

Conference Paper

Precipitated Silica by Precipitation Process of The Sodium Silicate Solution with Carbon Dioxide Gas (CO₂) on Fixed Bed Column

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Abstract

Precipitated silica is a silica product produced by a precipitation process, this silica product contains silica, white color, very porous and nanomaterial. This precipitate silica is required to support various types of industrial operations such as vehicle, rubber, cosmetics, electronics, agriculture and other industries. The precipitated silica is produced through two stages of the process: firstly is silica extraction process with the base solution and the secondly is precipitation processes of silica solution with various types of acids such as hydrochloric acid (HCl), sulfuric acid (H₂SO₄), acetic acid (CH₃COOH) and other acids. In this study, the source of silica was obtained from bagasse ash waste of sugar industry, the base solution as a solvent was sodium hydroxide (NaOH) 2 N and the precipitate process was carried out by using carbon dioxide gas (CO₂) and the precipitation process was carried out on a fixed bed column. In this study we studied the effect of silica concentration on the inlet feed and the high of the fixed bed on the quality of the precipitated silica product. Based on the results of this study, the quality of precipitated silica produced as follows: 95-98% silica content, weight loss due to temperature heating 105 C: 4 – 5 %, acidity (pH of slurry 5%) : 6.8-7,1, and surface area (BET): 175 m²/gram. The best conditions in this study were 30 cm high of fixed bed, and concentration of silica feed: 0,33% weight and acidity (pH) precipitation 7,1.

Keywords: bagasse ash, carbon dioxide gas, fixed bed, precipitation, silica

INTRODUCTION

Precipitated silica is a silica (SiO₂) produced by precipitation from a solution containing silicate salts. this silica product contains silica, white color, very porous and nanomaterial size. This precipitate silica is required to support various types of industrial operations such as vehicle, rubber, cosmetics, electronics, agriculture and other industries (Music et al., 2011). The data of Export and import precipitated silica in Indonesia as show on table 1 (Bogeshwaran et al., 2014; Otto et al., 2008; Ghosh & Sounak, 2013).

Table 1 shows the amount of imports is far greater than that of exports, this shows the need for silica precipitate in Indonesia is still very high, so the opportunity for the development of the silica industry precipitate is very large. The quality of precipitate silica products depends on the type of industrial users. One of the qualities of precipitated silica for the rubber industry (rubber) as show on table 2 (Shelke et al., 2010; Srivatava et al., 2013).

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Table 1. Export-import data of precipitated in Indonesia

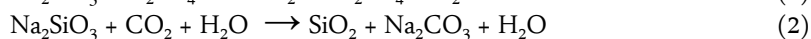
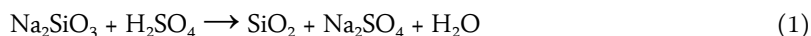
Year	Export (ton)	Import (ton)
2003	13,5	3.688,5
2004	84,3	4.975,0
2005	115,1	5.276,4
2006	502,4	4.988,0
2007	606,1	5.963,4

Source: Central Statistics Agency (2008)

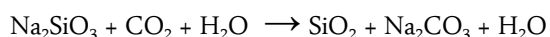
Table 2. Quality of precipitated silica for rubber industry

No	Parameters	Specifications / Limit
1	Appearance	White Powder
2	BET Surface area	160 - 180 m ² /gr
3	Moisture content (at 105°C for 2 hrs)	6 - 7 %
4	pH (5 % aqueous suspension)	6 - 7
5	Ignition Loss at 1000°C (for 2hr. in furnace)	5 - 6 %
6	SiO ₂ Content	98.5 (Min) (%)
7	Soluble Salt	1.5 (Max) (%)
8	Iron Content (As Fe+3)	200 (Max) ppm

The production of precipitated silica starts with the reaction of an alkaline [silicate](#) solution (Na₂SiO₃ solution) with various types of acids such as hydrochloric, sulfuric, acetate and other acids (Bogeshwaran et al., 2014). The acids and [sodium silicate](#) solutions are added simultaneously with agitation to water. [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 (Garrett, 1992).



In this study carbon dioxide (CO₂) gas is used as precipitate material, the reaction that occurs as below:



The precipitated silica production is carried out through two stages of reaction, namely :

Stage 1 : Extraction silica by sodium hydroxide (NaOH) to make sodium silicate solution (Na₂SiO₃)

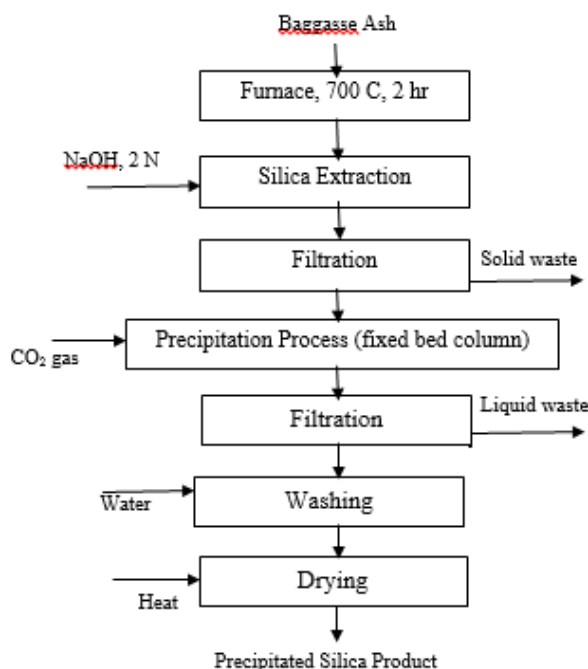
Stage 2 : Precipitation process of sodium silicate solution with carbon dioxide (CO₂) gas

METHODS

The material used in this study is bagasse ash obtained from the sugar industry, bagasse ash is burned in a furnace at 700 °C for 2 hours to obtain ash, the ash obtained is analyzed by the XRF method. The Other material are sodium hydroxide (NaOH, 2 N) as an extraction of silica in bagasse ash and carbon dioxide (CO₂) gas as precipitates. The ash bagasse was extracted with sodium hydroxide solution (NaOH, 2 N) at 95 °C for 2 hours, the extraction results in the form of sodium silicate solution were analyzed for the content of sodium oxide (Na₂O) with AAS method and silica content (SiO₂) by Spectrophotometry method (Venkataramana, 2016).

The Sodium silicate solution is pumped (dosage pump) with a discharge of 60 ml/min into the fixed bed column (diameter 7.5 cm and height 50 cm) and followed by injection of carbon dioxide (CO₂) gas with a discharge

of 4 liters /minute. The sodium silicate solution will reacted with carbon dioxide gas, pH of sodium silicate solution decrease from pH 12 to 7, precipitated silica product will flow from above the fixed bed column, were filtered and carried out the process of washing and drying. The precipitated silica product was analyzed of the characteristics by the XRF method. Block diagram of precipitated silica production as show on diagram below.



RESULT AND DISCUSSION

Chemical composition of bagasse ash

Bagasse ash from sugar industry after burned on furnace at 700 °C for 2 hr is analyzed by XRF method, The chemical composition of bagasse ash that use as raw sources of silica in this research is presented in the following table 3.

Table 3. Chemical composition of bagasse ash

No	Parameters	Concentration (%)
1	Silica (SiO ₂)	73,0
2	Difosforus pentaoksida (P ₂ O ₅)	2,90
3	Calcium oxide (CaO)	8,63
4	Potassium oxide (K ₂ O)	5,01
5	Ferry oxide (Fe ₂ O ₃)	7,2

Chemical composition of Sodium Silicate

The extraction of silica on bagasse ash with sodium hydroxide (NaOH) 2 N, chemical composition of sodium silicate is presented in the following table 2. Table 2 is extraction results of fumice by sodium hydroxide (NaOH) 2 N.

Table 4. Chemical composition of Sodium Silicate Solution

No	Parameters	Concentration (%)
1	Silica (SiO ₂)	4,85
2	Sodium oxide (Na ₂ O)	4,57

The effect of fixed bed high and concentration silica feed to silica content of precipitated silica product

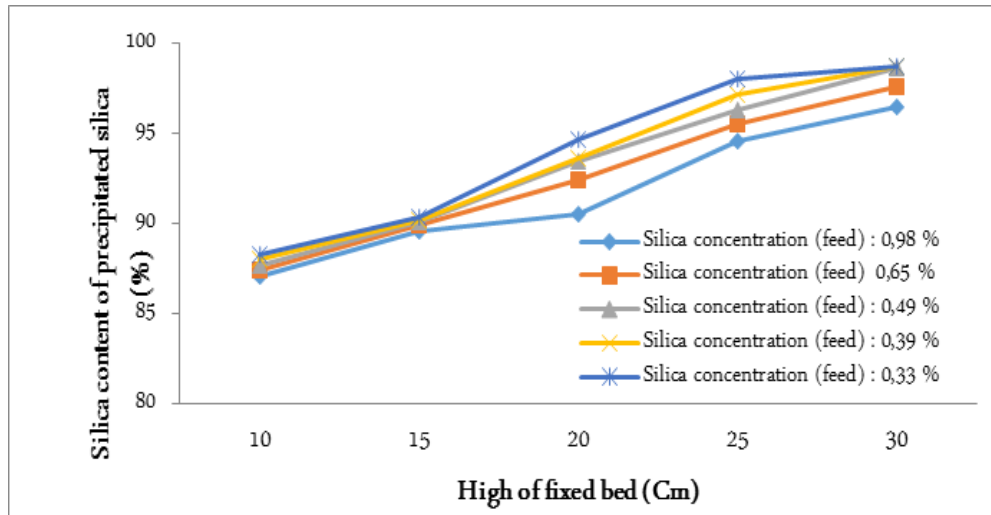


Figure 1. correlation of high of fixed bed to silica content on precipitated silica product

Figure 1. shows that at the same feed silica concentration, the higher of the fixed bed, silica content on the precipitate silica product is more high because the higher of the fixed bed, the carbon dioxide gas contact time with sodium silicate solution more longer. At the same height of fixed bed, the greater the concentration of silica that enters (feed) to the fixed bed column, the silica content on the precipitated silica product smaller this is due to the low concentration of silica salt (sodium carbonate) which forms also lower, so that when washing salt it is easily soluble which in turn will increase the silica content.

The effect of washing on precipitated silica product

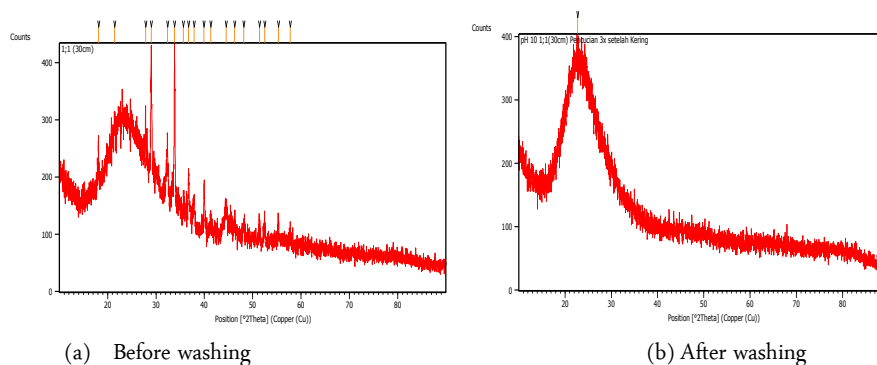


Figure 2. effect of washing on precipitated silica product; a) before washing and b) after washing

Figure 2.(a) and (b) show that the washing process has an effect on the purity of precipitated silica products. Before the washing process is carried out, there are still visible dissolved salts indicated by so many the peak.

The Scanning Electron Microscopy (SEM) of Precipitated Silica product

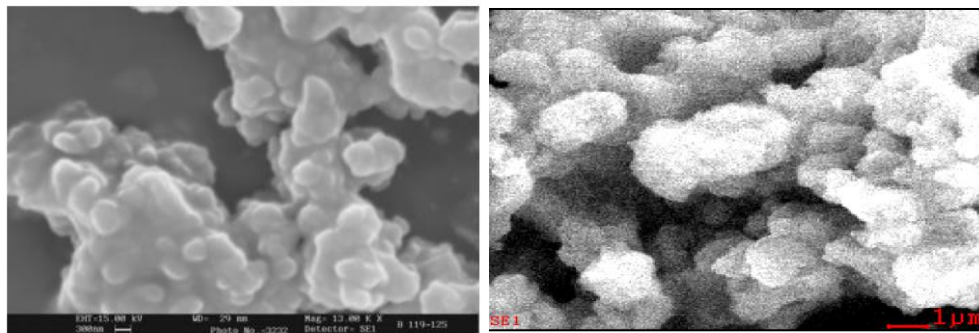


Figure 3. SEM of precipitated silica product

The photo above shows that the diameter of the particles is evenly distributed, it shows that the use of carbon dioxide gas can produce uniform sized silica precipitates.

CONCLUSION

Based on data research results can be concluded as follows :

- a. The bagasse ash from sugar industry as a silica source contains 73.% silica.
- b. Silica in bagasse ash can be extracted with sodium oxide (NaOH) 2N solution and produced a solution of sodium silica with a silica content (SiO₂) of 4.85% and sodium oxide (Na₂O) of 4.57%.
- c. Precipitate silica can be produced through a reaction between sodium silica (Na₂SiO₃) solution and carbon dioxide (CO₂) gas.
- d. The fixed bed column can be one of the technologies that can be considered in the production of silica precipitate liquid-gas systems.
- e. The fixed bed to column diameter ratio (H/D) is 4 can produce precipitated silica with silica content 98.7%.
- f. pH for 5% slurry precipitated silica product : 7.1.
- g. Loss of heating at 110 C for 2 hours : 4 - 5%.

Recommendations:

- a. Bagasse ash solid waste from sugar industry can used as raw material on precipitated silica production.
- b. The fixed bed column can be one of the technologies that can be considered in the production of silica precipitate liquid-gas systems.

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