

Challenges in Chemistry for Sustainable Development



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**CHALLENGES IN CHEMISTRY
FOR SUSTAINABLE DEVELOPMENT**

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Inorganic Chemistry

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Synthesis and characterization of quaternary ammonium salt on silica gel prepared from rice hull ash

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Abstract: In the present work, synthesis of quaternary ammonium salt on silica gel prepared from rice hull ash (RHA) and used for anion exchanger was investigated. Sodium silicate (Na_2SiO_3) solution was prepared by destruction of rice hull ash with 4 M NaOH solution. The synthesis of ethylenediamine-silica hybrid (ESH) was carried out by adding 3 M HCl solution to the mixture of (3-ethylenediaminopropyl)-trimethoxysilane (EDAPTMS) and sodium silicate solution till pH 7. The gel was characterized by using infrared spectroscopy (FTIR), X-ray Diffraction (XRD) and Surface Area Analyzer (SAA). The ESH then was reacted using methyl iodide and sodium chloride solution to form quaternary ammonium salt (ESH⁺/Cl⁻). The salt was then characterized using infrared spectroscopy (FTIR), Thermogravimetry (TG), Kjeldahl analysis and potentiometric titration. Result of characterization with FTIR showed that ESH had been successfully synthesized, indicated by the presence of characteristic absorbance of functional groups silanol (Si-OH), siloxane (Si-O-Si), amine ($-\text{NH}_2$) and methylene ($-\text{CH}_2$). The XRD data showed amorphous structure of ESH. From SAA result, the specific surface area and total pore volume of ESH was obtained. The ESH⁺/Cl⁻ had been successfully synthesized based on the data from FTIR, Kjeldahl analysis and potentiometric titration. From TG, it was discovered that ESH⁺/Cl⁻ was thermally stable.

Keywords: synthesis, quaternary ammonium salt, silica gel, rice hull ash.

Introduction

Being an agricultural country, Indonesia has a side advantage due to the amount wastes from the farming activities such as rice hull ash (RHA) that can be utilized as a source of silica to make an adsorbent. Rice hull ash (RHA) contains over 90% silica and can be an economically viable raw material for the production of silicates and silica gel. Adsorbent has been prepared from RHA as a by-product of brick burning process. The use of RHA has more advantages than quartz, because of its high crystallinity and stability so the destruction must be done at high temperature (1300°C) [1].

Silica is of particular interest as it doesn't swell and has good mechanical strength and thermal stability. Among the more promising highly developed surfaces are the-silica attached organosilanes which are widely used in various technologies. Silica gel can be synthesized through sol-gel process by the condensation of sodium silicate to an acid solution.

Silica gel has silanol (Si-OH) and siloxan (Si-O-Si) that can be used for many adsorption processes. On the other hand, the capability of silica gel in adsorption is limited. It is because of the active sites, both functional groups of silica gel only adsorb hard metal ions [2].

Recently, the development of silica gels have become modified with suitable functional groups, with the purpose of application in the adsorption of metal ions from solutions, in preconcentration processes and further analysis of metal ions at trace levels in aqueous and non-aqueous solutions and as materials to pack columns for use in high-pressure liquid chromatography [3-4].

The application of these materials in chromatography includes adsorption, preconcentration, and ion-exchange techniques; in the latter, charged groups are introduced onto the silica surface by chemisorption. Thus, cation or anion exchangers are obtained depending on the immobilized functional group charge. The application of these materials in ion exchange is dependent on their exchange capacity, stability and selectivity [5].

The exchange capacity is governed by the concentration of surface hydroxyl groups before the modification, by extension in which the reactions occur between the silanol groups and the modifier, and by the nature of the kind of modifier [6]. An ion exchanger anchored onto silica gel is stable in solutions over the pH range 0-8 and shows much more rigidity than the organic matrices and swelling negligible [4]. The selectivity is usually expressed through the separation factor (derivative of the isotherms) and through the selectivity coefficient (retention time).

The anion exchangers immobilized onto silica gel containing primary, secondary or tertiary amine groups show a behavior that is weakly acidic, while the quaternary salts are strongly acidic. In this paper, we report the synthesis and characterization of quaternary ammonium salts using silica gel from the sol-gel processing of rice hull ash and 3-ethylenediaminepropyltrimethoxysilane (EDAPTMS).

Materials and Methods

Material: Rice hull ash (RHA), NaOH (Merck), HCl (Merck), CH_3I (Merck), NaCl (Merck), DMF (Merck), AgNO_3 (Merck), ethylenediaminopropyl-

trimethoxysilane (EDAPTMS), aquadest, filter paper Whatman 42, filter paper, pH indicator universal.

Instrumentation: Infrared spectra of the components were obtained on a FTIR spectrophotometer, Shimadzu FTIR-8201 PC. The crystallinity of the components was obtained using X-Ray Diffractometer. The porosity and specific surface area was obtained by using Surface Area Analyzer (SAA). The TG analysis was made using a Shimadzu Model TGA 50 Thermobalance.

Preparation of Na_2SiO_3 solution: Rice hull ash was scrapped and sieved to obtain RHA 200 mesh. Here in after as much of 20 g RHA were then cleaned by 150 mL HCl 6M and neutralized by aquadest. The outcome was then dried in oven. The clean RHA taken about 20 g and enhanced by 167 mL NaOH 4M (stoichiometri), boiled at the same time swirled with the magnetic bar. After dried, condensation was infused by a porcelain cup and melted at temperature 500°C for 30 minutes. After chilled, it was enhanced by 200 mL aquadest, let for one night and filtered by using Whatman 42. Filtrate yielded represent the condensation of sodium silicate solution (Na_2SiO_3) that will be ready to use as substance for preparing the material.

Hybridization: About 20 mL of sodium silicate solution was put into a plastic glass, enhanced by 1 mL of EDAPTMS. Here in after enhanced by HCl 3M dropwise swirled with the magnetic stirrer until a neutral pH. Gel was hushed for a night, cleaned by aquadest till neutral and dried in oven with the pressure reduction at a temperature of 70°C . After dried, it was scrapped and sieved 200 mesh. The material then referred to as ethylenediamine-silica hybrid (ESH).

Synthesis: Twelve grams of ESH was suspended in 50 mL N,N-dimethylformamide and 4 mL methyl iodide was added gradually. The mixtures were stirred for 2 h in the dark, at room temperature. The solid products were filtered and washed with water, then with a 2% sodium bicarbonat solution, and again with water. The materials were dried under vacuum at 80°C for 6 h. The methylation reactions were repeated four times to ensure a good yield of the desired quaternary ammonium product, $\text{Si}(\text{CH}_2)_3\text{NH}(\text{CH}_2)_2\text{N}^+(\text{CH}_3)_3\text{I}^-$. Finally, about 0.1 g of ESH/T was dispersed in 50 mL of 15% sodium chloride solution. The mixtures were slowly stirred for 5 h at a temperature of 25°C . The solids were filtered, washed repeatedly with water, ethanol and diethyl ether, and then dried in air. The resulting derivative chloride, $\text{Si}(\text{CH}_2)_3\text{NH}(\text{CH}_2)_2\text{N}^+(\text{CH}_3)_3\text{Cl}^-$ then referred to as ESH⁺/Cl⁻.

Characterization: The nitrogen content of the aminated silica gels was determined by the Kjeldahl method. The chloride ion present on ESH⁺/Cl⁻ was determined by potentiometric titration. About 0.2 g of ESH⁺/Cl⁻ was shaken with 50 mL of $0.1 \text{ mol L}^{-1} \text{ HNO}_3$ solution for 1 h. The solids were filtered and washed repeatedly with water until the chloride anion had disappeared. The amount of Cl⁻ present was determined by potentiometric titration with $0.01 \text{ mol L}^{-1} \text{ AgNO}_3$ standard solution. This value corresponds

to the limit amount of adsorbed Cl⁻ on the supports. The infrared spectra of the unmodified silica gel, ESH, ESH⁺/I⁻ and ESH⁺/Cl⁻ were performed by using the pure material pressed disk technique. To obtain information about the thermal stability of the studied materials, the analysis of TG was made by using the dynamic heating method.

Result and Discussion

Preparation of modified silica gel by ethylenediamine group: Preparation of modified silica gel was conducted in two steps, namely sodium silicate solution (Na_2SiO_3) preparation from RHA and the addition of hydrochloride acid solution to form gel. The anion species interaction of silicate with EDAPTMS was anticipated first of all by protonation to oxygen atom of methoxy (-OCH₃) from EDAPTMS and then followed by the anion attack of silicate to the inorganic compound through $\text{S}_\text{N}2$ mechanism. The protonated oxygen atom from methoxy going will cause the atom progressively positive polarize so that it has a tendency to be attacked by negative species of silicate anion. This will cause the formation of its siloxan bonding accompanied with the methanol release [7]. Simple model of the reaction of the formation of ethylenediamine-silica hybrid is described in Figure 1.

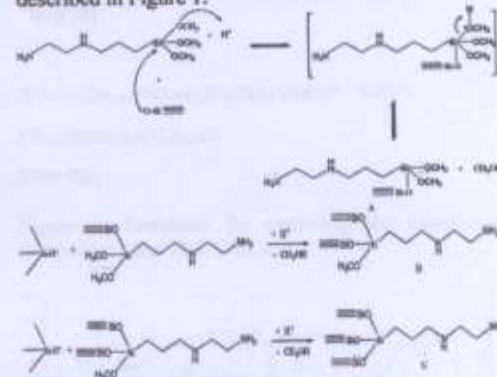


Figure 1: Reaction of the ESH formation

Infrared spectra: The characterization was to identify functional groups in ethylenediamine silica hybrid (ESH) is shown in Figure 2.

Table 1: Interpretation of infrared spectra of materials

Vibrational functional groups for each material (cm^{-1})		Interpretation
SG	ESH	
3425.3	3386.8	Stretching of -OH of Si-OH
-	2954.7	Aliphatic chain of -CH ₂ -
-	1461.9	Chain of C-C from methylene group (-CH ₂ -)
1087.8	-	Asymmetric stretching Si-O from Si-O-Si
956.6	-	Stretching Si-O from Si-OH
802.3	-	Symmetric stretching Si-O from Si-O-Si
466.7	-	Bending Si-O from Si-O-Si

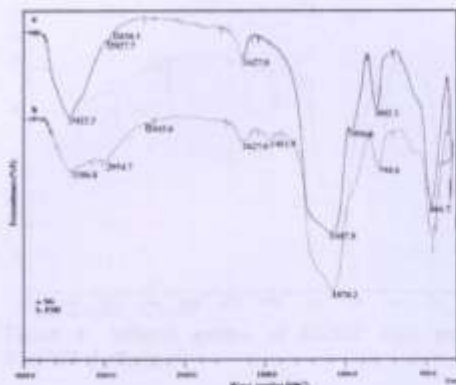


Figure 2: Infrared spectra SG (a) and ESH (b)

X-ray diffraction: The characterization is aimed to identify the crystallinity of materials, as shown in Figure 3.

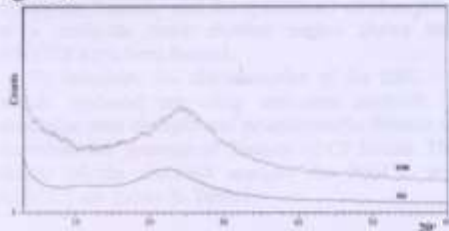


Figure 3. X-ray diffraction of SG and ESH

As shown in Figure 3, diffraction pattern of silica gel and ESH show the wide pattern around $2\theta = 21-23^\circ$, which indicates that the silica gel and ESH are amorphous. The structure of modification of silica gel by organic ligand did not change the structure of silica gels.

Porosity and specific surface area: Determination of specific surface area and pores of the materials of silica gel and ESH can be seen from the results of analysis by using surface area analyzer (SAA). The results of the analysis are shown in Table 2.

Table 2: Data Analysis of Surface Area of Materials

Materials	Specific surface area (m^2/g)	Total pore volume (mL/g)	Pore diameter (\AA)
SG	103,505	0,092	17,826
ESH	98,410	0,083	16,798

From the table above, it is clear that the total pore volume is proportional to the specific surface area of the material, thus, the larger the volume of the pores is, the larger the surface area will be. Silica gel has a surface area of $850 m^2/g$ and pore diameter of 22\AA [1]. The results showed a specific surface area and pore diameter of silica gel is relatively small. This is probably due to gel formation process so fast that the

formation of pores decreases and tends to form a solid. The result of research shows that ESH has a specific surface area smaller than SG. This is possible because the binding occurs between ESH and EDAPTMS compounds that will block the pores so that the smaller the pore size and surface area produces a smaller. In general, both materials are at mesoporous scale 2-50 nm in pore diameter [8].

Synthesis of ESH⁺/Cl⁻: A total of 12 grams of ESH which is then mixed with *N,N*-dimethyl formamide and methyl iodide is added gradually. Then, the solution is stirred for 2 h in darkness at room temperature. Solids obtained then filtered and washed by using water, sodium bicarbonate and re-washed by water. The use of sodium bicarbonate solution is to remove organic impurities present in the ESH⁺/T. Methylation is carried out four times. Formed solids dried at a temperature of $80^\circ C$. After it formed a solid solution of sodium chloride 15% is added, stirring for 5 h at a temperature of $25^\circ C$. The quaternary ammonium salt formed later referred to as ESH⁺/Cl⁻. The reaction that forms on the synthesis of the quaternary ammonium salts are as follows:

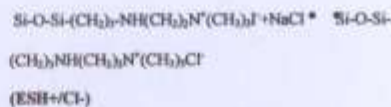
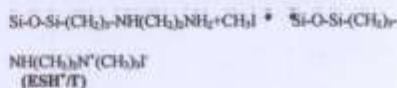
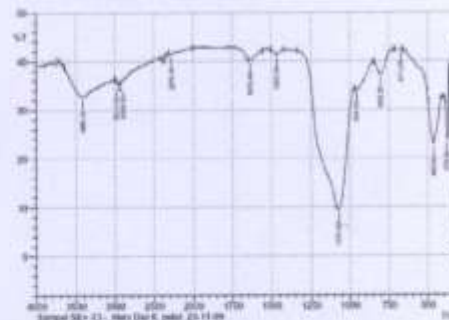


Figure 4: Reactions for obtaining the quaternary ammonium salts (ESH⁺/Cl⁻).



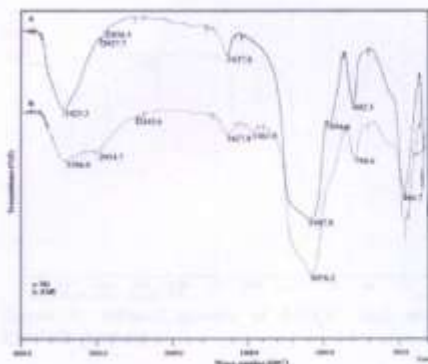


Figure 2: Infrared spectra SG (a) and ESH (b)

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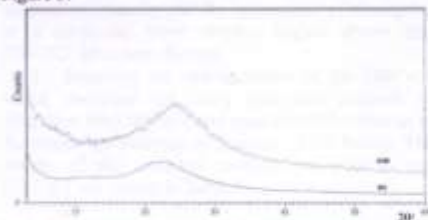


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Synthesis of ESH/CT: A total of 12 grams of ESH which is then mixed with *N,N*-dimethyl formamide and methyl iodide is added gradually. Then, the solution is stirred for 2 h in darkness at room temperature. Solids obtained then filtered and washed by using water, sodium bicarbonate and re-washed by water. The use of sodium bicarbonate solution is to remove organic impurities present in the ESH/T. Methylation is carried out four times. Formed solids dried at a temperature of 80°C. After it formed a solid solution of sodium chloride 15% is added, stirring for 5 h at a temperature of 25°C. The quaternary ammonium salt formed later referred to as ESH/CT. The reaction that forms on the synthesis of the quaternary ammonium salts are as follows:

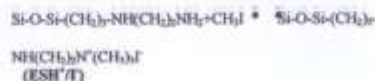
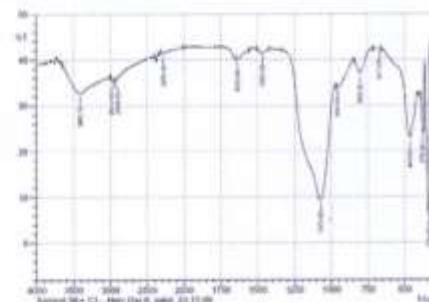


Figure 4: Reactions for obtaining the quaternary ammonium salts (ESH/CT).



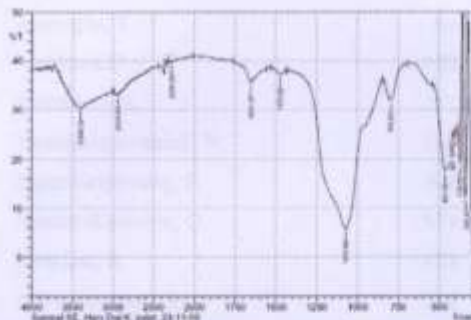


Figure 5: Infrared spectra of ESH⁺/T (up) and ESH⁺/Cl⁻ (bottom)

The main evidence from the infrared spectra is the strong band that observed at 1651 cm⁻¹ in ESH⁺/T which can be identified as bending vibrations of -NH₂ group. This frequency is disappeared in the ESH⁺/Cl⁻. The existence of a shift in absorption, changes in absorption intensity and the appearance of absorption at a particular wave number region shows that ESH⁺/Cl⁻ have been formed.

To determine the characteristics of the ESH⁺/Cl⁻ which produced are using analytical methods to determine total nitrogen and potentiometric titration to determine the presence or absence of Cl⁻ bound. The results of the chemical analysis for ESH⁺/T and ESH⁺/Cl⁻ are shown in Table 3.

Table 3: Nitrogen and Chloride Analyses of Materials

Materials	N (mmol g ⁻¹)	Cl ⁻ (mmol g ⁻¹)
ESH ⁺ /T	2.857	-
ESH ⁺ /Cl ⁻ (15%)	2.857	0.019

According to Thermogravimetric analysis (curves not shown) ESH⁺/Cl⁻ presented a sudden weight loss at 140°C and a total moderate weight loss between 140°C and 800°C. Therefore, the material is thermally stable up to 140°C.

Conclusions

The material of quaternary ammonium salt (ESH⁺/Cl⁻) has been successfully made using rice hull ash (RHA) as silica source. ESH⁺/Cl⁻ had been successfully synthesized based on the data from FTIR, Kjeldahl analysis and potentiometric titration. From thermogravimetric analysis, it was discovered that ESH⁺/Cl⁻ was thermally stable.

Acknowledgements

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