

# OPTIMIZATION OF BEST COMPOSITE PRIMER AMINE IN HYBRID SILIKA-AMINE SYNTHESIS

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## ABSTRACT

Silica, one of most porous materials having a unique silanol group, that can be replaced with other functional groups for more specific purposes. One of highlights is replacement of silanol function groups with amine groups. Amines are highly reactive to the carbon dioxide gas, so the silica amine hybrid has potential to absorb the carbon dioxide gas. Amine is often used is a type of primary amine. This research is development of previous research. The results data showed a significant relationship between type and composition of modifying agent on characterization of amine silica hybrid. This research is focused on optimizing best amine primer composition on hybrid synthesis of silica amine to surface area and pore size. As modifying agent used APTS in ethanol, where the integration of numerical analysis, matrix computing, signal processing and graphics are expressed in mathematical notation using matrix laboratory to solve optimization problem. It was concluded that ethanol solvent yields adsorbents with better characterization than water solvents, whereas the addition of APTS as a source of amine compounds gives a significant effect where increase in APTS increments will decrease pores produced.

## INTRODUCTION

Porous material is a much researched material support for purposes of adsorption processes such as zeolite and silica. For more specific uses, silica gives better value because of its uniqueness. The silanol groups present on the silica surface can easily be replaced with other functional groups. One of them with the amine group, which is very reactive to the carbon dioxide gas. As an adsorbent of carbon dioxide gas, the silica may be modified with amine groups. Silica characterization greatly influences modification process. To obtain the silica with the desired surface area and pore size can be done using a template, such as PEG (Polyethylene glycol) and gelatin (Rahman dkk., 2015; Balgis dan Setyawan, 2012).

Another affecting parameter carbon dioxide gas adsorption process is successful loading of amines to replace silanol groups. The more amines that can replace silanol group, the more carbon dioxide gas that succeeds in the adsorp. For this purpose, this study focused on optimizing best amine primer composition on synthesis of shybrid silica amine to surface area and pore size. As the modifying agent used APTS in ethanol, where the integrase of numerical analysis, matrix computing, signal processing and graphics are expressed in mathematical notation using matrix laboratory to solve the optimization problem.

Matrix Laboratory is one of expert system based software used to solve optimization problems. Use of software in solving optimization problems is very important. Especially when it involves many iterations in finding the optimum solution of a problem. By utilizing Matrix Laboratory, users can perform data analysis, develop algorithms and create models and applications.

Objective of this study was to obtain the best primary amine composition in ethanol cosolvent in hybrid synthesis of silica amine as carbon dioxide gas adsorbent and to solve optimization problem using matrix laboratory.

## **METHODOLOGY**

This study was conducted in two stages, where in the first stage experiments were conducted for one variable ie amine composition (amine ratio in ethanol solvent) in the synthesis of organic silica-based carbon dioxide adsorbent from bagasse ash.

After obtained the data for one variable amine composition then performed a simulation to get the best composition if using secondary amine and tertiary amine.

## **MATERIAL**

Raw material in this study was biogenic silica extracted from bagasse ash PG Kebon Agung Malang. The chemicals used are NaOH (Merck), Ethanol (Merck), Amino Propyl Triethoxy Silane (APTS) (Merck, 99%), Hydrochloric Acid (Merck). Materials are used without initial treatment.

## **EKSPERIMENT**

Biogenic silica from bagasse ash was extracted by alkali using developed methods in previous research (Rahman et al, 2015). As the modifying agent used APTS in ethanol, where the integrase of numerical analysis, matrix computing, signal processing and graphics are expressed in mathematical notation using the matrix laboratory to solve optimization problem.

## **CHARACTERIZATION**

Resulting silica amine hybrida was analyzed by BET to obtain pore size and surface area information. The determination of success of the amine group to replace the silanol group was analyzed by FTIR. While the amine capacity that successfully modified the silica surface was analyzed with TGDTA. To obtain information visually used image imaging with SEM.

## **MATRIX LABORATORY**

Use of software in solving optimization problems is very important. Especially when it involves much iteration in finding the optimum solution of a problem. Matrix laboratory is one of the most widely used software to solve optimization problems where the integrase of numerical analysis, matrix computation, signal processing and graphics are expressed in the form of mathematical notation.

## **RESULTS AND DISCUSSION**

This research was conducted in two stages, where in the first stage experiment was conducted for one variable that is amine composition (amine ratio in ethanol solvent) in synthesis of organic silica-based carbon dioxide adsorbent from ash bagasse.

After obtained data for one variable amine composition then performed a simulation to get best composition if using secondary amine and tertiary amine.

In this report, the experimental data for variation of the composition of primary amine compound to solvent are used, ethanol using 5 (five) compositions (1:5, 2:5, 3:5, 4:5 and 5:5).

All compositions are repeated up to 3 (three) times to obtain optimal results. After obtained data replication then taken average of the data.

**Tabel 1. BET Analysis Using Water for Washers**

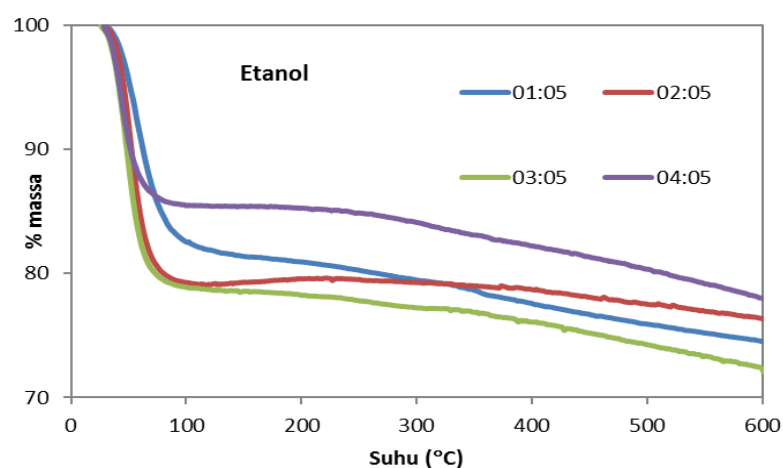
Ratio APTS:etanol	Surface Area (A) m <sup>2</sup> /g	Pore Volume cc/g	Pore Diameter nm
1 : 5	516.482	0.429	3.796
2 : 5	418.138	0.53	4.309
3 : 5	371.514	0.576	4.322
4 : 5	384.573	0.602	4.323
5 : 5	380.332	0.582	4.333

Source: Analysis Results

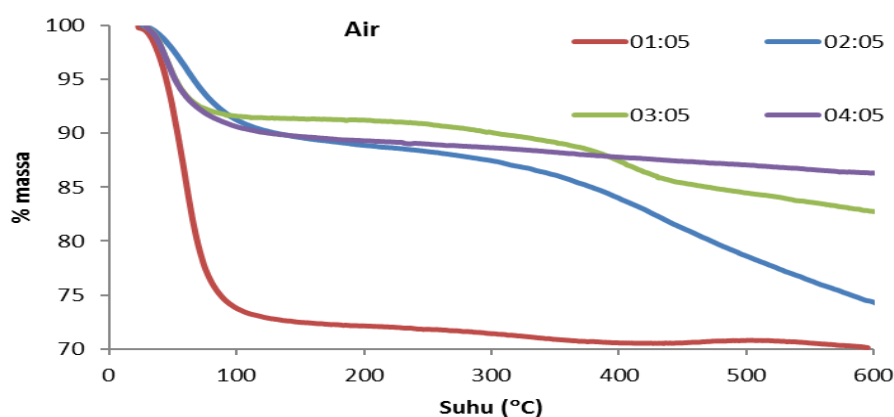
**Tabel 2. BET Analysis Using Ethanol for Washers**

Ratio APTS:etanol	Surface Area (A) m <sup>2</sup> /g	Pore Volume cc/g	Pore Diameter nm
1 : 5	516.482	0.429	3.796
2 : 5	418.138	0.53	4.309
3 : 5	371.514	0.576	4.322
4 : 5	384.573	0.602	4.323
5 : 5	380.332	0.582	4.333

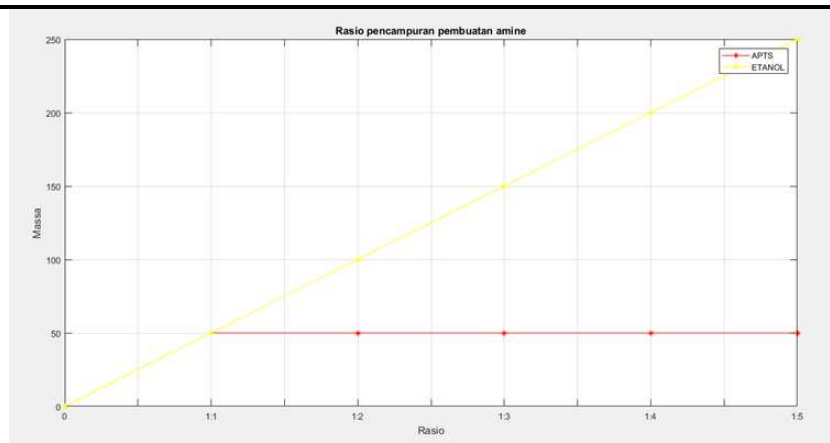
Source: Analysis Results



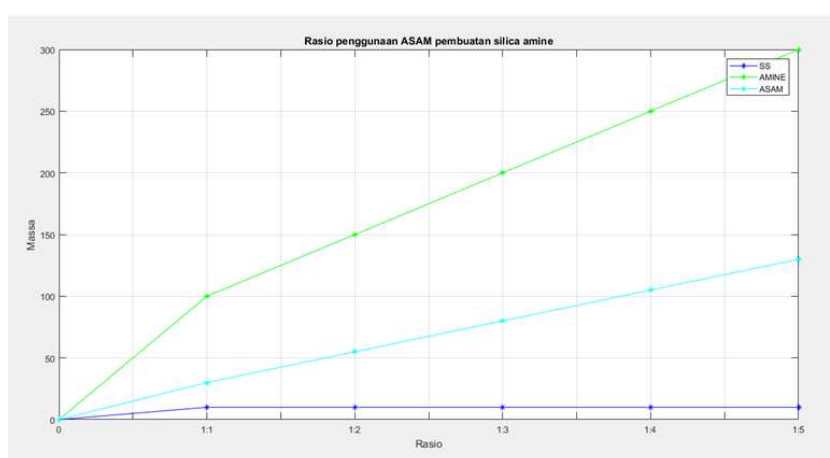
**Figure 1: Results of Sample TGA Analysis Prepared with Variations of APTS Composition on Solvent Using Ethanol Wash Media**



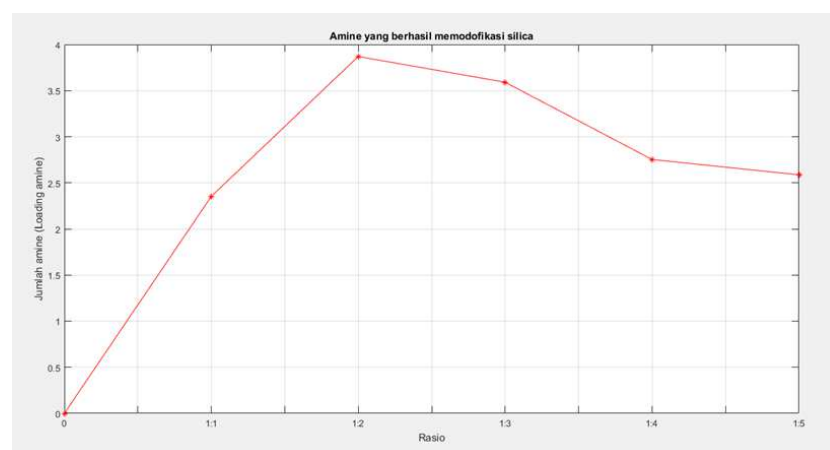
**Figure 2: Results of Sample TGA Analysis Prepared with Variation APTS Composition Against Solvent Using Waterwash Media**



**Figure 3: Ratio of Mixing APTS and ETANOL for Making AMINE**



**Figure 4: Use ACID Ratio in Formation Process of Silica Amine Gel**



**Figure 5: Loading Amine Gained on Various Variations of APTS Ratio in Ethanol**

Synthesized silica-amine from bagasse ashes with APTS as an amine source was successfully performed. APTS variations in ethanol co-solvent have significant effect on amine that successfully modify silica. It also affects the silica-amine adsorption capacity of carbon dioxide gas. Using matrix laboratory simulation can be estimated the possibility of the influence of amine species, both secondary and tertiary to silica-amine hybrids from ash bagasse.

## CONCLUSIONS

Silica Amine from bagasse ashes is done by varying APTS as the primary amine source in ethanol co-solvent. Combined experiments with matrix laboratory simulations provide a solution to estimate the results

of amine-silica hybrids from bagasse ash with other (secondary and tertiary) amine variations to amine loading and adsorption capacity to carbon dioxide gas.

## REFERENCES

- I. Affandi, S., Setyawan, H., Winardi, S., Purwanto, A., Balgis, R. (2009). "A facile method for production of high-purity silica gels from bagasse ash", *Adv. Powder Technol.*, 20, 468-472.
- II. Anbia M., Hoseini V., Mandegarzad S. (2012). "Synthesis and characterization of nanocomposite MCM-48- PEHA-DEA and its application as CO<sub>2</sub> adsorbent", *Korean J. Chem. Eng.* 29, 1776–1781.
- III. Brinker C.J., Scherer G.W., (1990). "Sol-Gel Science", *Academic Press, New York*.
- IV. Che, S., Garcia-Bennett, A.E., Yokoi, T., Sakamoto, K., Tatsumi, T. (2003). "A novel anionic surfactant templating route for synthesizing mesoporous silica with unique structure", *Nature materials*, 2, 801-805.
- V. Coradin, T., Bah, S., Livage, J. (2004). "Organic/silicate interactions: from nanoparticles to composite gels", *Coll. Surf. B*, 35, 53–58.
- VI. Dorcheh, A.S., Abbasi, M.H., (2008), "Silica aerogel; synthesis, properties and characterization", *J. Mater. Proces. Technol.*, 199, 10-26.
- VII. Guerrero S, Da'na R., Sayari, A., (2008) "New insights into the interactions of CO<sub>2</sub> with amine-functionalized silica", *Ind. Eng. Chem. Res.* 47, 9406–9412.
- VIII. Miyamoto M., Takayama A., Uemiya S., Yogo K. (2012). "Study of Gas Adsorption Properties of Amidoamine-Loaded Mesoporous Silica for Examining Its Use in CO<sub>2</sub> Separation," *J. Chem. Eng. Jpn.* 45, 395–400.
- IX. Rahman, NA., Widhiana, I., Juliastuti, SR., Setyawan, H. (2015). "Synthesis of Mesoporous Silica with Controlled Pore Structure from Bagasse Ash as A Silica Source", *Colloids Surf., A*, 476, 1 – 7.
- X. Setyawan, H., Balgis, R. (2012). "Mesoporous silicas prepared from sodium silicate using gelatin templating", *Asia-Pac. J. Chem. Eng.* 7, 448–454.
- XI. Witton, T., Chareonpanich, M., Limtrakul, J. (2008). "Synthesis of bimodal porous silica from rice husk ash via sol-gel process using chitosan as template", *Mater. Lett.* 62, 1476–1479.