Effect of Synthesis Conditions on % Yield of Silica Gel from Microwave Process

Nuchanaporn Pijarn*, Hasan Seng, Suparada Khanaruksombat, Pongtep Phonprasert, and Phatarapon Vorapracha

***Abstract*—** The objective of this work is to investigate the condition of silica gel synthesis from rice husk ash for obtaining the high yield of silica gel. The silica gel was synthesized from rice husk ash via microwave method in various conditions. Microwave power and irradiation time in microwave were varied for synthesis of silica gel. Microwave power was studied of 100, 200 and 300 watts, and irradiation time was studied of 30 min, 60 and 90 min as well as nine conditions. The yield of silica gel from each condition of SG-100-30 SG-100-60, SG-100-90, SG-200-30, SG-200-60, SG-200-90, SG-300-30, SG-300-60, and SG-300-90 was 80.76, 82.47, 100.00, 86.68, 83.47, 100.00, 100.00, 98.89 and 82.86%, respectively. It was found that when syntheses with higher microwave power the % yield had increased for all irradiation time except 90 min. Therefore, it was illustrated that when syntheses with longer irradiation time the %yield had increased for all irradiation time conditions except in the condition of 300 watts. Consequently, the appropriate condition for synthesis of silica gel is the condition 300 watts for 30. In addition, the silica gel from this condition had more transparent than the other conditions.

***Keywords*—** Silica gel, Sol-gel method, Microwave, Rice husk, % Yield

I. Introduction

Synthesis of silica gel (SG) from rice husk (RH) as the implementation of agricultural residues were used to benefit a certain way. Since Thailand is the most widely grown rice. The rice husk is separated from incontinence are numerous. The silica gel was synthesized from rice husk ash had been successfully in many years ago [1]. After that, the synthesis of silica gel was interested by many researchers may be caused by many applications [2]-[4]. In our previous work, reported on nanosized-silica gels formed under controlled conditions of synthesis of silica gel by refluxing [5]. The sol-gel processing of silica gel was synthesized by many methods such as conventional hydrothermal method [6]-[7], thermal hydrolysis method [8]-[10], and microwave-assisted method [11]. Although the synthesis of silica gels via microwave method had been successfully for few years, synthesis of silica gel from rice husk ash using microwave techniques has no reported. Therefore, in our previous research, the synthesis of silica gel from rice husk ash using microwave techniques had been successfully synthesized by microwave 300 watts for an hour reported in 2014 [12]. However, a condition of silica gel synthesis to produce in high yield has not been studied and reported.

Consequently, in this work, synthesis conditions for synthesis silica gel from rice husk ash were varied to investigate the effect on % yield of silica gel. The condition for synthesis of silica gel from rice husk ash in term of the % yields from various microwave condition syntheses were compared and reported. Moreover, the color and transparent of silica gel was described.

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II. Materials and Method

A. Materials

Analytical grade of sodium hydroxide (NaOH) was purchased from Ajax Finchem Pty. Ltd. Sulfuric acid (H_2SO_4) was purchased from Merck KGaA, 64271, Darmstadt, Germany. All standard and sample solutions were dissolved with reverse osmosis (RO) water from the Chemistry Laboratory Center, Faculty of Science and Technology, Bangkokthonburi University, Thailand. Rice husk was obtained from farmers in Thung Sriudom distinct, Ubon Ratchathani province, Thailand.

B. Method for Synthesis of Silica Gel

The rice husk was calcined (RHA) and added 1.0 mol/dm^3 of NaOH solution follows our previous work [12]. The suspension was subjected into microwave cavity at 100, 200 and 300 watts for 30, 60 and 90 min. The boiling suspension was filtered and washed with boiling water. The filtrate was cooled in room temperature before titrating with 1.0 mol/dm^3 H_2SO_4 until pH 7. The solution was aged at room temperature for overnight to form gel. The silica gel was washed with RO water for several times to remove sodium sulfate (Na_2SO_4) [5]. The gel was separated from solution using centrifugation

technique and dried for 1 hour in oven at 100 °C. The drying silica powder was weighed to calculate % yield. The synthesis steps are shown in Fig. 1.

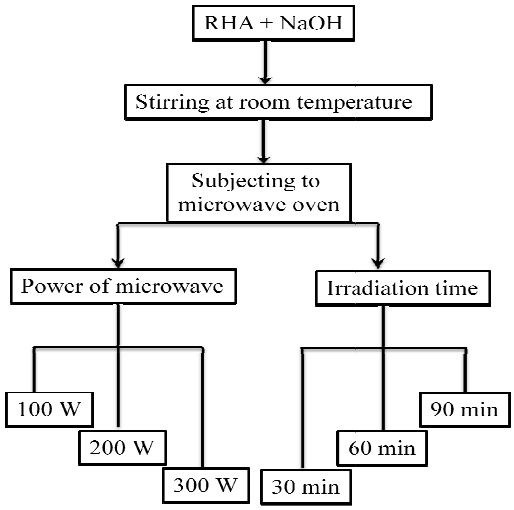

Fig. 1 synthesis steps of silica gel

The synthesis condition details are shown in TABLE I. The silica gel from the condition of 100 watts for 30, 60 and 90 min had code as SG-100-30, SG-100-60 and SG-100-90, respectively. The silica gel from the condition of 200 watts for 30, 60 and 90 min had code as SG-200-30, SG-200-60 and SG-200-390, respectively. And SG-300-30, SG-300-60 and SG-300-90 were used code of the silica gel from the condition of 300 watts for 30, 60 and 90 min, respectively.

TABLE I
THE CONDITION OF SYNTHESIS

Sample code	Microwave power (watts)	Irradiation time (min)
SG-100-30	100	30
SG-100-60	100	60
SG-100-90	100	90
SG-200-30	200	30
SG-200-60	200	60
SG-200-90	200	90
SG-300-30	300	30
SG-300-60	300	60
SG-100-90	300	90

III. ANALYSIS

A. Calculation of % yield

The % yield of silica gel from various synthesis conditions were calculated follow as in (1)

$$\%Yield = (\frac{SG}{RHA}) \times 100 \quad (1)$$

Where;

SG is weight of silica gel obtained from each condition

RHA is weight of rice husk ash used synthesis

The % yield value was obtained from three times for synthesis.

B. External morphology observation

External morphologies of silica gel from various conditions were compared. The color and transparent were observed by eyes observation.

IV. RESULTS AND DISCUSSION

A. Calculation of % yield

The summary of weights and % yield of silica gel from various conditions were presented in TABLE II. The silica gel obtained from the condition of 100 watts for 30, 60 and 90 min had weight and % yield of 8.08 and 80.76%, 8.25 and 82.47%, and 10.00 and 100.00%, respectively. The silica gel obtained from the condition of 200 watts for 30, 60 and 90 min had weight and % yield of 8.67 and 86.68%, 8.235 and 83.47%, and 10.00 and 100.00%, respectively. The silica gel obtained from the condition of 300 watts for 30, 60 and 90 min had weight and % yield of 10.00 and 100.00%, 10.00 and 100.00%, and 9.89 and 98.89%, respectively. These results were supported that the % yield increased with increasing of high temperature [13]. However, the % yield of silica gel increased with the increasing of irradiation time also that might be the main cause of the long time for extractions from RHA.

TABLE II
WEIGHT AND YIELD OF SILICA GEL FROM VARIOUS CONDITIONS

Sample code	RHA (g)	Silica gel (g)	Yield (%)
SG-100-30	10.00	8.08	80.76
SG-100-60	10.00	8.25	82.47
SG-100-90	10.00	10.00	100.00
SG-200-30	10.00	8.67	86.68
SG-200-60	10.00	8.35	83.47
SG-200-90	10.00	10.00	100.00
SG-300-30	10.00	10.00	100.00
SG-300-60	10.00	9.89	98.89
SG-100-90	10.00	8.29	82.86

Fig. 2 depicts the % yield of silica gel prepared from the synthesis condition of 100 watts in various irradiation times. This figure indicates that the % yield of silica gel increased with the increasing of irradiation time that might be the main cause of the long time for extractions from RHA.

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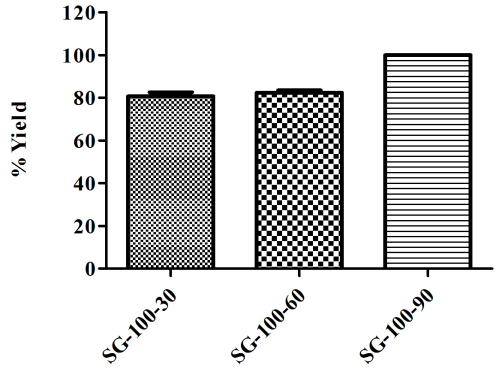

Fig. 2 The % yield of silica gel obtained from synthesis condition of 100 watts in various synthesis times

The % yield of silica gel prepared from the synthesis condition of 200 watts in various irradiation times, 30, 60 and 90 min as shown in Fig. 3. This figure specified that the % yield of silica gel increased with the increasing of irradiation time might be the main cause of the low power for extractions.

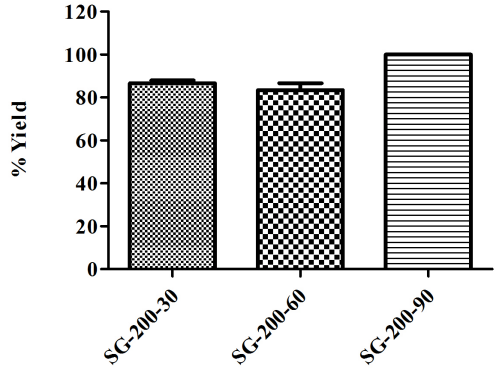

Fig. 3 The % yield of silica gel obtained from synthesis condition of 200 watts in various synthesis times

On the other hand, Fig. 4 shows the % yield of silica gel obtained from the synthesis condition of 300 watts in various irradiation times. The result indicates that the % yield of silica gel decreased with the rise of irradiation. This may be due to the use of power is too high and it takes too long irradiation time. Moreover, the condition of microwave power 300 watts for 90 min made the suspension of silica gel was vigorously boiled, burned, and dried. Therefore, this condition is not suiTABLE condition for synthesis of silica gel from RHA with microwave technique.

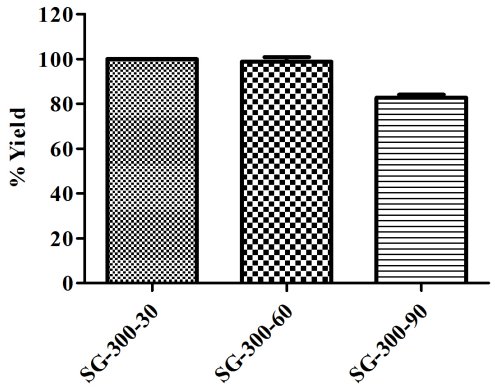

Fig. 4 The % yield of silica gel obtained from synthesis condition of 300 watts in various synthesis times

These results showed that with microwave power and microwave irradiation time rising hydrolyzate and extracted as well as yields of product were slightly increased [14]. However, when compared the silica gel from the condition of 100, 200 and 300 watts for 90 min that is the longest time for synthesis in this study as shown in Fig. 5. The result shows that the % yield of silica gel decreased with the increasing of power of microwave. This outcome implied that although yields of silica gel increased with the increasing of microwave power and irradiation time, it has critical point of microwave power and irradiation time. Therefore, it is depend on each situation and condition of synthesis.

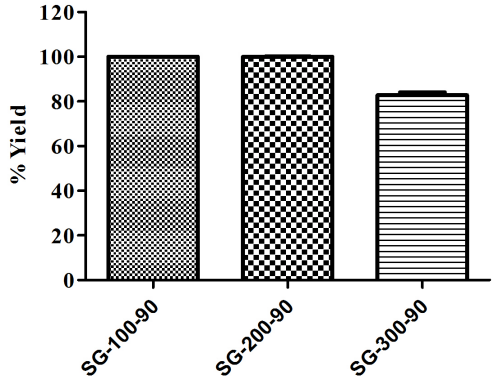

Fig. 5 The % yield of silica gel obtained from synthesis condition of 100, 200 and 300 watts for 90 min

B. External morphology observation

The external physical properties such as color and transparent were observed. Fig. 6 shows the color and transparent of silica gel from all conditions. When compared only the silica gel obtained from the synthesis condition of 100 watts, the silica gel obtained from synthesis time of 60 min had darker than the others. In the condition of 200 and 300 watts, the silica gel obtained from both microwave power for 90 min had darker than the others. These are show that too high power of microwave may burn of silica gel. The transparent of silica gel is related to the color. The most

transparent of silica gel from the synthesis condition of 100 watts is the silica gel obtained synthesis time for 90 min. The most transparent of silica gel from the synthesis condition of 200 watts is the silica gel obtained synthesis time for 60 min. And the most transparent of silica gel from the synthesis condition of 300 watts is the silica gel obtained the synthesis time for 30 min. These can be concluded in that if we used the low power, we should be used long time for synthesis. On the other hand, if we used the high power, we should be used short time for synthesis. However, if we used synthesis time less than 30 min, the silica gel was not occurred [12]. Moreover, in this work, it can be found that when using the microwave power 300 watts for more than 90 min obtained silica gel with brown color and opaque.

Fig. 6 External morphology of silica gel from all conditions

V. Conclusion

This work can be specified the percent yield of silica gel from rice husk ash with microwave technique. It can be concluded that percent yield of silica gel increased with the increasing of microwave power and irradiation time for synthesis. However it is not always possible to adhere to this statement because it may be depend on various the synthesis conditions. The results showed that the yield of silica gel from both conditions of 100 and 200 watts for 90 min had yield of 100%. Except for the yield of silica gel from conditions of 300 watts for 90 min, it had yield just of 82%. In addition, the silica gel from condition of 300 watts for 30 min had percent yield for 100%. Consequently, if want to save the energy, it can be followed the condition of 100 watts for 90 min, but if want to save time, it can be followed the condition of 300 watts for 30 min to prepare the silica gel that white color and transparent.

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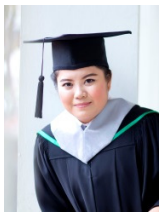

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