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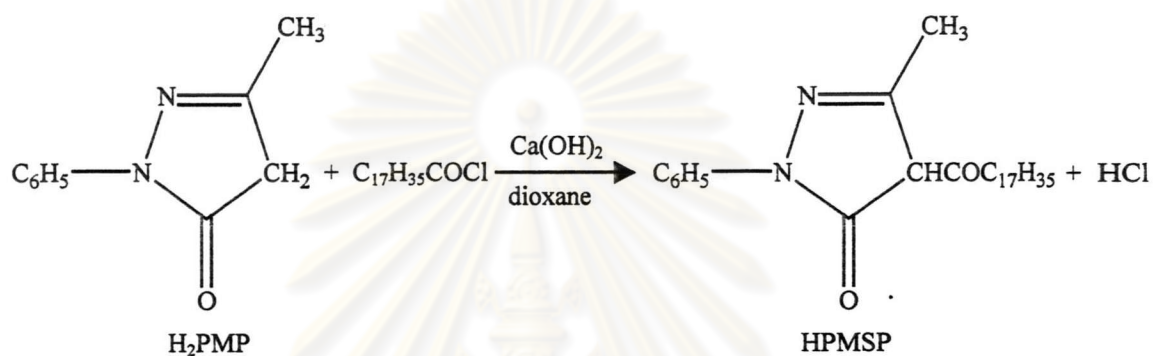ศูนย์วิทยทรัพยากร

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APPENDICES

APPENDIX I

SYNTHESIS OF 1-PHENYL-3-METHYL-4-STEAROYLPYRAZOL-5-ONE (HPMSP)

The preparation of HPMSP is described below.

15.7 g of 3-methyl-1-phenyl-2-pyrazoline-5-one (H_2PMP) were placed in a flask equipped with a stirrer, dropping funnel and a reflux condenser and dissolved in 70 ml dioxane by application of heat at 60°C. 12 g of calcium hydroxide were then slowly added and 30 ml of stearoylchloride were added dropwise within 2 minutes. The reaction mixture became a thick paste and the temperature was increased to 100°C during the first few minute. The mixture was reflux for 30 minutes. The calcium complex in the flask was decomposed by pouring dilute hydrochloric acid (100 mL, 2 M) into the mixture which caused cream coloured crystal to separate. The crystals were collected on a buchner funnel. They were recrystallized from toluene:ethanol (1:9) solution, washed with cold ethanol and dried at 45°C for 3 days. (75.20% yield), m.p. 66-67 °C. ^{1}H-NMR (CDCl$_3$) δ (ppm): 7.87-7.29 (6H, C_6H_5), 2.76 (2H, C(O)-CH_2), 2.51 (s, 3H, CH_3 aromatic), 1.78 (2H, C(O)CH$_2$$CH_2$-(CH$_2$)$_{14}$-CH$_3$), 1.29 (28H, -$(CH_2)_{14}$-), 0.91 (t, 3H, CH_3).
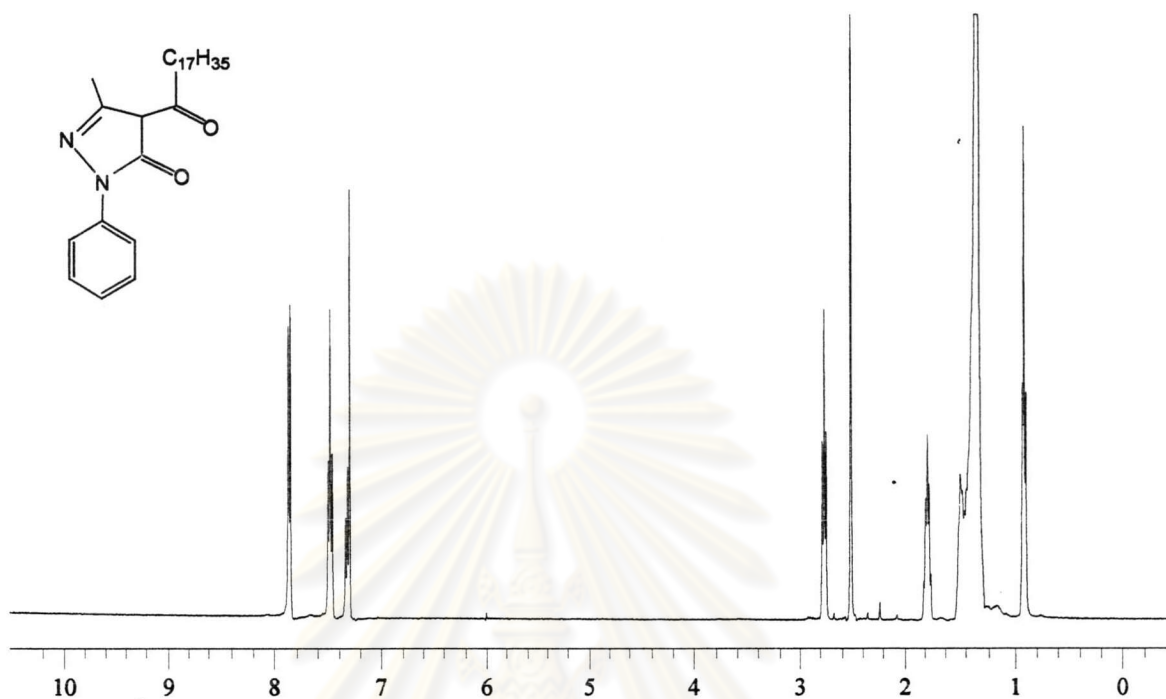
APPENDIX II

^{1}H-NMR spectrum of 1-phenyl-3-methyl-4-stearoylpyrazol-5-one (HPMSP)

<u>Figure II</u> ^{1}H-NMR spectrum of 1-phenyl-3-methyl-4-stearoylpyrazol-5-one in CDCl$_3$ with 400 MHz.

APPENDIX III

SYNTHESIS OF CALCINED MESOPOROUS SILICA

CTAB and an aqueous solution of 0.1 M NaOH were mixed and stirred at 60°C for 4 hours. Methanol was then added. Four hours later, TEOS was added. The final molar composition of the synthesis mixtures was 1 TEOS : 140 H_2O : 0.18 CTAB : 13 MeOH. The mixture was maintained stirring at 60 °C for 1 hour and at ambient temperature for 23 hours. The silica was then filtered, washed copiously with water and 0.001 M HNO_3 until neutral pH and dried at 110 °C overnight. The obtained silica was calcined in air at 540 °C for 10 hours. The synthesis procedure of calcined mesoporous silica was summarized in Figure III.
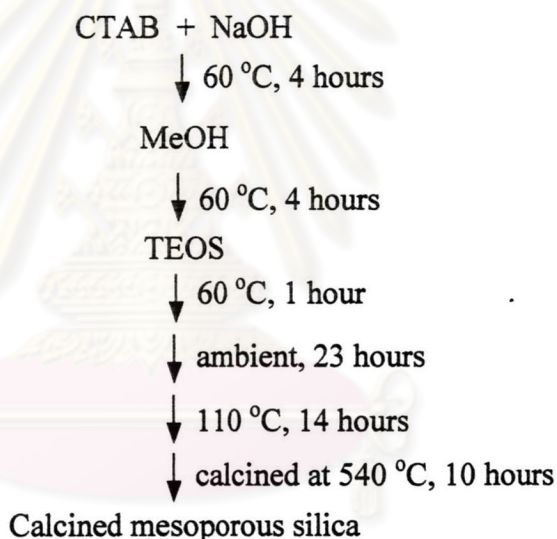
CTAB + NaOH

$\downarrow$ 60 °C, 4 hours

MeOH

$\downarrow$ 60 °C, 4 hours

TEOS

$\downarrow$ 60 °C, 1 hour

$\downarrow$ ambient, 23 hours

$\downarrow$ 110 °C, 14 hours

$\downarrow$ calcined at 540 °C, 10 hours

Calcined mesoporous silica

Figure III The synthesis procedure of calcined mesoporous silica.

APPENDIX IV

CALCULATION OF ORGANIC MATTER CONTENTS
IN MESOPOROUS SILICA

The organic matters in non-doped and HPMSP doped mesoporous silica could be calculated from the mole composition of starting materials. Some calculation examples of organic matter contents in non-doped and HPMSP-doped mesoporous silica from different silica precursors were described below.

IV.1. Non-doped mesoporous silica

<u>Table IV.1</u> The amounts of starting materials used for the synthesis of non-doped mesoporous silica from different silica precursors with the mole ratio of CTAB/Silica precursor equaled to 0.09.

Starting materials		Type of silica precursor		
		TEOS	Calcined mesoporous silica	Silica gel 60
TEOS	(g)	5.2090	-	-
	(mole)	0.0250	-	-
Calcined mesoporous silica	(g)	-	2.4995	-
	(mole)	-	0.0416	-
Silica gel 60	(g)	-	-	2.4995
	(mole)	-	-	0.0416
H_2O*	(g)	63.03	104.84	104.84
	(mole)	3.5019	5.8246	5.8246
CTAB	(g)	0.8202	1.3648	1.3647
	(mole)	0.0023	0.0037	0.0037
MeOH	(g)	10.4144	-	-
	(mole)	0.3250	-	-

*H_2O was introduced to the mixture in the form of 0.1 M NaOH.

IV.1.1. <u>When using TEOS as a silica precursor :</u>

TEOS 0.0250 mole provided SiO_2 0.0250 mole

$$= 0.0250 \times 60.0843 \text{ g} \qquad = 1.5023 \text{ g}$$

CTAB 0.8202 g provided CTA^+ = (0.8202/364.46) x 284.56 g = 0.6404 g

Organic matters in non-doped mesoporous silica = 0.6404 g

Mass of non-doped mesoporous silica = 1.5023 + 0.6404 g = 2.1427 g

Therefore, the percentage of organic matters in non-doped mesoporous silica

= (0.6404/2.1427) x 100 = 29.89 %

IV.1.2. <u>When using calcined mesoporous silica as a silica precursor</u> :

Calcined mesoporous silica 0.0416 mole provided SiO_2 0.0416 mole

= 0.0416 x 60.0843 g = 2.4995 g

CTAB 1.3648 g provided CTA^+ = (1.3648/364.46) x 284.56 g = 1.0529 g

Organic matters in non-doped mesoporous silica = 1.0529 g

Mass of non-doped mesoporous silica = 2.4995 + 1.0529 g = 3.5524 g

Therefore, the percentage of organic matters in non-doped mesoporous

= (1.0529/3.5524) x 100 = 29.64 %

IV.1.3. <u>When using silica gel 60 as a silica precursor</u> :

Silica gel 60 0.0416 mole provided SiO_2 0.0416 mole

= 0.0416 x 60.0843 g = 2.4995 g

CTAB 1.3647 g provided CTA^+ = (1.3647/364.46) x 284.56 g = 1.0529 g

Organic matters in non-doped mesoporous silica = 1.0529 g

Mass of non-doped mesoporous silica = 2.4995 + 1.0529 g = 3.5524 g

Therefore, the percentage of organic matters in non-doped mesoporous silica

= (1.0529/3.5524) x 100 = 29.64 %

IV.2. HPMSP doped mesoporous silica

Table IV.2 The amounts of starting materials used for the synthesis of HPMSP doped mesoporous silica from different silica precursors with the mole ratio of CTAB/Silica precursor equaled to 0.09.

Starting materials		Type of silica precursor		
		TEOS	Calcined mesoporous silica	Silica gel 60
TEOS	(g)	5.2106	-	-
	(mole)	0.0250	-	-
Calcined mesoporous silica	(g)	-	2.4996	-
	(mole)	-	0.0416	-
Silica gel 60	(g)	-	-	2.5015
	(mole)	-	-	0.0416
H_2O*	(g)	63.02	104.84	104.84
	(mole)	3.5012	5.8247	5.8246
CTAB	(g)	0.8203	1.3648	1.3647
	(mole)	0.0023	0.0037	0.0037
MeOH	(g)	10.4151	-	-
	(mole)	0.3251	-	-
HPMSP	(g)	0.6602	1.0993	1.0993
	(mmole)	1.5005	2.4983	2.4983

*H_2O was introduced to the mixture in the form of 0.1 M NaOH.

IV.2.1. When using TEOS as a silica precursor :

TEOS 0.0250 mole provided SiO_2 0.0250 mole

$$= \quad 0.0250 \times 60.0843 \text{ g} \quad\quad = 1.5028 \text{ g}$$

CTAB 1.6402 g provided CTA^+ $= \quad (0.8203/364.46) \times 284.56 \text{ g} \quad = 0.6405 \text{ g}$

Amount of HPMSP in silica $= \quad 1.5005 \times 10^{-3} \times 440 \text{ g} \quad = 0.6602 \text{ g}$

Organic matters in HPMSP doped mesoporous silica

$$= \quad 0.6405 + 0.6602 \text{ g} \quad\quad = 1.3007 \text{ g}$$

Mass of HPMSP doped mesoporous silica

$$= \quad 1.5028 + 0.6405 + 0.6602 \text{ g} \quad = 2.8035 \text{ g}$$

Therefore, the percentage of organic matters in HPMSP doped mesoporous silica

$$= \quad (1.3007/2.8035) \times 100 \quad\quad = 46.40 \text{ %}$$

IV.2.2. <u>When using calcined mesoporous silica as a silica precursor :</u>

Calcined mesoporous silica 0.0416 mole provided SiO_2 0.0416 mole

	=	0.0416 x 60.0843 g	= 2.4996 g
CTAB 1.3648 g provided CTA^+	=	(1.3648/364.46) x 284.56 g	= 1.0529 g
Amount of HPMSP in silica	=	2.4983×10^{-3} x 440 g	= 1.0993 g

Organic matters in HPMSP doped mesoporous silica

	=	1.0529 + 1.0993 g	= 2.1522 g

Mass of HPMSP doped mesoporous silica

	=	2.4996 + 1.0529 + 1.0993 g	= 4.6518 g

Therefore, the percentage of organic matters in HPMSP doped mesoporous silica

	=	(2.1522/4.6518) x 100	= 46.26 %

IV.2.3. <u>When using silica gel 60 as a silica precursor :</u>

Silica gel 60 0.0416 mole provided SiO_2 0.0416 mole

	=	0.0416 x 60.0843 g	= 2.5015 g
CTAB 1.3647 g provided CTA^+	=	(1.3647/364.46) x 284.56 g	= 1.0529 g
Amount of HPMSP in silica	=	2.4983×10^{-3} x 440 g	= 1.0993 g

Organic matters in HPMSP doped mesoporous silica

	=	1.0529 + 1.0993 g	= 2.1522 g

Mass of HPMSP doped mesoporous silica

	=	2.5015 + 1.0529 + 1.0993 g	= 4.6537 g

Therefore, the percentage of organic matters in HPMSP doped mesoporous silica

	=	(2.1522/4.6537) x 100	= 46.25 %

APPENDIX V

CALCULATION OF ELEMENTAL MASS

The percentage of elemental mass in each mesoporous silica could be determined from the mole composition of starting materials. Some examples of calculation were described below.

Table V The amounts of starting materials used for the synthesis of silica.

Starting materials	Non-doped silica (CTAB/TEOS = 0.18)		HPMSP doped silica (CTAB/TEOS = 0.18)	
	(g)	(mmol)	(g)	(mmol)
TEOS	5.2090	25.004	5.2106	25.011
H_2O*	63.02	3501.1	63.02	3501.2
CTAB	1.6404	4.5008	1.6402	4.5010
MeOH	10.428	325.47	10.4151	325.10
HPMSP	-	-	0.6601	1.5003

*H_2O was introduced to the mixture in the form of 0.1 M NaOH.

V.1. Non-doped mesoporous silica

TEOS 25.004 mmole provided SiO_2 25.004 mmole

$$= 25.004 \times 10^{-3} \times 60.0843 \text{ g} \quad = 1.5023 \text{ g}$$

CTAB 1.6404 g provided CTA^+ = (1.6404/364.46) × 284.56 g = 1.2808 g

Mass of non-doped mesoporous silica = 1.5023 + 1.2808 g = 2.7831 g

Then, $\quad$ % C $= \dfrac{(N_C \times W_C \times n_{CTAB})}{\text{Mass of silica}} \times 100$

$\quad$ % H $= \dfrac{(N_H \times W_H \times n_{CTAB})}{\text{Mass of silica}} \times 100$

$\quad$ % N $= \dfrac{(N_N \times W_N \times n_{CTAB})}{\text{Mass of silica}} \times 100$

Where $\quad N_C$ = number of carbon atoms in silica

$\quad N_H$ = number of hydrogen atoms in silica

$\quad N_N$ = number of nitrogen atoms in silica

$\quad n_{CTAB}$ = number of moles of CTAB

$$W_C = \text{Atomic weight of carbon (12.0110)}$$

$$W_H = \text{Atomic weight of hydrogen (1.0079)}$$

$$W_N = \text{Atomic weight of nitrogen (14.0067)}$$

Thus,

$$\% \, C = \frac{(19 \times 12.0110 \times 4.5008 \times 10^{-3})}{2.7831} \times 100$$

$$= 36.91 \, \%$$

$$\% \, H = \frac{(42 \times 1.0079 \times 4.5008 \times 10^{-3})}{2.7831} \times 100$$

$$= 6.85 \, \%$$

$$\% \, N = \frac{(1 \times 14.0067 \times 4.5008 \times 10^{-3})}{2.7831} \times 100$$

$$= 2.27 \, \%$$

V.2. HPMSP doped mesoporous silica

TEOS 25.011 mmole provided SiO_2 25.011 mmole

$$= 25.011 \times 10^{-3} \times 60.0843 \, g \qquad = 1.5028 \, g$$

CTAB 0.8202 g provided CTA^+ $\quad = (1.6402/364.46) \times 284.56 \, g \quad = 1.2808 \, g$

Amounts of HPMSP in silica $\quad = (1.5003 \times 10^{-3}) \times 440 \, g \quad = 0.6601 \, g$

Mass of non-doped mesoporous silica $\quad = 1.5028 + 1.2808 + 0.6601 \, g$

$$= 3.4437 \, g$$

Then,

$$\% \, C = \frac{(N_C \times W_C \times n_{CTAB}) + (N_C \times W_C \times n_{HPMSP})}{\text{Mass of silica}} \times 100$$

$$\% \, H = \frac{(N_H \times W_H \times n_{CTAB}) + (N_H \times W_H \times n_{HPMSP})}{\text{Mass of silica}} \times 100$$

$$\% \, N = \frac{(N_N \times W_N \times n_{CTAB}) + (N_N \times W_N \times n_{HPMSP})}{\text{Mass of silica}} \times 100$$

Where $\quad n_{HPMSP} = \text{number of moles of HPMSP}$

Thus,

$$\% \, C = \frac{(19 \times 12.0110 \times 4.5004 \times 10^{-3}) + (28 \times 12.0110 \times 1.5003 \times 10^{-3})}{3.4437} \times 100$$

$$= 44.48 \, \%$$

$$\% \, H = \frac{(42 \times 1.0079 \times 4.5004 \times 10^{-3}) + (44 \times 1.0079 \times 1.5003 \times 10^{-3})}{3.4437} \times 100$$

$$= 7.46 \, \%$$

$$\% \, N = \frac{(1 \times 14.0067 \times 4.5004 \times 10^{-3}) + (2 \times 14.0067 \times 1.5003 \times 10^{-3})}{3.4437} \times 100$$

$$= 3.05 \, \%$$

CURRICULUM VITAE

Chinnawat Satsananan was born on April 10th, 1979 in Lamphun, Thailand. He received a Bachelor's degree of Science, majoring in Chemistry at Chiang Mai University in 2002. Since 2003, he has been a graduate student studying in the Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University.

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