ภาคผนวก ก

เสนอผลงานวิจัยแบบบรรยายพร้อมบทความ

ครั้งที่ 1 การประชุมเสนอผลงานวิจัยระดับบัณฑิตศึกษาแห่งชาติ

(Grad-Research Conference)

ครั้งที่ 7 ณ มหาวิทยาลัยสงขลานครินทร์

4-5 เมษายน 2550

ครั้งที่ 2 การประชุมวิชาการนาโนเทคโนโลยีแห่งประเทศไทย ครั้งที่ 1 ณ โรงแรม

เซ็นทรัลดวงตะวัน จังหวัดเชียงใหม่

14-16 สิงหาคม 2550

Preparation of Nanosilica Powder from Rice Husk Ash.

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Abstract: Rice husk ash (RHA) is one of the most silica rich raw materials containing about 90-98% silica after complete combustion. Nanosilica has been added to polymer to increase strength, flexibility and aging resistance. Nanosilica which has amorphous structure can improve the strength of the concrete. Nanosilica was prepared by heat and sodium hydroxide treatments. Rice husk ash was burnt at 700 °C for 3 and 6 h, respectively. Consequently, silica content after heat treatment at 700 °C for 6 h was 98.14 %. Pure silica was prepared by alkaline extraction method provided higher SiO_2 content. However, Nanosilica powder which was extracted by refluxing with 6 N HCI and followed by dissolved in 2 N NaOH was observed by TEM. From transmission electron micrographs show nanosilica particles are in the agglomerate form which dimension of 50 nm. The particle size distribution is found to be uniform and agglomerated. The diffraction pattern of the particles showed a diffuse ring pattern which indicative of amorphous phase.

Introduction: The beneficiation of rice generates as by-product rice husk that corresponds to about 23 % of its initial weight. This husk can be used as a fertilizer in agriculture or as an additive for cement and concrete fabrication [1]. Rice husk ash (RHA) is one of the most silica rich raw materials containing about 90-98% silica after complete combustion. The selection of ash is important as the quality of ash determines the total amount as well as quality of silica recoverable. The initial step is extraction of silica from ash as sodium silicate using caustic soda.

The reaction is

SiO_2 + NaOH → Na_2SiO_3 + H_20

(ash) (sodium hydroxide) (sodium silicate) (water)

In the second step of the process, silica is precipitated from sodium silicate using sulphuric acid

The reaction is

Na_2SiO_3 + H_2SO_4 → SiO_2 + Na_2SO_4 + H_2O

(sodium silicate) (sulphuric acid) (silica) (sodium sulphate) (water)

In the present, nano-powder may be defined as the ability to work at the molecular level, atom by atom to create large structure with fundamentally new molecular organization. Since this technology is developed from the scale range of 10^{-9} m or10 A° such as nanosilica has been added to polymer to increase strength, flexibility and aging resistance. Nanosilica which has amorphous structure can improve the strength of the concrete. In this paper, preparation and characterization of as-received rice husk, pure silica from rice husk and nanosilica powder from pure silica are presented.

Experimental

Step 1. Preparation of as-received rice husk.

Rice husk ash from brick factory was washed by distilled water until pH 7 and burned at 700 °C for 3 and 6 h, respectively. Quantitative chemical analyses of RHA were accomplished by X-ray fluorescence.(Horiba Mesa-500w)

Step 2. Preparation of pure silica from rice husk ash.

10 grams of RHA samples were stirred in 80 ml of distilled sodium hydroxide solution at concentration 2.0, 2.5 and 3.0 N, respectively. RHA was boiled in cover 250 ml Erlenmeyer flask for 3 h. The solution was filtered and the residue was washed with 20 ml of boiling water. The filtrate was allowed to cool to room temperature and added H_2SO_4 5 N with constant stirring to pH 10 and then added NH_4OH until pH 8.5 left for 3.5 h. The filtrate was dried at 120 °C for 12 h and pure silica powder was investigated by fourier transform infrared (FTIR- model tenser 27).

Step 3. Preparation of nanosilica powder from pure silica.

Pure silica was extracted by refluxing with 6 N HCI for 4 h and then washed repeatedly using deionised water. Extracted silica was then dissolved in 2 N NaOH by continuous stirring for 10 h on a magnetic stirrer and then concentrated H_2SO_4 was added to adjust pH in the range of 7.5-8.5. The precipitated silica was washed repeatedly with warm deionised water. The washing process continues by deionised water repeatedly and dried at 50 °C for 48 h in the oven. Particle size and morphology of synthesized nanosilica powder were examined by TEM (Jeol, JEM2010).

Results and discussion

The rice husk ash (RHA) sample after burning out at 700 °C for 6 h presented the highest amount of silica content as shown in Table 1 compared to the other samples. The sample which was heated up to 700 °C for 6 h generated the percentage yield up to 92.76 % due to some of organic matter was burnt out as shown in Table 2. The optimum concentration of sodium hydroxide is 2.5 N.

Table 1. Chemical composition of RHA before after burning out at 700 °C for 3 and 6 h.

Components expressed as oxides	RHA as-received	RHA after burning out at 700 °C for 3h	RHA after burning out at 700 °C for 6h
SiO_2	96.51	97.86	98.14
Al_2O_3	0.15	-	-
Fe_2O_3	0.17	0.07	0.07
CaO	0.66	0.52	0.46
K_2O	1.23	1.01	1.05
ZrO	0.05	0.01	0.03
MgO	0.77	0.29	-
P_2O_3	0.21	-	-
Mn_2O_3	0.21	0.16	0.16
SO_3	0.04	0.07	0.07
LOI	-	0.01	0.02

Table 2. Percent yield of silica from RHA after burning at 700 °C for 3 and 6 h.

Temperature (°C)	Yield of silica (%)
700 °C 3 h.	61.52
700 °C 6 h.	92.76

Table 3. Effect of concentration of sodium hydroxide on the percent yield of pure silica.

Concentration of sodium hydroxide (Normal)	Yield of pure silica (%)
2.0	64.22
2.5	90.34
3.0	91.11

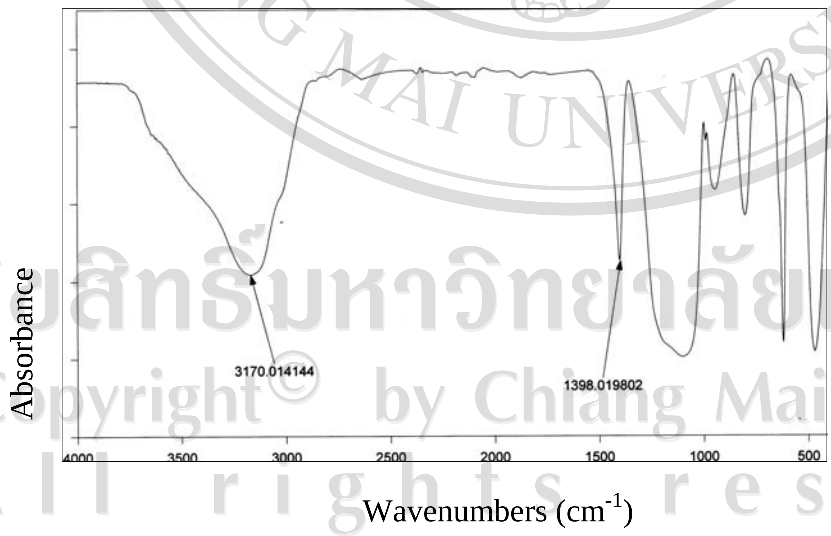

Fig. 1. Fourier transform infrared spectra of spectra of pure silica produced from RHA.

The major chemical groups present in silica are identified by the FTIR spectra shown in Fig. 1. The broad band between 2800 and 3750 cm^{-1} was due to silinol OH groups and adsorbed water. The predominant absorbance peak at 1320 cm^{-1} was due to siloxane bonds (Si-O-Si). The peaks between 1200 and 700 cm^{-1} are attributed to vibration modes of the gel net work. TEM micrographs of nanosilica after extracted from pure silica are shown in Fig. 2. Micrograph of the nanosilica showed agglomerate particles of dimension about 50 nm. The particle size distribution is found to be uniform and agglomerated species. The diffraction pattern of a main phase particle is shown in Fig. 3, shows a diffuse ring pattern, indicative of an amorphous phase.

Fig. 2.TEM micrograph of nanosilica powder

Fig. 3. Diffraction pattern of nanosilica powder

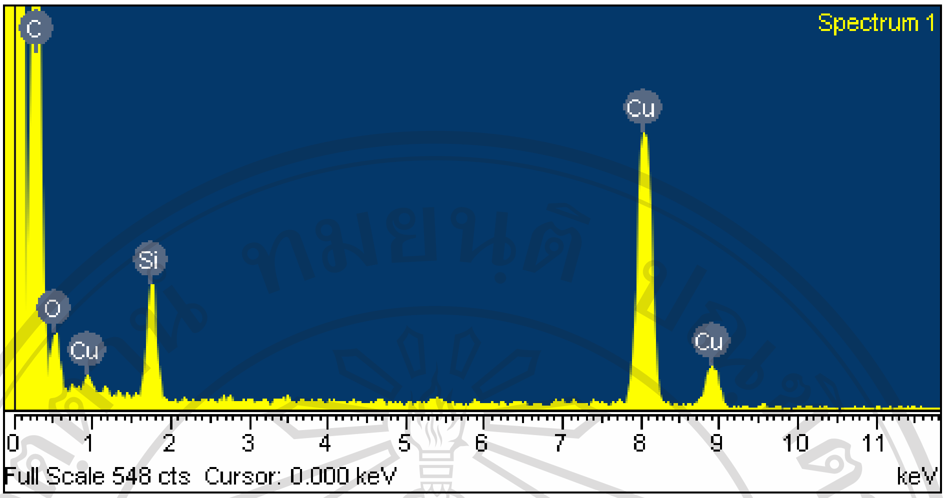

Fig. 4. EDS of nanosilica particles.

The chemical composition of nanosilica particles were determined by EDS. EDS profile of nanosilica particles contained predominantly the elements of Si, O, C and Cu. Both Si and O peaks corresponded to the silica. The dominant signals originated from copper and carbon due to TEM copper grid and carbon coating.

Conclusions

This study revealed that silica with 92.76 % silica content can be produced from RHA using a simple heat treatment. The 2.5 N sodium hydroxide treatment resulted in a higher SiO_2 content. Nanosilica particles are in the agglomerate form which dimension of 50 nm. The particle size distribution is found to be uniform and agglomerated species. The diffraction pattern of the particles showed a diffuse ring pattern which indicative of amorphous phase.

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(3) Kalapathy U., Proctor A., and Shultz J., (2000), “An Improved Method for Production of Silica from Rice Hull Ash”, *Bioresource Technology*., **85**, 285-289.

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Keyword : Rice husk ash, Precipitation, Nanosilica, TEM

Synthesis and Characterization of Nanosilica from Rice Husk Ash Prepared by Precipitation Method

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ABSTRACT

Nanosilica was prepared by precipitation method and characterized by various analytical techniques. From transmission electron micrographs show nanosilica particles are in the agglomerate form with dimension of 50 nm. The particle shape is found to be uniform and agglomerated. The diffraction pattern of the particles shows a diffuse ring pattern with indicative of amorphous phase. From x-ray diffractograms show that the obtained product is amorphous nanosilica and the specific surface area is 656 m^2g^{-1}. Subsequently, the infrared spectra data supports the presence of hydrogen bonded silanol group and siloxane groups in silica. In this study, nanosilica was introduced in cement paste. From the experimental results, it was found that the incorporation of the nanosilica in the cement paste increase the compressive strength compared with the portland cement.

Key words: nanosilica, precipitation, cement paste, compressive strength

INTRODUCTION

The beneficiation of rice generates as by-product rice husk that corresponds to about 23 % of its initial weight. It can be used as a fertilizer in agriculture or as an additive for cement and concrete fabrication [1]. Rice husk ash (RHA) is one of the most silica rich raw materials containing about 90-98% silica after a complete combustion. The selection of ash is important as the quality of ash determines the total amount as well as quality of silica recoverable. The initial step is extraction of silica from ash as sodium silicate using caustic soda.(Kalapathy et al., 2000)

The reaction is

SiO_2 +	$NaOH$	$\rightarrow$ Na_2SiO_3 +	H_2O	
(ash)	(sodium hydroxide)	(sodium silicate)	(water)	

In the second step of the process, silica is precipitated from sodium silicate using sulphuric acid

The reaction is

Na_2SiO_3 +	H_2SO_4	$\rightarrow$ SiO_2 +	Na_2SO_4 +	H_2O
(sodium silicate)	(sulphuric acid)	(silica)	(sodium sulphate)	(water)

In the present, nano-powder may be defined as the ability to work at the molecular level, atom by atom to create large structure with fundamentally new molecular organization. Since this technology is developed from the scale range of 10^{-9} m or10 A° such as nanosilica has been added to polymer to increase strength, flexibility and aging resistance. Nanosilica which has amorphous structure also can improve the strength of the concrete. (Jal et al., 2004) In this paper, preparation and characterization of pure silica from rice husk and nanosilica powder from pure silica were presented. The nanosilica powder which was introduced in cement paste was investigated.

MATERIAL AND METHODS

2.1 material

Rice husk ash which obtained from brick factory was washed by water and burned at 700 °C for 3 and 6 h. Quantitative chemical analyses of RHA were accomplished by X-ray fluorescence.(Horiba Mesa-500w)

2.2 Synthesis pure silica powder from rice husk ash.

Ten grams of RHA samples were stirred in 80 ml of 2.0, 2.5 and 3.0 N sodium hydroxide solutions, respectively. RHA was heated in a covered 250 ml Erlenmeyer flask for 3 h. The solution was filtered and the residue was washed with 20 ml boiling water. The filtrate was allowed to cool down to room temperature and then added 5 N H_2SO_4 with constant stirring until pH 2 and then added NH_4OH until pH 8.5 left for 3.5 h. The filtrate was dried at 120 °C for 12 h and pure silica powder was investigated by fourier transform infrared (FTIR- model tenser 27).

2.3 Preparation of nanosilica powder from pure silica powder.

Pure silica was extracted by refluxing with 6 N HCI for 4 h and then washed repeatedly using deionised water to make it acid free. It was then dissolved in 2 N NaOH by continuous stirring for 10 h on a magnetic stirrer and then concentrated H_2SO_4 was added to adjust pH in the range of 7.5-8.5. The precipitated silica was washed repeatedly with warm deionised water till the filtrate becomes completely alkali free. The washing process continues by deionised water repeatedly and dried at 50 °C for 48 h in the oven. Nanosilica powder was examined by TEM (Jeol, JEM2010), XRD (Megix-Pro Philips), specific surface area (Quanta Chrome Autosorp-1) and FTIR.(Model tenser 27)

2.4 Preparation of paste specimen.

Nanosilica was used to replace portland cement by direct weight 2, 4, 6 and 10 wt%. For all the paste 50×50×50 mm cubes were cast in mould, using a water to binder ratio of 0.5 wt %. The samples were demoulded and cured in water at 20 °C for specified period (7, 28 days). After curing, cement paste specimens were determined compressive strength. The fracture surface (interface) of cement paste was examined the microstructure by SEM (Jeol JSM-5910).

RESULTS AND DISCUSSION

The rice husk ash (RHA) sample after completely burned at 700 °C for 6 h presented the highest amount of silica content as shown in Table 1. The sample which was heated up to 700 °C for 6 h generated the percentage yield up to 92.76 % due to some of organic matter was burnt out as shown in Table 2. The optimum concentration of sodium hydroxide is 2.5 N.

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Concentration of sodium hydroxide (Normal)	Percentage yield of pure silica (%)
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2.5	90.34
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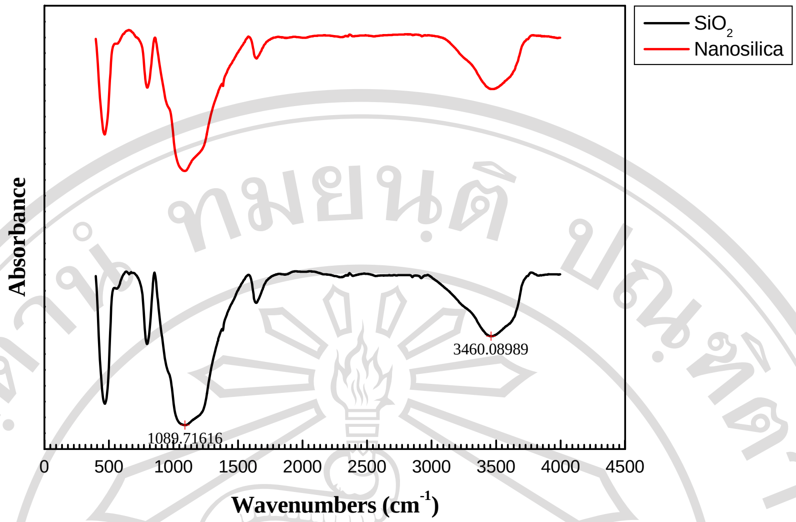


Fig. 1. Fourier transform infrared spectra of pure silica product.

The major chemical components in pure silica are shown in Fig. 1. The broad band between 2800 and 3750 cm^{-1} is due to silinol OH groups and adsorbed water. The predominant absorbance peak at 1320 cm^{-1} is due to siloxane bonds (Si-O-Si). The peaks between 1200 and 700 cm^{-1} are attributed to vibration modes of the gel net work.(Jal et al., 2004) TEM micrographs of nanosilica after extracted from pure silica are shown in Fig. 2. Micrograph of the nanosilica particles show agglomerate form of dimension about 50 nm and specific surface area is 656 m^2g^{-1}. The particle shape distribution is found to be uniform. The diffraction pattern of a main phase particle is shown in Fig. 3, shows a diffuse ring pattern, indicatives of an amorphous phase.

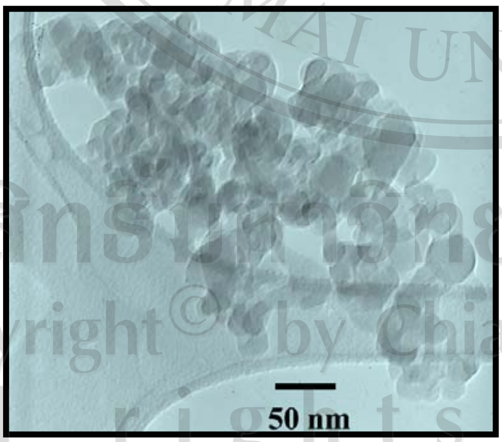
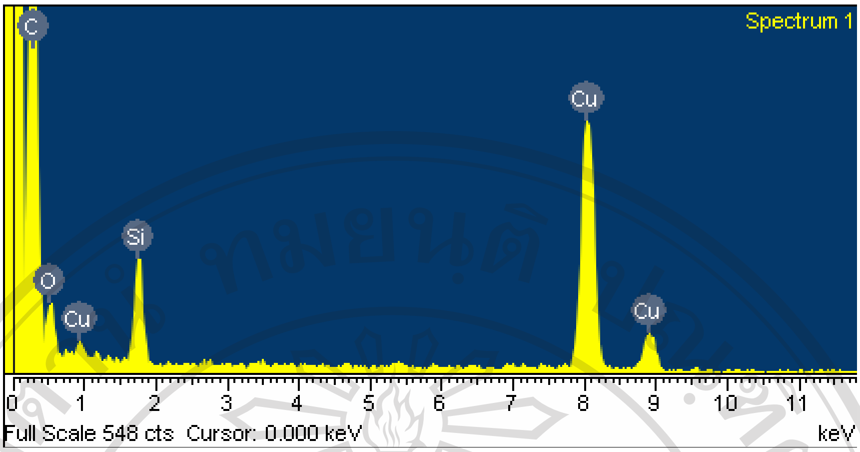


Fig 2. TEM micrograph of nanosilica powder.

Fig 3. Diffraction pattern of nanosilica powder.



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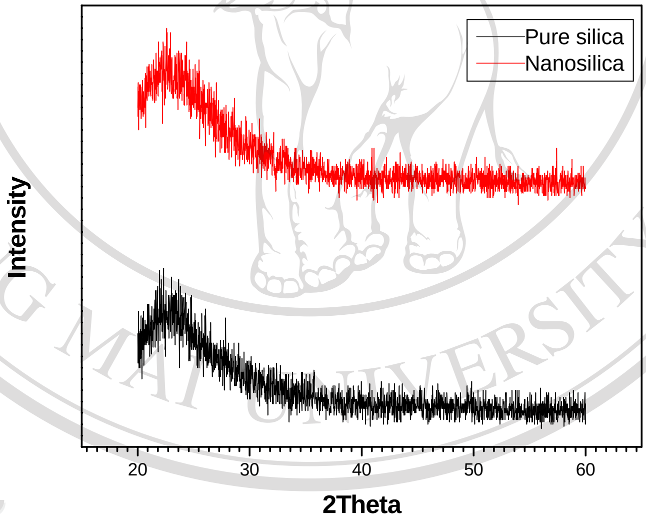


Fig 5. Diffractograms of pure silica and nanosilica.

Strong broad peaks of nanosilica and pure silica are centered range on 23 ° and 22 °(2θ), which are in keeping with the strong broad peak of a characteristic of amorphous SiO_2.(Byung et al., 2007) The results show that both nanosilica and pure silica are in an amorphous state.

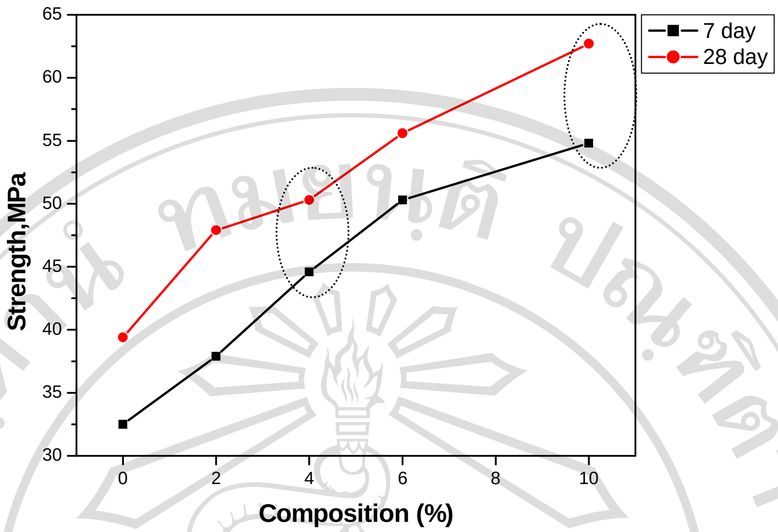
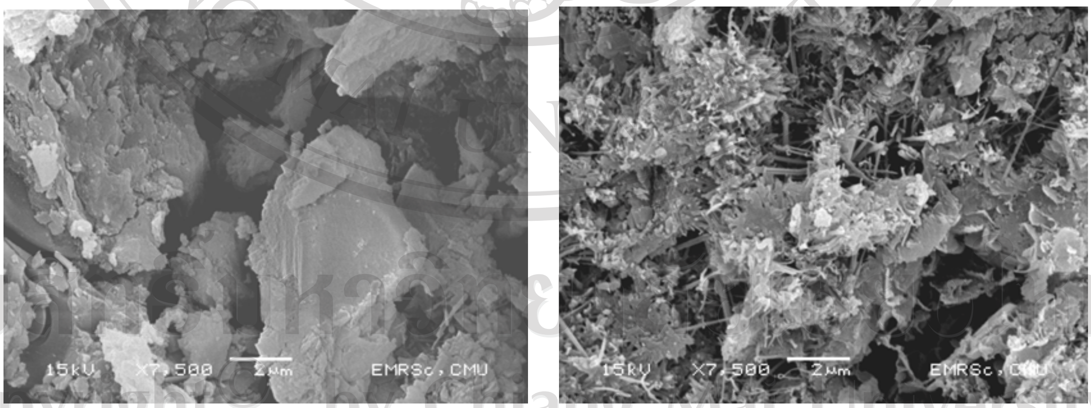


Fig 6. Compressive strength of nanosilica.

It can be seen that the compressive strength was developed in cement paste containing nanosilica particles in every case and higher than that of control cement paste. The difference in the strength development of the cement paste can be attributed to pozzolanic reaction. The strength of the cement paste was found to increase as the nanosilica content increased from 2 to 10 wt%.



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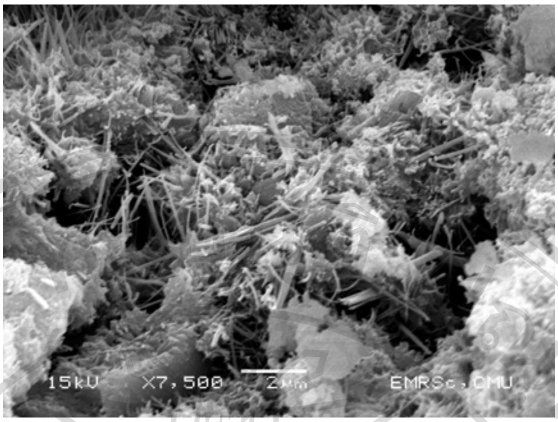

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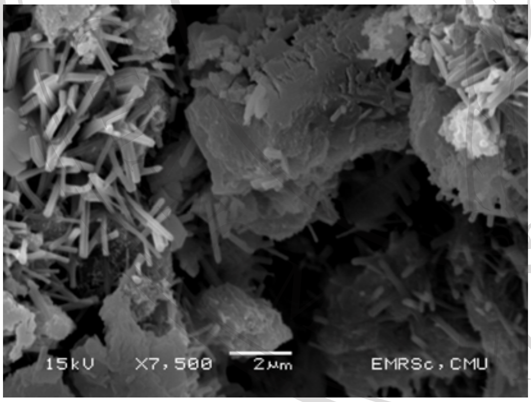
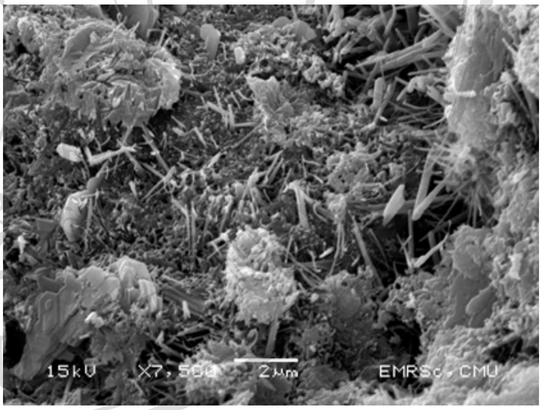
Fig 7. SEM micrograph of portland cement pastes at 7 days., (a.) SEM micrograph of portland cement paste,(b.) SEM micrograph of cement paste addition of NS 4 %wt, (c.) SEM micrograph of cement paste addition of NS 10 %wt.





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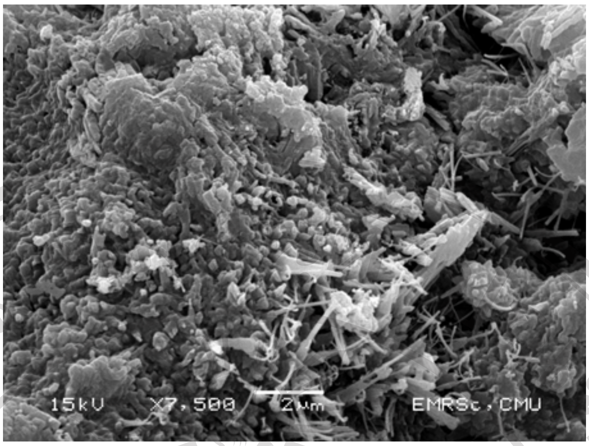

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Fig 8. SEM micrograph of portland cement paste at 28 days., (d.) SEM micrograph of portland cement paste,(e.) SEM micrograph of cement paste addition of NS 4 %wt (f.) SEM micrograph of cement paste addition of NS 10 %wt.

The mechanism predicted by the compressive strength test and SEM examinations were performed. Addition of nanosilica in cement paste led to differences in the microstructure of the hardened pastes. SEM micrographs of cement pastes which added nanosilica show C-S-H gel in isolation surrounded and connected with many needle-hydrates in the SEM micrograph of portland cement paste. On the other hand, dense and compact phases reveal that a formation of hydration products and reducing the number of $Ca(OH)_2$ crystals.(Hui et al., 2003)

CONCLUSIONS

Nanosilica particles which obtained from the rice husk ash are in the agglomerate from which dimension of 50 nm and specific surface area 656 m^2g^{-1}. The particle shape distribution is found to be uniform. The diffraction pattern of the particles show a diffuse pattern which indicative of amorphous phase and supported by XRD patterns. The infrared spectral data also supports the presence of hydrogen bonded silanol group and the siloxane groups in the silica. Based on the results of compressive strength test, it is expected that nano scale silica behaves not only as filler but also improve the strength of cement paste. Therefore, the addition of nanosilica particles in cement paste, introduce high performance to concrete.

ACKNOWLEDGEMENTS

The author is grateful to the Thailand Research Found for fundings this research and financially supported by Grad Research Chiang Mai University.

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ภาคผนวก ข

ผลการวิเคราะห์พื้นที่ผิวจำเพาะด้วย BET

สภาวะในการวิเคราะห์

สารตัวอย่าง ซิลิกา

ลักษณะผง ผงละเอียดสีขาว

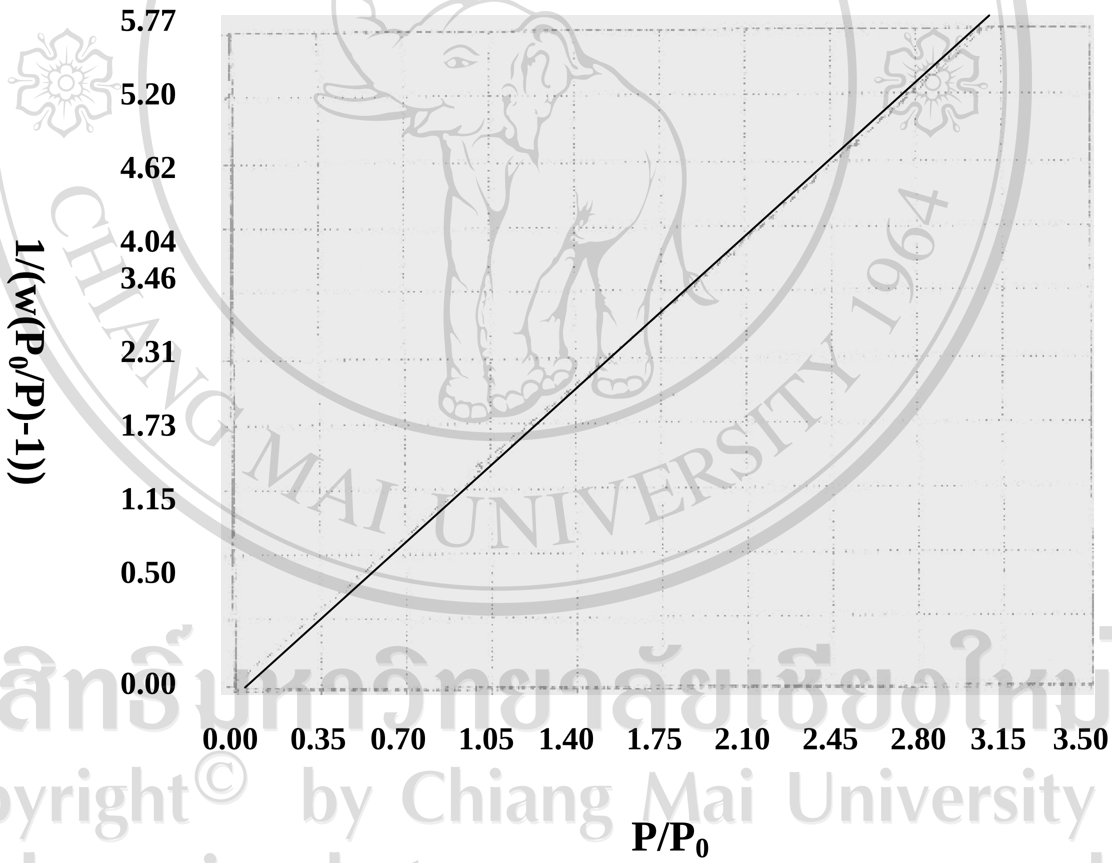
จำนวนสาร 0.0336 กรัม

Absorbate แก๊สไนโตรเจน

Out gas temperature 110 °ซ

Out gas time 6 ชั่วโมง

Analysis time 41.9 นาที



รูป ข-1 กราฟผลการทดลองที่ได้จากการคำนวณโดยใช้สมการของ BET ของผงนาโนซิลิกาโดยใช้สารละลายโซเดียมไฮดรอกไซด์เริ่มต้นที่ความเข้มข้น 2.0 นอร์มอล

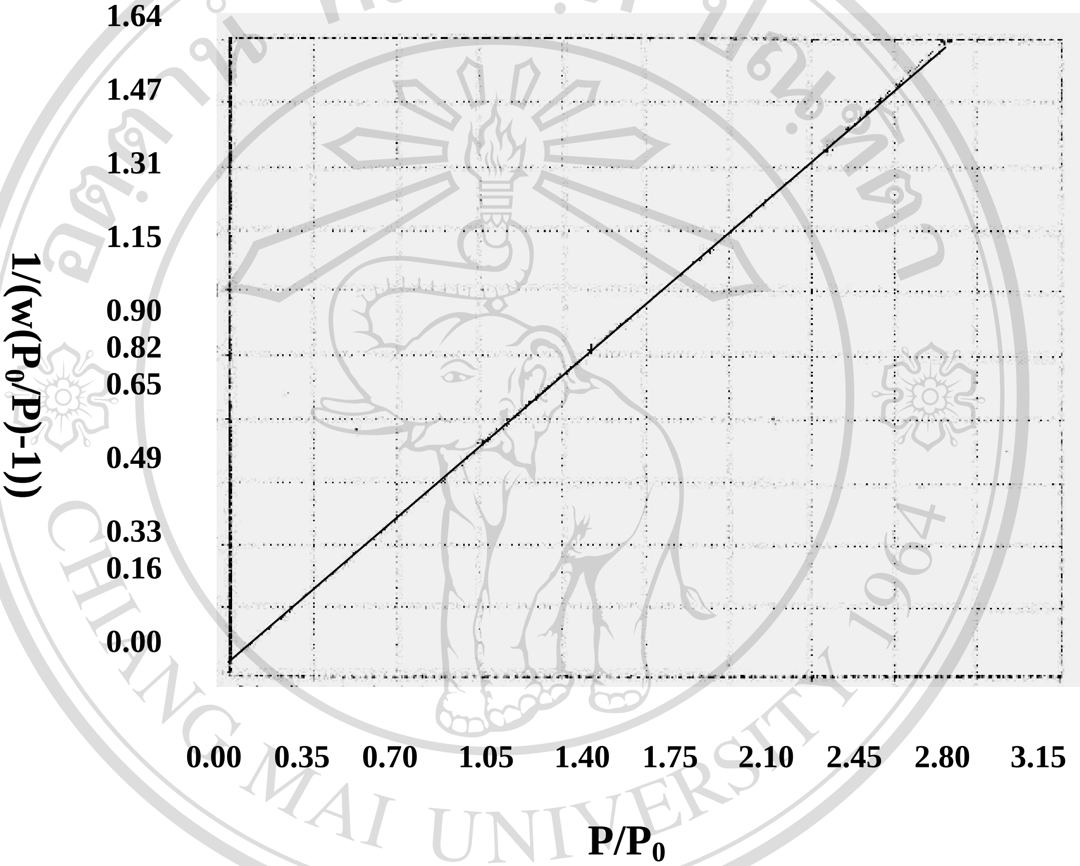
ผลการวิเคราะห์พื้นที่ผิวแบบหลายจุด (multiple point) ผงนาโนซิลิกาที่สังเคราะห์ด้วยสารละลายโซเดียมไฮดรอกไซด์ความเข้มข้น 2.0 นอร์มอล โดยมีพื้นที่ผิวจำเพาะคือ 187 m^2/g

ความชันกราฟเส้นตรง คือ 1.815×10^{2}

จุดตัดแกน Y คือ 3.08×10^{-2}

Correlation Coefficient คือ 0.9996

ค่า C คือ 6.039×10^{2}



รูป ข-2 กราฟผลการทดลองที่ได้จากการคำนวณโดยใช้สมการของ BET ของผงนาโนซิลิกาโดยใช้สารละลายโซเดียมไฮดรอกไซด์เริ่มต้น 2.5 นอร์มอล

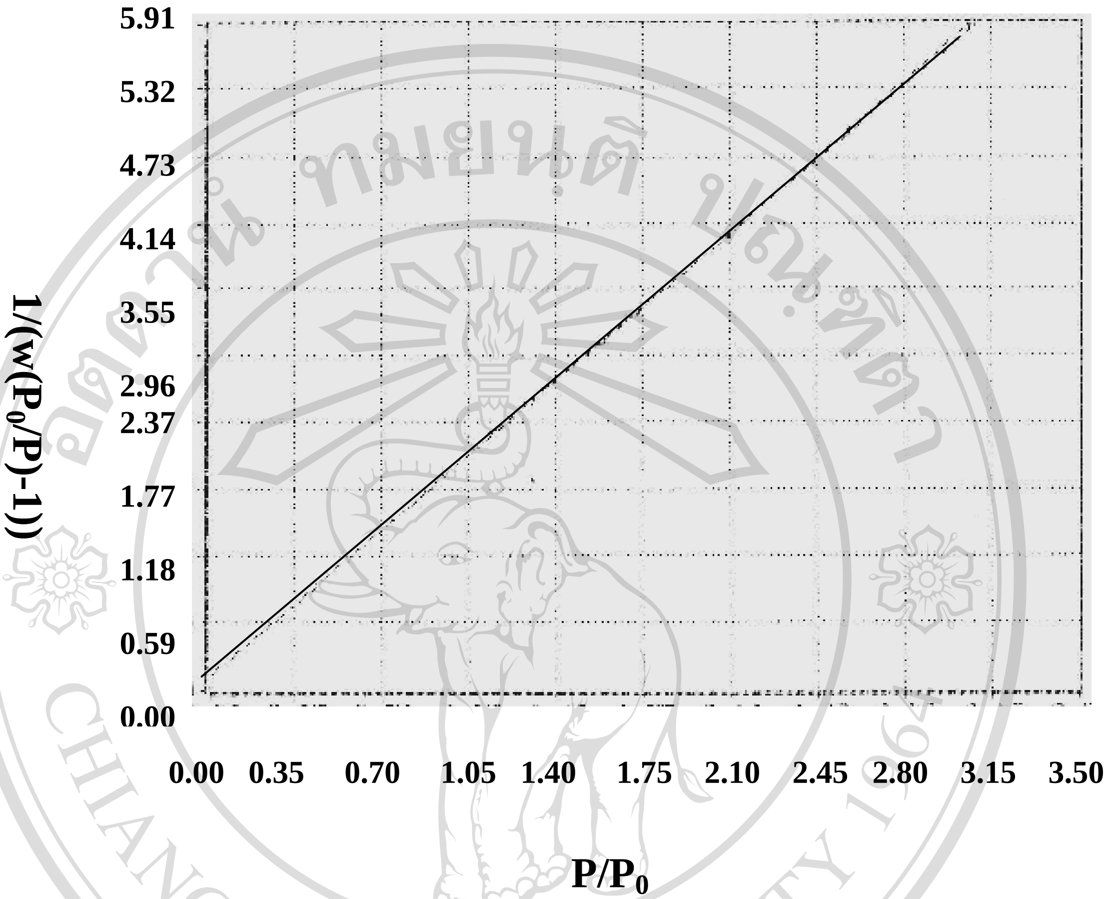
ผลการวิเคราะห์พื้นที่ผิวแบบหลายจุด (multiple point) ผงนาโนซิลิกาที่สังเคราะห์ด้วยสารละลายโซเดียมไฮดรอกไซด์ความเข้มข้น 2.5 นอร์มอล โดยมีพื้นที่ผิวจำเพาะคือ 656 m^2/g

ความชันกราฟเส้นตรง คือ 5.282

จุดตัดแกน Y คือ 2.74×10^{-2}

Correlation Coefficient คือ 0.9998

ค่า C คือ 1.938×10^{2}



รูป ข-3 กราฟผลการทดลองที่ได้จากการคำนวณโดยใช้สมการของ BET ของผงนาโนซิลิกาโดยใช้สารละลายโซเดียมไฮดรอกไซด์เริ่มต้น 3.0 นอร์มอล

ผลการวิเคราะห์พื้นที่ผิวแบบหลายจุด (multiple point) ผงนาโนซิลิกาที่สังเคราะห์ด้วยสารละลายโซเดียมไฮดรอกไซด์ความเข้มข้น 3.0 นอร์มอล โดยมีพื้นที่ผิวจำเพาะคือ 184 m^2/g

ความชันกราฟเส้นตรง คือ 1.881×10^{1}

จุดตัดแกน Y คือ 1.054×10^{-1}

Correlation Coefficient คือ 0.9999

ค่า C คือ 1.795×10^{2}

สภาวะในการวิเคราะห์

สารตัวอย่าง ซิลิกา

ลักษณะผง ผงละเอียด

จำนวนสาร 0.0336 กรัม

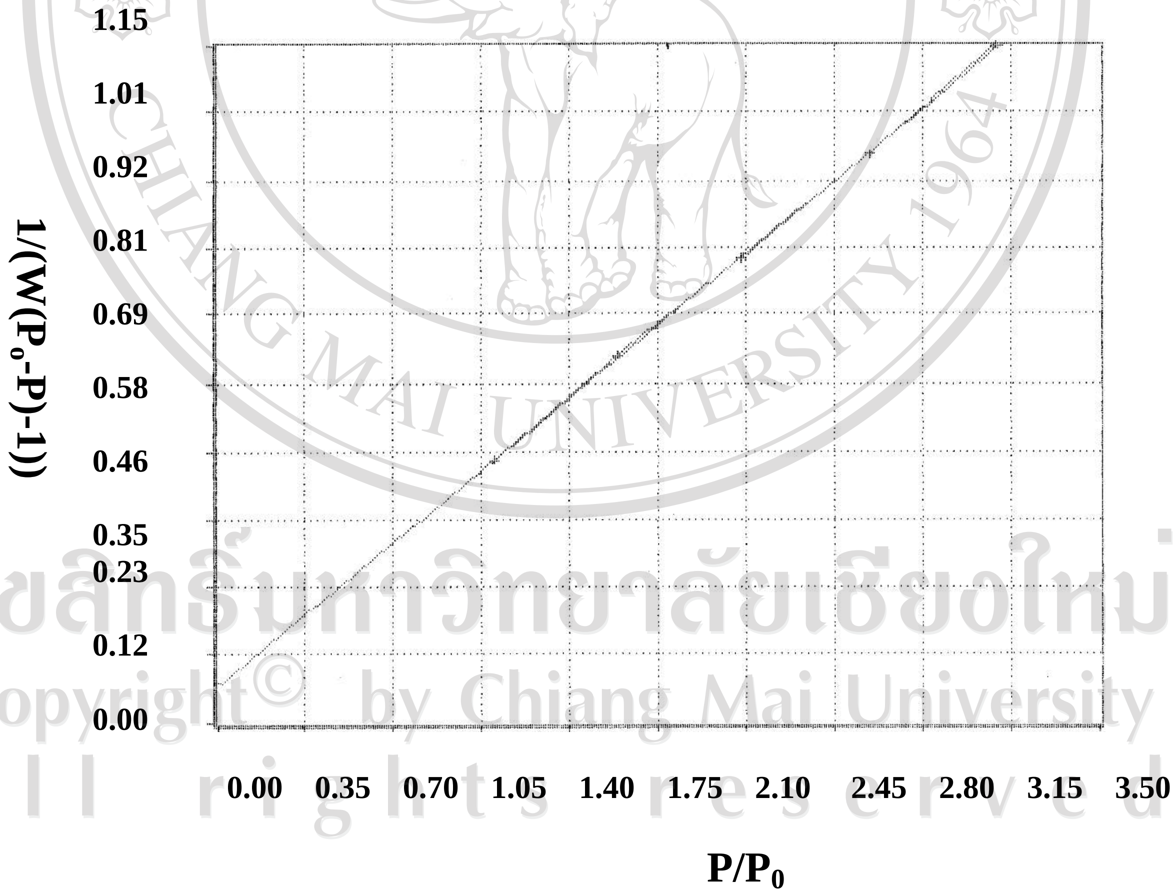
Absorbate แก๊สไนโตรเจน

Out gas temperature 110 °ซ

Out gas time 6 ชั่วโมง

Analysis time 41.9 นาที

ผลการทดลองจะออกมาในลักษณะกราฟเส้นตรง ซึ่งสอดคล้องกับทฤษฎีในหัวข้อ 2.8 และค่าพื้นที่ผิวจำเพาะต่อการศึกษาเวลากวนผสมแตกต่างๆเปรียบเทียบกับขนาดตะกอน จะแสดงได้ดังนี้



รูป ข-4 กราฟผลการทดลองที่ได้จากการคำนวณโดยใช้สมการของ BET ของผงนาโนซิลิกา โดยใช้เวลากวนผสมที่ 8 ชั่วโมง

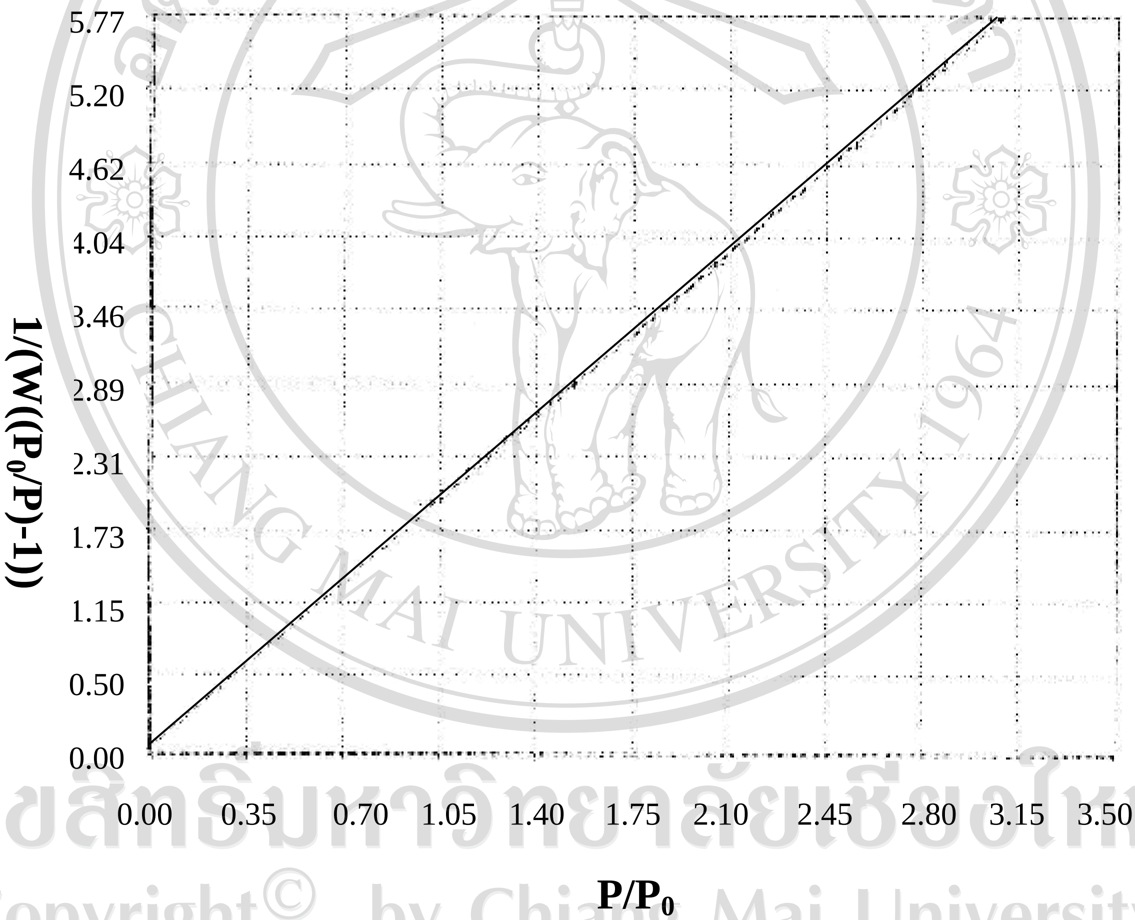
ผลการวิเคราะห์พื้นที่ผิวแบบหลายจุด (multiple point) ผงนาโนซิลิกาที่สังเคราะห์ด้วยสารละลายโซเดียมไฮดรอกไซด์ที่ความเข้มข้น 2.0 นอร์มอล เป็นเวลา 8 ชั่วโมง โดยมีพื้นที่ผิวจำเพาะเท่ากับ 97 m^2/g

ความชันกราฟสันตรงคือ 3.519×10

จุดตันแกน Y คือ 6.120 $\times 10^{-1}$

Correlation Coefficient คือ 0.9998

ค่า C คือ 5.850×10



รูป ข-5 กราฟแสดงผลการทดลองที่ได้จากการทดลองคำนวณโดยใช้สมการ BET ของผงนาโนซิลิกาโดยใช้เวลากวนผสมที่ 10 ชั่วโมง

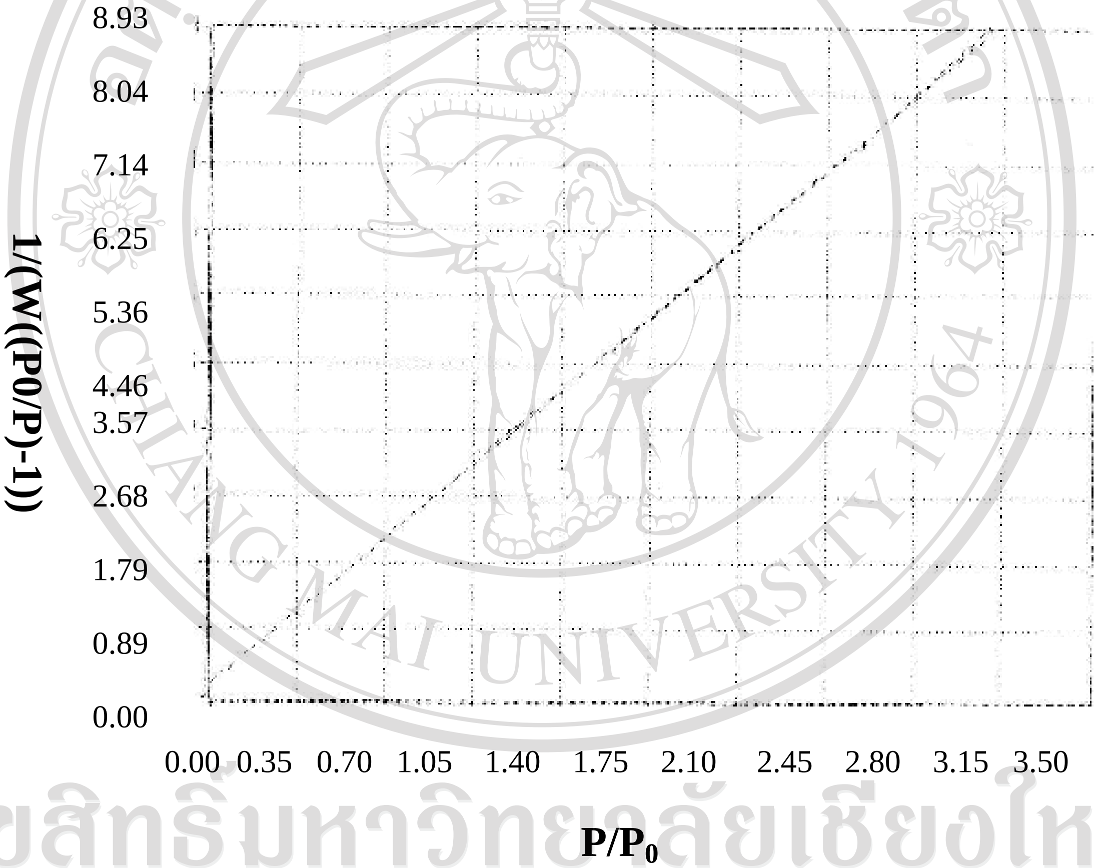
ผลการวิเคราะห์พื้นที่ผิวแบบหลายจุด (multiple point) ผงนาโนซิลิกาที่สังเคราะห์ด้วยสารละลายโซเดียมไฮดรอกไซด์ที่ความเข้มข้น 2.0 นอร์มอล เป็นเวลา 10 ชั่วโมง โดยมีพื้นที่ผิวจำเพาะเท่ากับ 187 m^2/g

ความชันกราฟสันตรงคือ 1.859 ×10

จุดตัดแกน Y คือ 3.084 $\times 10^{-2}$

Correlation Coefficient คือ 0.9996

ค่า C คือ 6.039×10^2



รูป ข-6 กราฟแสดงผลการทดลองที่ได้จากการทดลองคำนวณโดยใช้สมการ BET ของผงนาโนซิลิกาโดยใช้เวลากวนผสมที่ 12 ชั่วโมง

ผลการวิเคราะห์พื้นที่ผิวแบบหลายจุด (multiple point) ผงนาโนซิลิกาที่สังเคราะห์ด้วยสารละลายโซเดียมไฮดรอกไซด์ที่ความเข้มข้น 2.0 นอร์มอล เป็นเวลา 12 ชั่วโมง โดยมีพื้นที่ผิวจำเพาะเท่ากับ 123 m^2/g

ความชันกราฟสันตรงคือ 2.814 $\times 10^2$

จุดตัดแกน Y คือ 2.814 $\times 10^{-1}$ Correlation Coefficient คือ 0.9997 ค่า C คือ 1.841×10^2

ประวัติผู้เขียน

ชื่อ นางสาวนิตยา ทวดอาจ

วัน เดือน ปี เกิด 12 ตุลาคม 2525

ประวัติการศึกษา จบการศึกษาระดับมัธยมศึกษาตอนปลายจาก โรงเรียนสารคามพิทยาคม จังหวัดมหาสารคาม ปีการศึกษา 2544
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จบการศึกษาระดับปริญญาโทหลักสูตร (วท.ม.) สาขาเคมีอุตสาหกรรม ปีการศึกษา 2550

การนำเสนอผลงาน

1. Oral Presented on 7 th National Graduate Research Conference ; 2007 ; 4-5 April / Thailand
2. Oral Presented on The First Thailand National Nanotechnology Conference: Pharmaceutical, Nanomaterials, Devices and Applications at Duang Tawan Hotel ;14-16 August / Thailand
3. Oral Present on The 17 th Thailand Chemical Engineering and Applied Chemistry Conference ; 2007 ; 29-30 October / Thailand

การตีพิมพ์

1. Synthesis and Characterization of Nanosilica from Rice Husk Ash by Precipitate method ในวารสาร Chiang Mai University Journal.