Phosphorus Elimination at Sodium Silicate from Quartz Sand Roasted with Complexation using Chitosan-EDTA

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Abstract: A phosphorus elimination from sodium silicate solution has been studied. Phosphorus elimination was performed by adding chitosan-EDTA to remove cation phosphorus. Characterization of chitosan-EDTA material was performed using FT-IR, while the decreasing level of phosphorus content was analyzed by quantitative analysis using spectrophotometer UV-Vis refers to SNI 06-6989-2004. The results showed that the content of the sodium silicate can be reduced up to 67.1% through Chitosan-EDTA complexation with phosphorus.

1. Introduction
The development of the photovoltaic industry greatly improved after the 2000s. In 2005 the production of the world's photovoltaic devices has reached 1787 MW. It is planned that by 2020 the production of photovoltaic devices will be increased to 18 GW. In 2005, over 90% of the photovoltaic devices production (solar cells) was made using crystalline silicon. So, now the supply of solar cell grade is an urgent need. Development of low cost silicon production and purification techniques is essential.

One of the source cheap silicon solar cell grade is quartz sand. In Indonesia, high quality quartz sand is available in Belitung (SiO$_2$ = 98%), Kendawang (SiO$_2$ = 99%), Samarinda and Kutai Kertanegara (SiO$_2$ = 98%) and Mandor (SiO$_2$ = 96%). Lower quality quartz sand is found in Tuban and Banten. Through of the roasted process, the quartz sand is processed into high purity silica to form sodium silicate, but in the sodium silicate there are still minor impurities that must be removed in order to be used as a solar panel material [1].

The common impurities found in natural silicon are Fe, Al, Ca, Ti, P and B. These impurity elements can be reduced by metallurgical methods. Physically, the metal elements with the price of small segregation coefficients are more easily separated from silicon through a sedimentation process, such as Ca and some Fe, Ti and Al. P and B are still in the silicon material will affect the efficiency of solar cell conversion. The minimum requirement of silicon purity of quartz sand silica for solar panels must reach SiO$_2$ 99.999%, with the content of boron and phosphorus elements zero [2].

Phosphorus elements that have speciation from PH$_3$ to P$_2$O$_5$ with oxidation numbers ranging from -3 to +5 make phosphorus having its own technique for the reduction process. In this research, the decrease of phosphorus impurities on sodium silicate solution of roasted sand quartz was done by
adding chitosan-EDTA and coagulant. The chitosan-EDTA will bind the cation phosphorus species while the coagulant phosphorus anion species [3,4]. The process of coagulant addition using two coagulants is aluminum sulphate \([\text{Al}_2(\text{SO}_4)\cdot 18\text{H}_2\text{O}]\) and calcium hydroxide \([\text{Ca}(\text{OH})_2]\) [5,6,7,8,9]. Then a variation of coagulant concentration was used to decrease the optimal phosphorus content.

2. Experimental

2.1. Process of sodium silicate solution

100 grams of sodium silicate salt dissolved in 1000 mL of boiling water. The mixture is placed inside the closed system and stirrer for 2 hours. The condition maintained at the distilled boiling water. The sodium silicate solution is filtered and diluted in a 1000 mL measuring flask tough filtration. Three sodium silicate solutions (solution A, B, C) were collected at 100 mL for each solution to identified the phosphorus content using UV-Vis spectrophotometer.

2.2. Synthesis and characterization of chitosan-EDTA

2 grams of chitosan were dissolved in 200 mL of 0.1% acetic acid until dissolved, 10 grams of EDTA was added. The chitosan-EDTA solution distilled for 24 hours, and centrifuge to form precipitate. The precipitate is then dried and in a desiccator to remove the water. The dry precipitate then weighed, and characterized by FTIR. The centrifuged solution concentrated and characterized by FTIR and TGA/DTA.

2.3. Reduction of phosphorous using Chitosan-EDTA

The optimal comparison results of chitosan-EDTA were dissolved in 100 mL of 0.1% acetic acid and distilled for 24 hours. Each of 100 ml Sodium Silicate solution (solution A, B, C) were added with 50 mL of chitosan-EDTA solution and stirred slowly for 30 minutes to form chitosan gel-EDTA. Separate filtrate with chitosan gel-EDTA formed by filtering process. Then filtrate sodium silicate in the analysis of its phosphorus content with UV-Vis spectrophotometry.

3. Result and Discussion

Sodium silicate from roasted quartz sand dissolved with aquades that have been boiled. Boiled aquades used to soluble the sodium completely. After dissolved and filtered, a clear-colored sodium silicate solution was obtained. After the sodium silicate sample solution was prepared, chitosan-EDTA synthesis was performed. The ratio of chitosan-EDTA in this process is \(= 1: 5\) (chitosan-EDTA) [10]. The reaction that may occur between chitosan and EDTA (Fig. 1).

![Figure 1. Chitosan-EDTA formation reaction](image-url)
Characterization of Chitosan-EDTA using FT-IR were showed in Fig. 2.

![Figure 2. FTIR chitosan-EDTA](image)

FT-IR spectroscopy of chitosan-EDTA in Fig. 2, shows that chitosan has an uptake of N-H group in the 3300-3500 cm\(^{-1}\) range, it refers to the chitosan-EDTA formation. In addition, the presence of C=O groups in the range 1600-1700 cm\(^{-1}\), shows the existence of EDTA on chitosan-EDTA. Then in the range 2900-3000 cm\(^{-1}\), shows the CH\(_2\) group on chitosan-EDTA that has been successfully formed.

Thermogram data from DTA/TGA characterization (Fig. 3) shows the difference between chitosan and chitosan-EDTA. With the addition of EDTA, the thermal properties of chitosan will increase. Chitosan degradation process starts from 100-200°C, but for chitosan-EDTA starts degraded 200-300 °C with an upward DTA peak (exothermic reaction). These different thermogram characteristics indicate the changing properties of the material that will increase the phosphorous binding of EDTA.
By using the absorbance measurement method with UV-Vis spectrometer that refers to SNI 06-6989-2004, the average concentration of phosphorus is shown in Table 1.

**Table 1.** The results of measurement of phosphorus content in sodium silicate solution after chitosan-EDTA addition

| Sodium Silicate Solutions | Mean of Initial Concentration (ppm) | Mean of Final Concentration (ppm) | Mean of Effectivities (%) |
|---------------------------|-------------------------------------|----------------------------------|----------------------------|
| Solution A                | 0.0589 ± 0.00327                    | 0.0194 ± 0.00119                 | 67.063                     |
| Solution B                |                                     |                                  |                            |
| Solution C                |                                     |                                  |                            |

From the data of initial phosphorus concentration and chitosan-EDTA concentration can be calculated the decreasing of phosphorus level up to 67.063%. This can happen because sodium silicate solution contain several phosphorus that may not converted into phosphorus cation (phosphonium). Actually chitosan-EDTA is able to attract positively charged ions with three active carboxylate groups (COO\(^-\)) such as phosphonium (PH\(_4^+\)) in sodium silicate solution. However, phosphorus is more easily deposited into phosphate ions (PO\(_4^{3-}\)) by forming strong bonds with O atoms on silicate compounds rather than being pentated into phosphonium (PH\(_4^+\)).

### 4. Conclusion

Chitosan-EDTA can be used to decrease the phosphorus content in sodium silicate. The decrease of phosphorus level up to 67.063%. The decrease of phosphorus content showed very low levels due to the chitosan-EDTA ability to bind the dissolved phosphorus in sodium silicate solution.
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