

## Conference Paper

### Precipitated Silica from Pumice and Carbon dioxide Gas (CO<sub>2</sub>) in Bubble Column Reactor

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#### Abstract

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 industry, rubber industry, cosmetics industry, electronics industry, agriculture and others. This precipitate silica can be produced by a chemical reaction process between the sodium silicate solution and various types of acids such as hydrochloric, sulfuric, acetate and other acids. In this study, the source of silica was obtained from a pumice while precipitation formation was carried out with the use of carbon dioxide gas (CO<sub>2</sub>). The sodium silicate solution was obtained by extracting the silica contained in a pumice with sodium hydroxide (NaOH) solution and heated to 100C for 1 hours. The carbon dioxide gas is injected into the aqueous solution of sodium silicate in a bubble column reactor and there is a reaction to form a precipitated silica. Based on research results found that the quality of precipitated silica has a quality: white colors, 85-90% silica content, 12-20% aluminum oxide content and surface area 108-227 m<sup>2</sup>/g. The best condition of the reaction to precipitated silica is pH 8 and sodium silicate/water ratio (1:2.5).

**Keywords:** Bubble column reactor, carbon dioxide gas, precipitated silica, pumice stone.

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#### INTRODUCTION

Precipitated silica is a silica (SiO<sub>2</sub>) produced by precipitation from a solution containing silicate salts. Precipitated silica is a silica product, containing silica, a white powder, a large enough porous and nano particle sized (Musid et al., 2011). The precipitated silica products are needed to support the operation of various types of industries such as automotive tire industry, rubber industry, cosmetics industry, electronics industry, agriculture and others (Handayani et al., 2015). 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 (Ghosh and Bhattacharjee, 2013).

The production of precipitated silica starts with the reaction of an alkaline silicate solution with a mineral acid. Sulfuric acid and sodium silicate solutions are added simultaneously with agitation to water. Precipitation is carried out under alkaline conditions (Srivastava et al., 2013). 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 (Mihaddilla and Putra, 2013). 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 (Hanafi and Nandang, 2010).

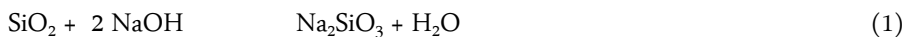
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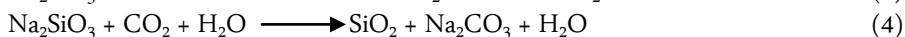
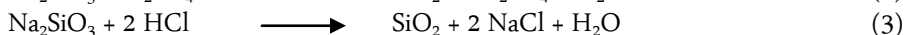
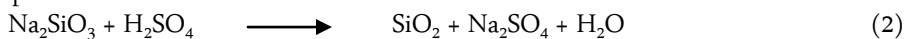
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The production of precipitated silica through two stage process, the first process is silica extraction from sources of silica with base like sodium, potassium or calcium oxides and make alkaline silicate (Bogeshwaran et al., 2014). The second process is precipitation process of alkaline silicate with a mineral acid. The overall reaction of The precipitate silica production mechanism is shown as the following reactions (Shelke et al., 2010).

The extraction reaction of silica with alkaline:



The precipitation reaction of sodium silicate:



The precipitated silica has particles are porous, primary particles with a diameter of 5 - 100 nm, and specific surface area 5-100 m<sup>2</sup>/g. Agglomerate size is 1-40 μm with average pore size is > 30 nm. Density: 1.9–2.1 g/cm<sup>3</sup>. The chemical composition and properties of precipitated silicate are presented in the following table 1.

Table 1. The chemical composition and properties of precipitated silicate

No	Characteristics	Limit
1	Appearance	White free flowing powder or lump
2	Silica content SiO <sub>2</sub> min	88 to 90%
3	Surface area	Around 170 m <sup>2</sup> /g (by BET-N2) 5 – 100 m <sup>2</sup> /g (another sources)
4	pH of 5% slurry	7 + 0.3

## METHODS

In this study using pumice as a source of silica and carbon dioxide (CO<sub>2</sub>) gas as a medium of precipitation formation. The precipitation reaction of CO<sub>2</sub> gas with sodium silicate occurs in a semi-batch bubble reactor. The CO<sub>2</sub> gas is injected continuously into the column containing the sodium silicate solution until the pH is reached (Miskah, 2010). Quality of precipitated silica analyzed by XRD/XRF method, and BET analysis. The two process steps is presented figure 1.

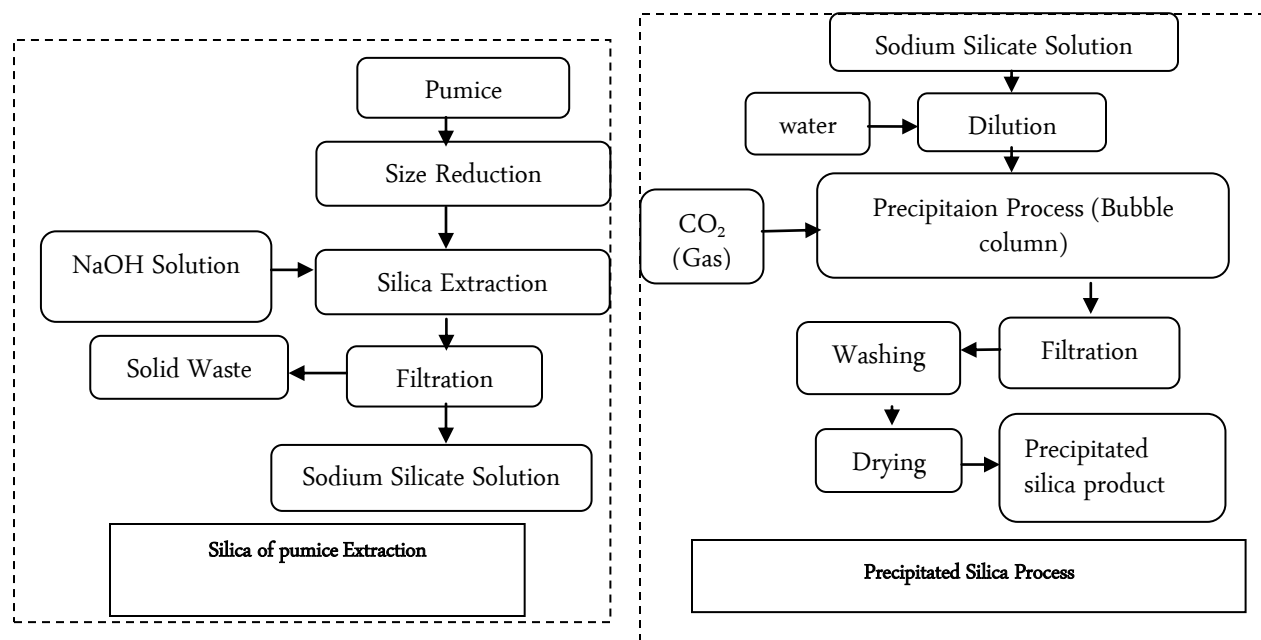


Figure 1. Schematic of the research

## RESULT AND DISCUSSION

### Chemical Composition of the Pumice

The chemical composition of Pumice that use as raw sources of silica in this research is presented in the following table 2.

Table 2. Chemical composition of Pumice

No	Parameters	Concentration (%)
1	Silica ( $\text{SiO}_2$ )	63.0
2	Aluminum oxide ( $\text{Al}_2\text{O}_3$ )	13.3
3	Calcium oxide ( $\text{CaO}$ )	8.43
4	Potassium oxide ( $\text{K}_2\text{O}$ )	9.95
5	Ferry oxide ( $\text{Fe}_2\text{O}_3$ )	5.20

### Chemical Composition of Sodium Silicate

The chemical composition of sodium silicate that use as raw material to precipitated silica production in this research is presented in the following table 3.

Table 3. Chemical composition of Sodium Silicate Solution

No	Parameters	Concentration (%)
1	Silica ( $\text{SiO}_2$ )	2.16
2	Sodium oxide ( $\text{Na}_2\text{O}$ )	11.68

### The Effect of Ph Process and Ratio Sodium Silicate/Water to Silica Content of Precipitated Silica Product

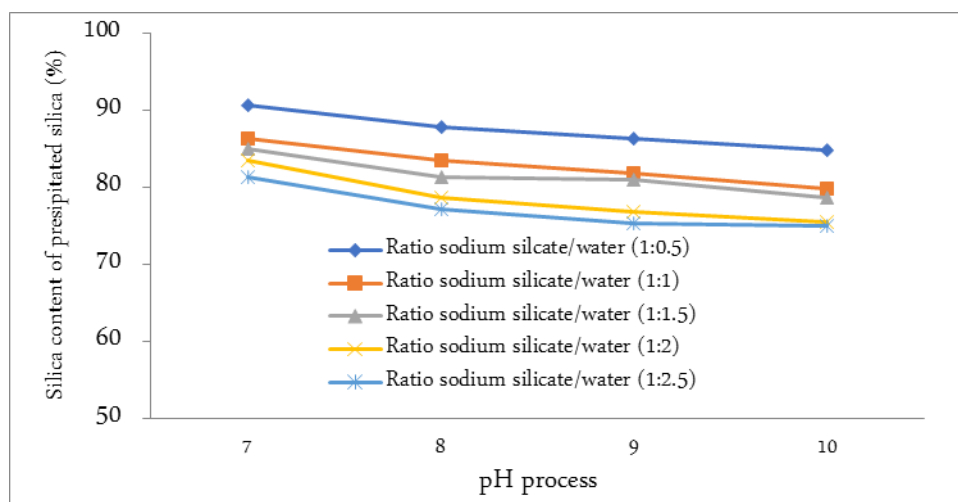


Figure 2. Correlation increasing of pH and ratio sodium silicate/water to silica content of precipitated silica product (%)

Figure 2 shows that the increasing of pH silica content on precipitated silica will decrease because the increasing of pH will need more  $\text{CO}_2$  gas injected and will produce more sodium carbonate salt. the increasing of sodium silicate/water ratio, silica content on precipitated decrease also, because increasing of sodium silicate/water ratio amount of silica will decrease. Base on silica content, the best process condition is pH 7 and sodium silicate/water ratio 1: 0.5, the silica content 90.7 %.

### The effect of pH process and ratio sodium silicate/water to aluminum oxide content of precipitated silica product

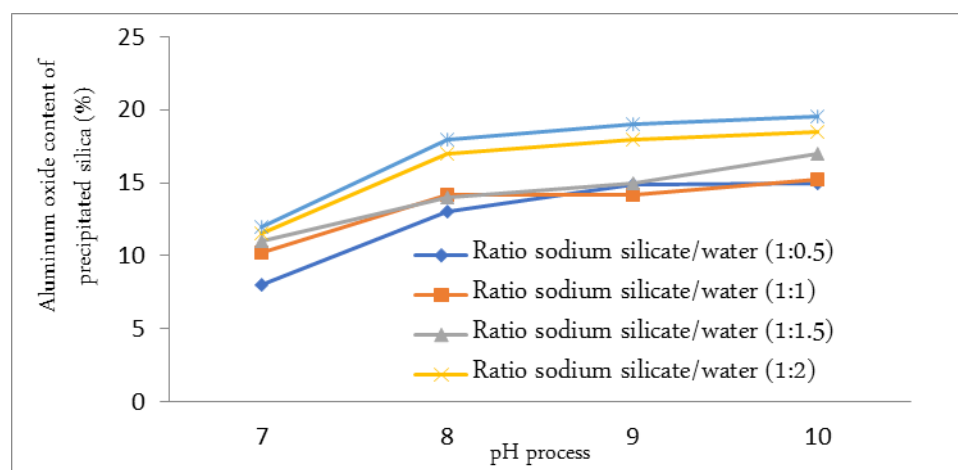


Figure 3. Correlation increasing of pH and ratio sodium silicate/water to aluminum oxide content of precipitated silica product (%)

Figure 3 shows that the increasing of pH, aluminum oxide content increased, this is due to the decreasing amount of silica while the aluminum oxide content remains due to no reaction. Base on silica content, the best process condition is pH 7 and sodium silicate/water ratio 1:0.5, the silica content 92 %. Aluminum oxide content 8.1%.

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

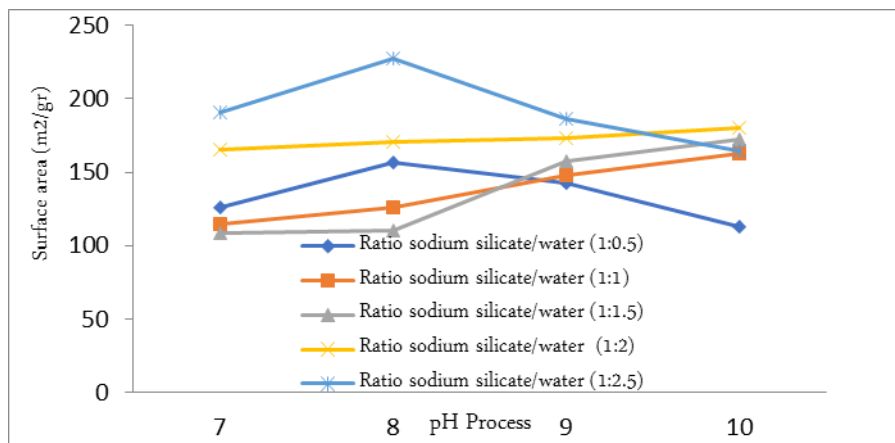


Figure 4. Correlation increasing of pH and ratio sodium silicate/water to surface area of precipitated silica product (%)

Figure 4 shows that the increasing in pH influences the surface area of the precipitated silica product, the largest surface area occurs at pH 8, sodium silicate/water ratio 1: 2.5 but the silica content is low about 77.3%.

### CONCLUSION

Based on data research results can be concluded as follows:

1. The pumice used as a silica source contains 66.3% silica and 13.3% aluminum oxide
2. The best pH of the precipitate silica production is 8
3. The silica content of the precipitate silica product is 90.1% and the aluminum oxide is 8.1%
4. The surface area of the precipitate silica product is 108–227 m<sup>2</sup>/g

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