Precipitated Silica from Pumice and Carbon Dioxide Gas (CO₂) in Bubble Column Reactor

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Abstract. Precipitated silica from silica and carbon dioxide gas has been studied successfully. The source of silica was obtained from pumice stone while precipitation process was carried out with carbon dioxide gas (CO₂). The sodium silicate solution was obtained by extracting the silica from pumice stone with sodium hydroxide (NaOH) solution and heated to 100 °C for 1 h. The carbon dioxide gas is injected into the aqueous solution of sodium silicate in a bubble column reactor to form precipitated silica. m²/g. The results indicate that the products obtained are precipitate silica have surface area in the range of 100 – 227 m²/g, silica concentration more than 80%, white in appearance, and silica concentration reached 90% at pH 7.

1. Introduction

Precipitated silica is a silica product, containing silica, a white powder, a large enough porous and nano particle sized. The precipitated silica products are needed to support the operation of various types of industries such as automotive tire, rubber, cosmetics, electronics, agriculture and others [1]. This precipitate silica can be produced by a chemical reaction process between the sodium silica solution and various types of acids such as hydrochloric, sulfuric, acetate and other acids [2,3]. The properties of precipitated SiO₂depend strongly on the conditions of its synthesis, such as the synthesis temperature, time of precipitation, pH, addition of coagulant, and modes of washing and drying. These factors influence the size of the SiO₂particles, their aggregation and specific surface area.

Precipitation is carried out under alkaline conditions. The choice of agitation, duration of precipitation, the addition rate of reactants, their temperature and concentration, and pH can vary the properties of the silica. The formation of a gel stage is avoided by stirring at elevated temperatures. The resulting white precipitate is filtered, washed and dried in the manufacturing process [4]. Conventionally precipitated silica is manufactured by of fusion silica sand and sodium carbonate at high temperatures of around 1400 °C [5] and the sodium silicate obtained is precipitated using acid mainly, sulphuric acid. To reduce energy consumption and prevent the impact of sulfuric acid pollution, the extraction process was carried out by digesting silica present in rice husk by the addition of sodium hydroxide to manufacture sodium silicate and precipitating silica from sodium silicate by reaction with carbon dioxide [6]. Several studies on the synthesis of silica using CO₂ gas have been developed [7,8]. The silica precipitated using a homogenizer and ultrasound bath have shown much higher specific surface area than that of the obtained...
silica using the top propeller stirrer [10]. This research develops bubble column where CO₂ gas will contacted with sodium silicate solution inside. The characteristic of precipitated silica particles are porous, agglomerate size is 1 - 40 μm with average pore size is > 30 nm. Density: 1.9 - 2.1 g/cm³ [3], appearance: white free flowing powder or lum, primary particles with a diameter of 5 - 100 nm, and specific surface area 5-100 m²/g. Silica content SiO₂ min 88 to 90%, pH of 5%slurry 7+0.3 [4]

2. Materials and Method
In this study using pumice as a source of silica and carbon dioxide (CO₂) gas as a medium of precipitation formation. The precipitation reaction of CO₂ gas with sodium silicate occurs in a semi-batch bubble reactor. The CO₂ gas is injected continuously into the column containing the sodium silicate solution until the pH is reached. Quality of precipitated silica analyzed by XRD/XRF method, and BET analysis. The two process steps are presented in Figure 1.

3. Results and Discussion

a. Chemical composition of the Pumice
Tabel 1 shows the chemical composition of Pumice analyzed by X-ray fluoroscense.

| No | Parameters              | Concentration (%) |
|----|-------------------------|-------------------|
| 1  | Silica (SiO₂)           | 63.0              |
| 2  | Aluminum oxide (Al₂O₃)  | 13.3              |
| 3  | Calcium oxide (CaO)     | 8.43              |
| 4  | Potassium oxide (K₂O)   | 9.95              |
| 5  | Ferryl oxide (Fe₂O₃)    | 5.20              |

The chemical composition of sodium silicate prepared by extraction of fumice with sodium hydroxide (NaOH) 1 N is 2.16% SiO₂ and 11.68% of Na₂O
b. The effect of pH and volume ratio of sodium silicate/H₂O on silica concentration of precipitated silica product

![Graph showing the effect of pH on silica and aluminum oxide concentration](image)

Figure 2. Effect of pH and volume ratio of sodium silicate/H₂O on a) silica concentration and b) aluminum oxide concentration on precipitated silica products

Figure 2 showed the effect of pH and ratio sodium silicate/H₂O on a) silica concentration and b) aluminum oxide concentration on precipitated silica products. The increasing of pH silica content on precipitated silica will decrease because the increasing of pH will need more CO₂ gas injected and will produce more sodium carbonate salt. Sodium carbonate will covering the pores of the silica particle. The silica con 90.7 The higher the pH of the lower silica concentration for all dilution ratios are from 1:0.5 to 1:2.5. Thus the highest silica concentration (90.7%) was obtained at pH 7. It clears from Figure 3b showed that when the silica concentration is low the concentration of aluminum becomes high. The results showed the highest aluminum content was on precipitated silica product pH 10 in the range of 15-19.5%.

c. The effect of pH and ratio sodium silicate/water to surface area of precipitated silica product

Figure 3 showed the effect of pH and ratio sodium silicate/H₂O on surface area of precipitated silica product. At pH 7 and pH 8 it appears that there is a similar tendency of surface area decrease in the dilution sodium silicate ratio of 1:0.5 to 1:1.5, then the surface area increases to reach the ratio of 1:2.5. At pH 9 the surface area increases steadily along with the addition of dilution from 1:0.5 to 1:2.5. While at pH 10 happened opposite of pH 7 and pH 8 that is surface area increase in ratio 1:0.5 until 1:1.5 then decrease until reach ratio 1:2.5 dilution. The largest surface area occurs at pH 8, sodium silicate/H₂O ratio of 1:2.5 but the silica content is low about 77.3%.
Figure 3. Effect of pH and ratio sodium silicate/H₂O on surface area of precipitated silica product

4. Conclusion

Based on data research results can be concluded as follows:

a. The pumice used as a silica source contains 66.3% silica and 13.3% aluminum oxide.
b. The best pH of the precipitate silica production is 8.
c. The silica content of the precipitate silica product is 90.1% and the aluminum oxide is 8.1%.
d. The surface area of the precipitate silica product is 108 – 227 m²/g.

5. Acknowledgment

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6. References

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