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Original Article

Silica gel derived from palm oil mill fly ash

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Abstract

Agro-wastes, especially ash containing silica, are promising sources of silica for synthetic amorphous silica production. In this research, palm oil mill fly ash (POMFA) was used as a raw material for silica gel production. The response surface method-central composite design was applied to study and to optimize the temperature and stirring speed for silica extraction from POMFA using sodium hydroxide solvent. Filtrates were analyzed using inductively plasma optical emission spectroscopy to measure their silica content. At the optimum condition, 60.42±0.83% of the silica can be extracted from the POMFA. The extract silica was acidified using 10% (v/v) H_2SO_4 to form silica gel. The chemical composition, the phases, and the micrograph of silica gel product are similar to the commercial silica gel.

Keywords: extraction, optimization, palm oil mill fly ash, response surface methods, silica gel

1. Introduction

Synthetic amorphous silica (SAS), including silica gel, precipitated silica and silica fume, is a highly versatile product because of its inherent properties and the flexible manufacturing process. A wide variety of grades with a range of physical properties of SAS can be produced. The characteristics of SAS which allow for its wide use in chemical industries are good flow ability, unique surface structure, and high adsorptive characteristic. For example, precipitated silica is used as filler in rubber and plastic industry to improve tensile strength, hardness, tear strength, and abrasion resistance. The precipitated silica can also be used as a rheological agent and a thickener for controlling the flow properties of products, such as paint and coating, tooth paste, cosmetic creams, and lotion. The silica fume is used in electronic industry for chemical mechanical wafer polishing (Sampat, 2008). The silica gel can be applied as an absorbent, a desiccant, a catalyst, and a catalyst support (Prasad & Pandey, 2012). The high temperature fusion for melting the silica sand and soda ash and the high temperature digestion for dissolving the sodium silicate in the commercial process of making the SAS use a huge amount of energy, which is expensive. In addition, the SAS produced contains metal impurities, which are found in the earth in the amount ranging from 400 to 10,000 ppm (Stephens *et al.*, 2002). In order to search for a cost-effective and eco-friendly process, many studies have been conducted regarding this issue. The agro-waste, especially ash containing silica as the source for silica production, is promising. One of the advantages is that the process involves fewer steps since each plant species has a constant chemical composition, and the final product contains only a narrow range of metal oxide impurities (Zemnukhova *et al.*, 2006). However, until now the studies focused on rice hull ash (Lima *et al.*, 2011; Rozainee *et al.*, 2008; Sousa *et al.*, 2009), sugar cane bagasse ash (Affandi *et al.*, 2009), and corn cob ash (Shim *et al.*, 2015) as the silica sources.

One of the agro-wastes containing silica, which is abundant in Indonesia, Malaysia, and Thailand, is palm oil ash. In 2014 the estimated palm oil production in those

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countries was 54,800 million tons or almost 89% of the world palm oil production (Indexmundi, 2016). From mass balance, the production of 1 ton of crude palm oil generates 1.41 tons of empty fruit bunch, 0.93 ton of fibers and 0.34 ton of shells as solid waste (Kramanandita *et al.,* 2014). About 85% of fibers and 15% of shells and empty fruit bunches are used as fuel for steam production in boilers. It is estimated that 5% of the fuel will be unburned and generate ash. This amounts to more than 2,880 million tons of ash that is sent to landfills rather than put into productive usage (Tay & Show, 1995). There are many studies on the utilization the palm oil ash as a replacement of cementing material (Bamaga *et al.*, 2013; Sata *et al.,* 2004; Tay & Show, 1995); however, only few studies have used the palm oil ash as a raw material for silica production. The leaching process using citric acid has been studied to produce silica from palm oil ash. This method can produce silica which has a purity of 92% (Faizul *et al.*, 2013). Nonetheless, the leaching process cannot separate the amorphous silica and the crystalline silica which are found in the ash. In a previous work, the extraction process of silica from palm oil mill fly ash (POMFA) was optimized (Utama *et al.,* 2013). The variables optimized included time, ratio of POMFA mass to NaOH volume and NaOH concentration. This research found that the optimum conditions were extraction time of 50 min, sodium hydroxide concentration of 1.40 N, and mass of ash to volume of solvent ratio of 0.23. In this work, the variables optimized are temperature and the stirring speed. The sol-gel process was applied to obtain silica gel from extract silica. The physical and chemical properties of silica gel product were characterized using the Fourier transform infrared (FTIR), the X-ray fluorescence (XRF), X-ray diffractometer (XRD), the scanning electron microscope (SEM), and scanning electron microscope-energy-dispersive X-ray (SEM-EDS). The results were then compared with silica gel from the reference.

2. Materials and Methods

2.1 Materials

The POMFA was obtained from Sawee Industrial Palm Oil Ltd., Chumporn, Thailand. The POMFA was dried at 110 °C before being used as the raw material. The chemical composition of the sample POMFA obtained using the XRF and C, H, N and O analyzer (CE instrument Flash EA 1112 series, Thermo Quest) are shown in Table 1.

From the analysis, silica and carbon are the main components in the POMFA. The laboratory grade sodium hydroxide with minimum NaOH content of 97% (dry basis) was obtained from Boss Official Limited Partnership, Hat Yai, Thailand. The commercial grade sulfuric acid (with minimum total acidity of 98% H_2SO_4 by mass) was obtained from K.K. Superstore, Hat Yai, Thailand.

2.2 Methods

The extraction process was done in 2,500 cm^3 temperature controlled glass extractor equipped with a 5 cm propeller stirrer driven by a variable speed motor for mixing. The process variables studied and optimized were temperature (75-100 °C) and stirring speed (500-1,200 RPM) in order to achieved highest silica conversion. The other process variables such as NaOH concentration, mass of POMFA to NaOH volume ratio and extraction time were used the optimized variables obtained from the previous research and set as fixed variables. The fixed variables used were NaOH concentration 1.4 N, mass of POMFA to NaOH volume ratio 468.2 g/2,000 cm^3 and extraction time 50 minutes (Utama *et al.,* 2013). The optimization of the extraction process was done using response surface method-central composite design (RSM-CCD). The full factorial design with 4 cube points, 4 axial points and 5 cubic center points was used. The proposed model for the response (η) was:

$$\eta = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^{\,2} + \beta_{22} x_2^{\,2} + \beta_{12} x_1 x_2 + \varepsilon \qquad (1)$$

where η is the predicted response for silica conversion (%); β_0 is the constant term; β_1; β_2 are the linear effects; β_{11} and β_{22} are the quadratic effects; β_{12} is the interaction effect and ε is the random error. The independent variable x_1 was the temperature (°C) and x_2 was the stirring speed (RPM). Regression analysis, ANOVA and the optimization were done using the Minitab 16.1.1 software. ANOVA was used to test the compatibility of the model with the experimental data by showing the lack of fit (LoF) which can be used to investigate the adequacy of the model. The optimum condition was verified by conducting experiments at that condition. Responses were monitored and results were compared with model predictions (Ramos de la Pena *et al.,* 2012).

The ICP-OES (Perkin Elmer Optima 4300 DV) was used to analyze the silica content in the extract. The sol gel process was applied to obtain silica gel. The extract silica from the optimum conditions was acidified using 10% (v/v) H_2SO_4 to form silica gel. The gel was aged for 18 hrs then crushed, washed and dried. The dried silica was then crushed and washed with water, while maintaining the pH at 7 by adding H_2SO_4 (Kalapathy *et al.,* 2000). According Iler (1979), the silica gel is made up of interconnected pore with a silicon dioxide core where water is entrapped and a surface consisting of silanol group. The characterization below was done to

Table 1. Chemical composition of POMFA

No	Element	Weight (%)	Oxide[b]	Weight (%)
1	H[a]	0.24	H[a]	0.24
2	C[a]	30.44	C[a]	30.44
3	N[a]	0.05	N[a]	0.05
4	O	30.78		
5	Al	0.63	Al_2O_3	1.19
6	Si	18.21	SiO_2	39.02
7	P	2.25	P_2O_5	5.16
8	S	0.36	SO_3	0.90
9	K	5.14	K_2O	6.20
10	Ca	6.71	CaO	9.39
11	Mn	0.15	MnO	0.20
12	Mg	2.47	MgO	4.11
13	Fe	1.20	Fe_2O_3	1.71
14	Rb	0.03	Rb_2O	0.03
15	Cu	0.05	CuO	0.06
16	Sr	0.36	SrO	0.04
17	Cl	0.40	Cl	0.40

[a] analyzed using C,H,N,O analyzer (CE instrument Flash EA 1112 series, Thermo Quest); [b] calculated.

confirm that the product is silica gel. The SEM EDS (JEOL JSM-5800LV) was used to identify the chemical composition of dried silica gel obtained to ensure that the silicon was more abundant compared to other mineral in product. The characterization using The XRF (PW 2400 Philips) was used to confirm the SEM EDS result. The characterization using XRD (X-ray Diffractometer, Philips X'Pert MPD, Philips) was done to confirm that the silicon dioxide in the product in the amorphous form. The FTIR (Vertex 70, Bruker) is used to identify the chemical bonds in the product to ensure the existing of the silanol group. The morphology of the dried silica gel was characterized using the SEM (FEI Quanta 400). The properties of POMFA were studied using the SEM (FEI Quanta 400) and the XRD (X'Pert MPD, Philips).

3. Results and Discussion

From Table 1, it can be calculated that the silica content of the POMFA free of volatile matter is 57.04%. This result is in accordance with the finding that palm oil fuel ash contains approximately 59.62% silica content (Awal & Shehu, 2013) and slightly higher than the reported silica content in sugar cane baggase ash of 50.36% (Affandi *et al.*, 2009). Even though the silica contents of the POMFA and sugar baggase ash are lower than silica content of rice husk ash which contains 95.03% (Sousa *et al.*, 2009), both POMFA and sugar cane baggase ash are industrial waste that are available in situ in huge amount compared to rice husk ash.

The morphology of POMFA is shown in Figure 1 at magnifications of 500 and 5000 times. In the palm oil plant, the fiber and 15 % of shell produced was burned as a fuel in a boiler to produce steam. The fixed bed with overfeed combustion system is used. The primary combustion air used to burn fixed carbon and cooled the grate is passed below the grate in the combustion chamber and the secondary combustion air used to burn the volatile matter produced in primary burning process is passed above the grate. According to Kutchko and Kim (2006), the mineral in fly ash will appear to be fused or partially melted because of heat, while the amorphous particles will tend to form spherical particles. At high temperature combustion, the minerals will become liquid and react with oxygen forming oxides. During the cooling process, the minerals may become crystals or spherical amorphous particles. In the production of palm oil, the waste materials from the shells, fibers and empty fruit bunch are used as fuel for the boiler. The ash that is produced during this burning process is collected upstream of the boiler and is called POMFA.

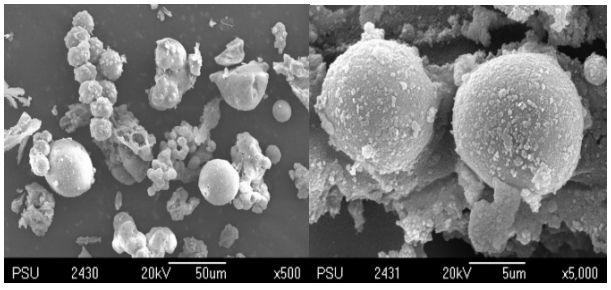

Figure 1. SEM image of POMFA at different magnifications.

As shown in Figure 1, irregular shaped particles and spherical particles can be seen in the POMFA, indicating the presence of silica crystals and amorphous silica. To investigate whether the silica is in the form of crystalline or amorphous, the POMFA was characterized using the XRD, as shown in Figure 2. The peak list of the XRD pattern was compared with the standard quartz pattern from the Scientific Equipment Center (SEC), Prince of Songkla University. The pattern is in accordance. Therefore, it can be concluded that part of silica in the POMFA is in the form of quartz.

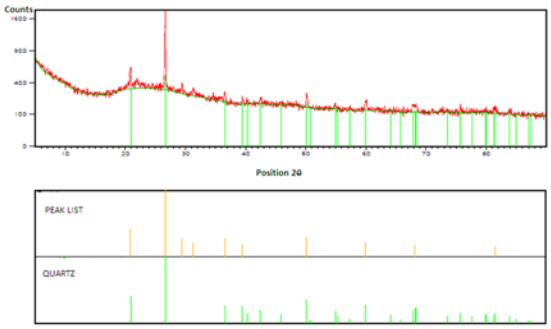

Figure 2. XRD patterns of POMFA and comparison of the peak list of the XRD pattern to the peak list of standard of quartz.

Experimental results using the RSM experimental design are given in Table 2. The data indicate that the highest conversion of silica occurred at 105 °C and 850 RPM. It can be seen that the higher the temperature used, the higher the conversion of the silica extracted. At low stirring speed range (375 to 850 RPM), the higher the stirring speed used, the higher the conversion of the silica extract; however, at the high stirring speed range (850–1,375 RPM), the increase in the conversion of the extracted silica was small as the stirring speed increases.

Table 2 Extraction variables and experimental data.

No.[a]	Variables			SiO_2 Concentration in Extract (mg/L)	Conversion (%) (η)
	Pt Type	Temperature (°C) x_1	Stirring Speed (RPM) x_2		
1	-1	105.0	850	43502.5	60.64
2	0	87.5	850	30871.4	43.03
3	1	100.0	1200	39628.6	55.24
4	0	87.5	850	28686.6	39.99
5	0	87.5	850	30314.3	42.26
6	-1	87.5	1345	31414.3	43.79
7	-1	70.0	850	14528.6	20.25
8	-1	87.5	355	23558.6	32.84
9	1	75.0	1200	22100.0	30.81
10	1	75.0	500	16700.0	23.28
11	0	87.5	850	31171.4	43.45
12	1	100.0	500	36600.0	51.02
13	0	87.5	850	30714.3	42.82

[a] Treatments are run in random order

The response surface model proposed was fitted to the experimental data using RSM procedure. The coefficients of the proposed model, the significance of variables and adequacy of the model are shown in Table 3.

Table 3. Significance of regression coefficient for silica conversion (%) and ANOVA.

Source	Variable Constant	Regression Coefficient	p values
Regression			0.000[a]
Linear			0.000[a]
	β_0	-123.418	0.000[a]
x_1	β_1	2.09997	0.000[a]
x_2	β_2	0.05154	0.000[a]
Square			0.025[a]
x_1^2	β_{11}	-0.00483	0.191
x_2^2	β_{22}	-1.48E-05	0.010[a]
Interaction			0.268
$x_1.x_2$	β_{12}	-1.89E-04	0.268
Residual error			
Lack of Fit			0.466

R-Sq = 99.18% R-Sq(pred) = 96.74% R-Sq(adj) = 98.60%; [a] significance at $\alpha = 0.05$.

From Table 3, it can be seen that the regression model for silica conversion is significant (p-value = 0.000 < 0.05). At least, one of the terms in the regression equation makes a significant impact on the silica conversion. All the p values for the linear effect are less than 0.05. So, all variables give a significant linear effect. The p value of the square effect for the silica conversion is 0.025 (p<0.05), so there is a significant quadratic effect. Only the square of the stirring speed gives a significant effect. The p-value of the interaction term is higher than 0.05. So, there is no significant effect to the mean response.

The lack of fit test gives a p value of 0.466 (p >0.05), meaning that there is no evidence that the model does not adequately explain the variation in the responses. The statistical analysis above indicates that the proposed model is adequate. The empirical regression equation relationship between silica conversion (%) and the research variable is given by the following equation:

$$\eta = -123.418 + 2.09997x_1 + 0.05154x_2 - 0.00483x_1^2 - 1.48E-05x_2^2 - 1.89E-04x_1x_2 \quad (2)$$

with satisfactory value of R^2 (0.9918).

The optimization process was done using Minitab 16.1.1. The goal is to maximize the conversion of silica. The optimum operating conditions obtained were the temperature of 105 ^oC and the stirring speed of 1065 RPM. The predictive result for this optimum operating condition for silica conversion was 60.79%. When the stirring speed was over 1,065 RPM, the effect of the increasing stirring speed to extraction process was small. Normally, for diffusion-controlled extraction, increasing the stirring speed will linearly increase the extraction rate. For stirring speed above 1,065 RPM, the chemical reaction or intra-particle diffusion may control the extraction process. In the chemical reaction or intra-particle diffusion controlled extraction process the rate of extraction will not change when the stirring speed reached the certain value (Zhou *et al.*, 2014).

The optimization process was validated by performing additional laboratory experiments under the optimum operating conditions. The conversion of silica extraction from the experimentation was 60.42±0.83% which is in agreement with the predictive value. The low extraction efficiency may be caused by the presence of silica in the form of quartz in the POMFA (Sousa *et al.*, 2009).

The FTIR was used to characterize major chemical group in the silica gel obtained. The FTIR spectra are depicted in Figure 3. The characteristic and peaks position of the FTIR spectra of the silica gel in Figure 3 aligns well with the FTIR spectra of commercial silica which was reported by Thuadaij and Nuntiya (2008). According to Kamath and Proctor (1999) the broad peak between 2,800 and 3,750 cm^{-1} is due to the silanol OH group and the adsorbed water bond to silica surface by hydrogen bond and the peak shown at 1,639 cm^{-1} is due to H-OH bond. The strong peak at 1,107 assigned to asymmetric Si-O-Si stretching vibrations whiles the peak at

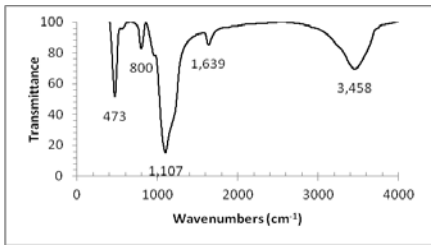

Figure 3. FTIR spectra of the silica gel product.

800 cm^{-1} assigned to symmetric Si-O-Si stretching vibrations and the peaks at 473 $cm^{-1}$is due to O-Si-O bending vibrations (Music *et al.*, 2011).

The XRD pattern of silica gel product is shown in Figure 4. There is no sharp peak in XRD pattern indicating no crystalline mineral in the product. The characteristic of amorphous material is given by the broad hump at the diffraction angle 2Θ between 15 and 35 degree. The XRD pattern of the silica gel obtained is in accordance with the XRD pattern of commercial silica gel Trysil 300 (Kamath & Proctor, 1998).

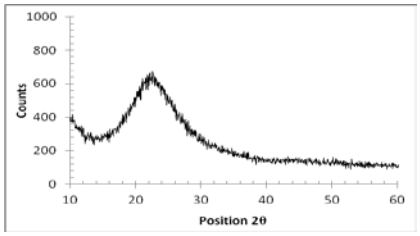

Figure 4. XRD pattern of silica gel product.

The SEM images of the silica gel product with magnifications of 500 and 5000 times are shown in Figure 5.

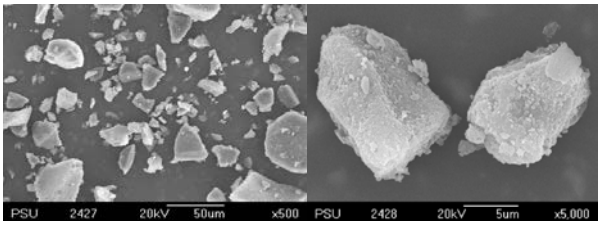

Figure 5. SEM image of silica gel obtained at different magnifications

From the above images, it can be estimated that the range of size of the aggregate in the silica gel obtained is <50 µm, which is almost the same as the range of size of the aggregate of Trysil 300, that is of <25 µm. Furthermore, the shape of the aggregate is similar to one another (Kamath & Proctor, 1998).

The SEM-EDS analysis of the silica gel product is depicted in Figure 6. The SEM-EDS spectrometry shows the element contents of the silica gel product. The silica gel product contains impurities of C, Na, K, Al, and S. The elements of C, K and Al are present in the POMFA and a small amount of those elements has been carried over in the silica gel product. The Na and S elements are from the sodium hydroxide and sulfuric acid, which were used in the extraction and sol-gel precipitation process and cannot be washed completely in the washing process. The Na element in the silica gel obtained is 1.3% (wt) which is lower than Na element in the silica powder made from sodium silicate using pressurized carbonation process reported by Chai et al. (2009) which is 1.72% (wt). The composition of silica gel which was analyzed using XRF is shown in Table 4.

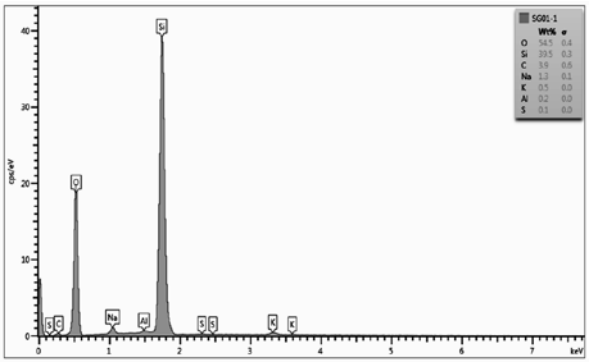

Figure 6. SEM-EDS spectrometry of the silica gel product.

Table 4. Chemical composition of the silica gel product.

No	Element	Weight %	Oxide[a]	Weight %	Standard (Roemp, 2001)
1	Si	42.99	SiO_2	95.33	≥ 95
2	Na	0.90	Na_2O	2.50	0.2-2.4
3	S	0.10	SO_3	0.48	0.2-3.0
4	Al	0.10	Al_2O_3	0.39	-
5	K	0.52	K_2O	1.29	-
6	O	52.01			
7	LOI	3.38	LOI	3.38	2-15

The semi quantitative XRF analysis confirms the presence of the impurities above. Both of the SEM-EDS and the XRF analysis are shown that the silicon and oxygen element more abundant compare to other elements. This is confirming that the presence of silica is predominant in the product obtained. The chemical composition of silica gel derived from palm oil mill fly ash is in the range of the standard of commercial silica gel.

4. Conclusions

The proposed model adequately explains the variations of the responses and can be used for optimizing the operating conditions of silica extraction from POMFA. The optimum operating conditions were at the temperature of 105 ^oC and the stirring speed of 1,065 RPM. The resulting 60.42±0.83% of silica from the POMFA can be extracted under the optimum condition. The chemical composition and physical characteristic of silica gel product, which were analyzed with FTIR, XRD, SEM, SEM-EDS and XRF, are similar to the commercial silica gel from the references.

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