

# The Effect of Various Acids to the Gelation Process to the Silica Gel Characteristic Using Organic Silica

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**Abstract.** Bagasse ash is solid waste of cane sugar industry which contain of silica more than 51%. Some previous study of silica gel from bagasse ash have been conducted often and been applied. This study concerns about the effect of various acid used in the process of gelation to the characteristic of silica gel produced. Then, this silica gel will be used as adsorbent. As that, the silica gel must fulfill the requirements of adsorbent, as have good pores characteristics, fit in mesoporous size so that adsorbent diffusion process is not disturbed. A fitted pores size of silica gel can be prepared by managing acid concentration used. The effect of acid, organic acid (tartaric acid) and inorganic acid (hydrochloric acid), is investigated in detail. The acid is added into sodium silicate solution in that the gel is formed, the pores structures can be investigated with BET, the crystal form is analyzed with XRD and the pore structure is analyzed visually with SEM. By managing the acid concentration added, it gets the effect of acid to the pore structure of silica gel. The bigger concentration is, the bigger the pore's size of silica gel produced.

## 1. Introduction

Bagasse, the solid waste of cane sugar industry cause environmental pollution if it is not handled well. All this time, bagasse is utilized as animal feed, brick or landfill. Bagasse is also utilized as boiler fuel which the result of the burning in a form of bagasse ash is containing of high level of silica and is potential as source of silica. Material exploration consisting of silica is regularly conducted, such as rice husk [1,2,3] bagasse ash [4,5,6] and geothermal sludge [7]. Silica gel prepared from bagasse ash has been conducted and applied for wastewater purifier [4]. Many methods developed to get expected characterization based on the needs, e.g., as adsorbent. This research is aiming to develop the technique for expanding surface area and pore size by adding organic and inorganic acids as the controller of pore structure and external morphology in the synthesized mesoporous silica from bagasse ash. Acid added to the gel-forming process gives effect as a template. The pores are formed when the acid enters the silica matrix and is trapped inside. Strong acids provide the effect of accelerating the condensation polymerization so that the formation of silica webs has not been uniformly formed. The otherwise, the polymerization condensation process proceeds more slowly when a weak acid is used. The pores are formed when the acid is released from the silica matrix in the process of washing the gel [8].



## 2. Material and Method

### 2.1. Materials

Raw material used on this study is bagasse ash from PG. Kebon Agung, Malang, East Java, Indonesia. All chemical used to prepare mesoporous of silica gel from bagasse ash were reagent grade and used without further purification. The sodium hydroxide (NaOH, Merck), tartaric acid ( $C_4H_6O_6$ , Sigma), hydrochloric acid (HCl, Merk). Demineralized water was used during all synthesis and treatment process.

### 2.2. Synthesis of Silica Gel

The process of sodium silicate made from bagasse ash according to the process that has improved before [4]. Briefly, silica in bagasse ash was extracted with 2M NaOH solution on its boiling point under stirring for 1 hour. After that, the mixture was cooled until room temperature. Then, solution was filtered through an ashless filter Whatman No. 41 (Whatman Plc, Kent, England). The filtrate solution was sodium silicate. Sodium silicate solution will be used as raw material for silica gel fabrication. HCl solution 2M is added into sodium silicate solution and the gel is formed. Then, aging at 40 °C for 18 hours. Later, the gel is washed using demineralized water until neutral and then dried in temperature 100 °C for 24 hours. Mesoporous silica obtained and characterizing the pores.

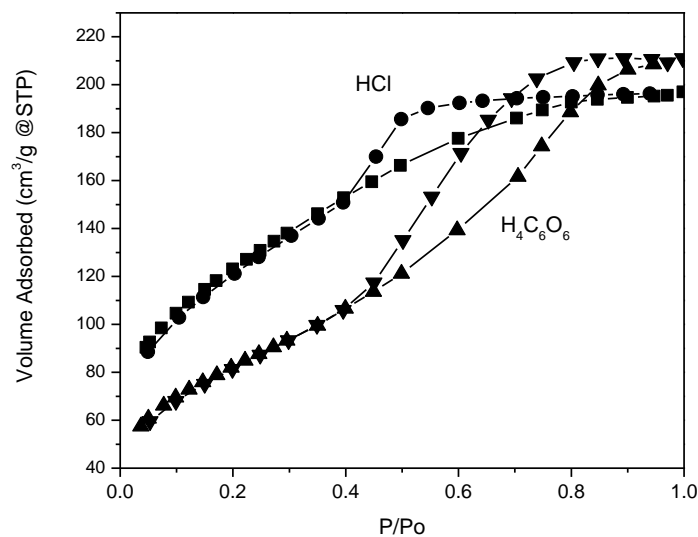
### 2.3. Characterization

N<sub>2</sub> adsorption-desorption analysis was done using a Nova 1200e apparatus (Quantachrome) under liquid N<sub>2</sub> temperature (77 K). The samples were degassed at 150 °C under vacuum for 4 hours. The specific surface area was calculated by the BET method. The pore size distribution analysis was done by the BJH method using the desorption branch of the isotherm. The grafting of the functional groups of adsorbents were analyzed by Fourier transform infrared spectroscopy (FTIR, Shimadzu 8400s), using KBr pellets. X-ray diffraction (XRD) analysis was used to obtain information on the diffraction model and particle's crystallography.

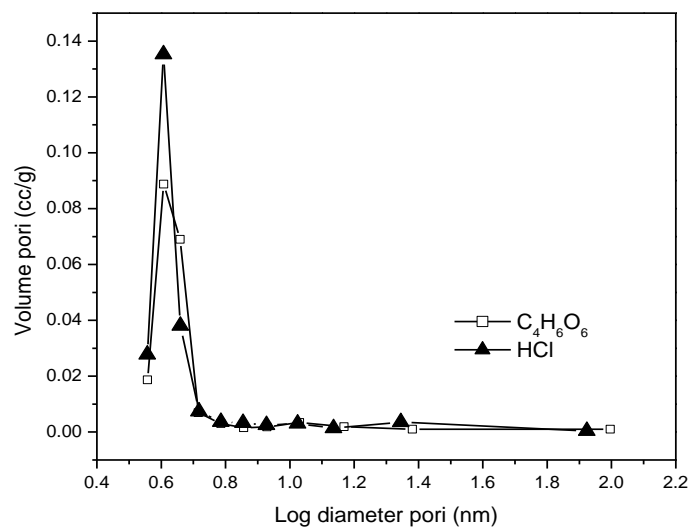
## 3. Result and Discussion

Isotherm adsorpsi-desorpsi of nitrogen of silica gel prepared using HCl and  $C_4H_6O_6$  is shown in Fig. 1. The both curve are identical and according to IUPAC classification type IV. The isotherm of silica prepared HCl has low slope and narrow hysteresis. This isotherm different if silica prepared with  $C_4H_6O_6$ , the hysteresis more wider. It indicated that  $C_4H_6O_6$  has ability to increase the pore. So, adsorption capacity of nitrogen in silica with  $C_4H_6O_6$  was higher than silica with HCl. It means that the silica gel which is fabricated by pH control has uniform pore size.

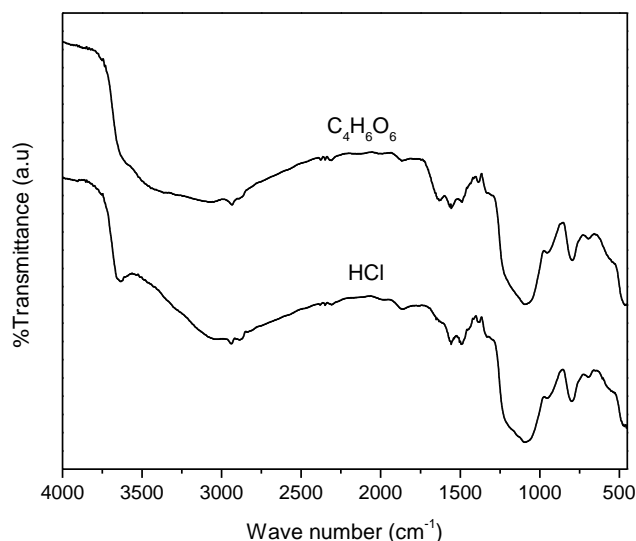
The typical pore size distributions of silica gel prepared by hydrochloric acid (HCl) and tartaric acid ( $C_4H_6O_6$ ) are shown in Fig. 2. The distribution is narrow indicating that the pore size is nearly uniform. the pore size distribution curve on silica prepared with HCl indicates that many particles are 0.62 nm in size, which also means that the pores formed are smaller than those prepared with tartaric acid.



**Figure 1.** Isoterm adsorption-desorption  $\text{N}_2$  in silica gel prepared from bagasse ash using HCl and  $\text{C}_4\text{H}_6\text{O}_6$

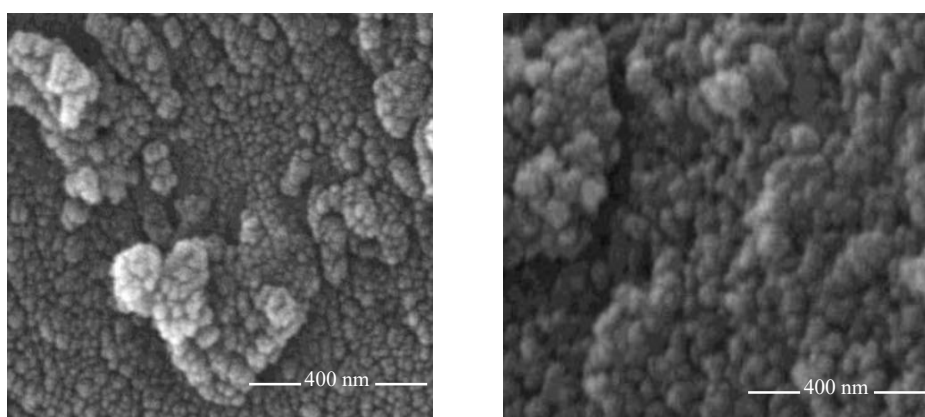


**Figure 2.** Typical pore size distribution of porous silica prepared by various acids (HCl and  $\text{C}_4\text{H}_6\text{O}_6$ )



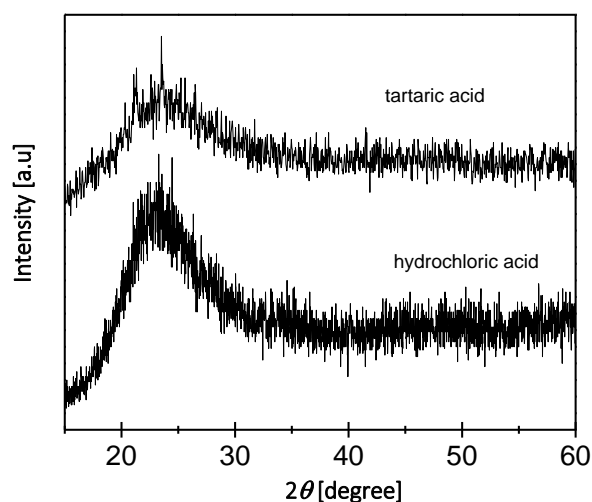
**Figure 3.** FTIR Spectrum of silica using HCl and C<sub>4</sub>H<sub>6</sub>O<sub>6</sub>

Figure 3 shows the FTIR spectra of the sample prepared by various acid treatments. It showed that the curve of silica prepared using HCl and C<sub>6</sub>H<sub>6</sub>O<sub>6</sub> are identical. The peaks appearing in 500–1700 cm<sup>-1</sup> are typical of silica gel (800, 960, 1090 and 1630 [9]). The results show that functional groups of silica products obtained from various acids are similar to each other. The peak at 805 cm<sup>-1</sup> related to the Si–OH bond, and 1115 cm<sup>-1</sup> related to the Si–O bond. The O–H stretching vibrations appear in the region of 3200–3700 cm<sup>-1</sup>. The band resulted from silanol OH groups and water bound to the silica surface by hydrogen bonding.



**Figure 4.** SEM image of silica gel prepared from bagasse ash using HCl (left) and C<sub>4</sub>H<sub>6</sub>O<sub>6</sub> (right)

Figure 5 shows SEM image of silica gel prepared from bagasse ash using HCl and C<sub>4</sub>H<sub>6</sub>O<sub>6</sub>. This figure to further characterize the physical properties of the prepared silica gel. That, the silica prepared using HCl and C<sub>4</sub>H<sub>6</sub>O<sub>6</sub> were irregular in shape. In addition, XRD analysis indicated that the silica gel were amorphous, which was confirmed by the absence of any ordered crystalline structure, at 23 – 25 (2 $\theta$  degree) show in Fig. 6.



**Figure 5.** X-ray diffraction patterns of silica prepared from bagasse ash using HCl and C<sub>4</sub>H<sub>6</sub>O<sub>6</sub>

#### 4. Conclusion

Base on the research finding, it can be concluded that acid addition is significantly influenced to pore structure of silica. The organic acid (tartaric acid) gives more effect to extend pore size than the inorganic acid (hydrochloric acid).

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