research aims to find the optimum STPP content that produces ceramic foam filters with the best properties so that the effective removal of impurities from the molten aluminum can be ensured, thus improving the quality of the castings^{20–22)}.

2. Experimental

Making ceramic slurry began with preparing a plastic container and then weighing the ingredients using a scale with a composition of 73.04% kaolin or 3.652 kg and 26.96% or 1.3 kg of limestone powder. The two compositions added up to 5 kg, then 2 liters of water was added. After getting the right concentration, the mixture is put into a pot mill and then milled for 18 hours until homogeneous. After that, 3 kg of the mixture was taken, and then 1.1 liters of water and STPP-type deflocculant with different compositions were added. The composition of slurries can be seen from Table 1.

Table 1. Variable composition of kaolin, limestone, water, STPP

| 5111 | | | | | |
|------|--------------------|------|-------|--|--|
| Code | Kaolin : Limestone | STPP | Water | | |
| 1 | | 5 g | | | |
| 2 | 73% : 27% | 10 g | 1.1 L | | |
| 3 | | 15 g | | | |

Fabrication of porous anorthite ceramic filters began after the slurry was made. This process was carried out by preparing 30 pores per inch of polyurethane foam and several casts, then dipping the foam into each slurry with different STPP deflocculant content²³⁾. Make sure the polyurethane foam is evenly immersed, then place the foam on top of the plaster that has been prepared and dried at room temperature for 7 days. Polyurethane foam that has been coated with slurry and dries is burned in the furnace slowly²⁴⁻²⁶). The combustion temperature starts from 2°C per minute up to 600°C followed by 5°C per minute up to 1200°C with a total burning time of 564 minutes. The following process is transferring the sample to a container and then carrying out various material characterizations to obtain the morphology and thermal properties of porous anorthite ceramic filters, such as microstructural testing, composition testing, porosity testing, viscosity testing, thermal expansion testing, and permanent linear change testing²⁷⁾.

3. Result and Discussion

3.1 Chemical Composition

The chemical composition of the ceramic filter produced can be known using XRD. XRD characterization of ceramic materials refers to their mineral and phase structure. As seen in Fig. 1 below, XRD characterization identified mullite (Al₄SiO₈) and cristobalite (SiO₂) as the samples' main minerals, confirming the EDS test results (Table 2). These findings also follow the results of Chargui (2018), who stated that sintering kaolin at 1200°C resulted in a significant amount of mullite crystallization followed by cristobalite crystallization triggered by the presence of amorphous silica in kaolin²⁸.



Fig. 1: XRD result of anorthite-based ceramic foam filter

Table 2. EDS characterization result of local filters

| Element | Unit | 5 g STPP | 10 g STPP | 15 g STPP | |
|---------|------|----------|-----------|-----------|--|
| С | | 05.33 | 01.23 | 01.84 | |
| 0 | | 33.37 | 31.85 | 37.34 | |
| Na | | 01.19 | 0.66 | 0.27 | |
| Al | wt% | 15.64 | 11.40 | 16.67 | |
| Si | | 18.47 | 14.79 | 19.49 | |
| K | | 01.16 | 0.37 | 0.26 | |
| Ca | | 24.84 | 39.70 | 24.15 | |

3.2 Morphology

To analyze the morphology of the sample, SEM and optical microscope (OM) were utilized, accompanied by the mean area of the pore as the quantitative data that are calculated using ImageJ software. The SEM images of the ceramic foam filter samples are presented in Fig. 2. The SEM images of every sample show the production of struts due to the polyurethane foam being submerged in the slurry, resulting in the same shape as the foam itself. Other than that, not many distinctive features can be observed. Therefore, OM was employed to analyze the morphology further.

The OM images of the ceramic foam filter samples can be seen in Fig. 3. From the images, it can be seen that not all pores were open, and the strut looked thin. The closed pores are produced by the retained excess suspension. This is because the samples were dried on a plaster or flat surface. So that over time, the excess suspension could not escape from the sample, resulting in closed pores. Despite the fact that some of the pores are closed, the pore structure (open or closed) cannot be fully distinguished from the macroscopic observation alone. To find out the average pore size of each sample, ImageJ software is used, which is shown in Table 3.

From all the data obtained in Table 3, it can be concluded that the largest average pore size is owned by the 15 gr STPP deflocculant addition sample. The pore size of each porous anorthite ceramic sample did not depend on the addition of STPP deflocculants. However, it only depended on the pore size of the polyurethane foam, whereas in this study, the polyurethane foam used had a size of 30 pores per inch. The addition of STPP deflocculants depends on the number of pores formed in porous anorthite ceramics.





Fig. 2: SEM images of (a) sample 1, (b) sample 2, and (c) sample 3



Fig. 3: Optical microscope result (a) 5 g, (b) 10 g, and (c) 15 g STPP samples

Table 3. Calculated mean area of pore using the ImageJ software

| Sample | Mean of Area Pore (mm ²) | | | | |
|-----------|--------------------------------------|--|--|--|--|
| 5 g STPP | 0.222 | | | | |
| 10 g STPP | 0.310 | | | | |
| 15 g STPP | 0.329 | | | | |

3.3 Viscosity

The Lehmann viscosimeter was used to conduct a viscosity test after combining water and STPP to create the slurry to determine the STPP content's impact on the slurry's rheology. Fig. 4 and Fig. 5 depict the test's results with Lehmann formula²⁹.



Fig. 5: Linear regression of the viscosity result

Based on the graphical observations in Fig. 4 and Fig.5, it can be seen that the graph decreases as the STPP deflocculant increases. It means that the addition of STPP can certainly affect the slurry viscosity. STPP, as the deflocculant, restricts the agglomeration of the slurry, thus reducing the agglomeration size and decreasing the viscosity. Viscosity value will have an effect when carrying out the process of dipping polyurethane foam into anorthite slurry, where if the viscosity value is too low, the slurry is difficult to adhere to each pore because it is too runny and if the viscosity value is too high, the slurry will clog at the top surface of the pore due to being too thick. This is in accordance with R. J. Manulangg, et al who stated that the decrease in the viscosity value is caused by constant pressure and stirring speed. Viscosity will increase if the concentration of the solution is increased, but in this study, the amount of solution remains the same, which makes the viscosity value only affected by the pressure and speed of stirring given. Otroj S et al, said that the viscosity value tends to decrease with increasing STPP deflocculant concentration³⁰⁾. The negative trend on the graph is caused by the pH of the STPP deflocculant (pH 9.2 - 10), which is above the Isoelectric Point or IEP (pH 4.8 - 6.3). The viscosity value of the addition of the STPP deflocculant will continue to decrease because there is repulsion between the particles, so the graph also continues to decrease³¹).

3.4 Apparent Porosity, Water Absorption and Bulk Density

The ASTM C20-00 was followed when conducting this test. For each sample code, three separate specimens were weighted in accordance with the technique. The outcomes are depicted in Fig. 6 and Fig. 7. Procedures for testing porosity, density, and ability to absorb water refer to ASTM C20- 00^{32}).

The graph of the porosity value and density value provides information that the porosity value has a value relationship that is inversely proportional to the density value in this study because the density value is influenced by the quantity of porosity formed (Fig. 6(a), Fig. 6(b), Fig.7(a), and Fig.7(b)). The more porosity is formed, the lower the resulting density because the density value is affected by the shape and size of the ceramic filter. The decrease in the density value is also caused by the ability to absorb water because density is also affected by the water content and the type of material used. In addition to these two graphs, the graphs of porosity values and water absorption values have a value relationship that is directly proportional. This is in accordance with Widodo N, who stated that as the concentration of the STPP deflocculant increases, the porosity value will increase, but the density value of the porous anorthite ceramic filter will decrease. Widodo N also stated that by increasing the porosity value, the ceramic filter will easily absorb water because water absorption affects the physical properties of ceramic filters, which are characterized by the shape of ceramic filters that do not change or are consistent. The high ability to absorb water also proves that the type of pores formed from porous anorthite ceramic filters is open pores (Fig. 6(c) and Fig. 7(c)).





Fig. 6: (a) Porosity test result, (b) density test result and (c) water absorption test result



absorption and (c) density

3.5 Thermal Properties

Thermal expansion and persistent linear change are the thermal qualities tested in this investigation. The sample was gradually heated throughout the permanent linear change test until it reached the 700°C pouring temperature for aluminum and was held there for five hours. After the sample had cooled, the measurement was then completed. The sample is exposed to 700°C of heat directly during the thermal expansion test. Using a dilatometer, the measurement is completed concurrently with the heat exposure. For each code sample, this test is run three times with different specimens. The PLC test procedure refers to ASTM C113-14, while the thermal expansion test procedure refers to ASTM E228-95^{33,34}).



Fig. 8: (a) Permanent linear change test result and (b) thermal expansion test result





Fig. 9: Linear Regression Charts for (a) Permanent linear change test result and (b) thermal expansion test result

As the STPP deflocculant increases, the PLC value will show a decrease in this study (Fig. 8(a) and Fig. 9(a)). This negative trend indicates that shrinkage will occur when the sample is added with STPP deflocculant. This is in accordance with Kockal N, who stated that the more STPP deflocculants added, the higher the shrinkage value³⁵). As the concentration of the STPP deflocculant increases, the sample reacts more due to the evolved gas during the heating process. This proves that porous anorthite ceramic filter samples with the addition of STPP deflocculant concentration experience shrinkage when heated at 700°C.

As the concentration of STPP deflocculant increases, its thermal expansion value increases (Fig. 8(b) and Fig. 9(b)). This positive trend also indicates that the porous anorthite ceramic filter sample is expanding. This is in accordance with Doloksaribu et al. who stated that there is an inverse relationship between thermal expansion and density, where the smaller the density value, the greater the thermal expansion value because a small density value gives information that the ceramic filter is not dense, so that the ceramic filter easy to expand.

In metal casting, a higher thermal expansion and lower permanent linear change in ceramic foam filters could potentially be beneficial. These properties allow the filter to withstand the extreme temperatures involved in the casting process while maintaining its structural integrity and filtration efficiency. The ability to accommodate thermal expansion can result in good thermal shock resistance so that structural damage can be avoided and dimensional stability can be achieved during temperature fluctuations.

4. Conclusion

Using kaolin and limestone as starting materials, the replica method has been used to fabricate anorthite-based ceramic foam filters. The anorthite phase can be successfully synthesized at a firing temperature of 1200°C. The polyurethane foam, not the STPP content or the amount of flocculant, determines the pore size of the ceramic foam filter. Due to their large capacity for water

absorption, ceramic foam filters have been shown to contain a large number of open pores. However, STPP was shown to affect the properties and performance of the resulting ceramic foam filter product. Based on the results, it was found that the filter sample with the addition of 15 grams of STPP provides optimum properties, with an average pore area of 0.329 mm², viscosity of 1.54 cP, porosity level of 68%, density of 0.55 g/cm³, water absorption of 126%, permanent linear change (PLC) of -4.8%, and thermal expansion of 1.06%.

Acknowledgements

This work was supported by a research grant from Direktorat Inovasi dan Science Techno Park of Universitas Indonesia for the P5 Programme in Program Pendanaan Inovasi 2023 (Grant No. PKS-562/UN2.INV/HKP.05/2023). The authors would also like to thank Balai Besar Standarisasi dan Pelayanan Jasa Industri (BBSPJI) Keramik dan Mineral Nonlogam, Ministry of Industry, Bandung.

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