# SYNTHESIS AND CHARACTERIZATIONS OF POLYBENZOXAZINE-BASED CARBON XEROGELS

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#### ABSTRACT

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In this present work, the porous carbon, called carbon xerogels, derived from polybenzoxazine (PBZ) were prepared via a sol-gel process prior to the carbonization. By using different types of solvents, the cluster growth behaviour of PBZ during the sol-gel process were different due to the differences in solubility parameters between PBZ and solvents, resulting in different porous properties of carbon xerogels. The solvent with low dielectric constant like dioxane provides the slowest rate of cluster growth and also the smallest clusters of PBZ, yielding carbon xerogel with small particles sizes and pore diameter of 40-200 nm after carbonization. This is in part related to the tendency of the oxazine ring-opening in the solvent with large dielectric constant. PBZ-based carbon xerogels obtained through dioxane system were effectively used as a template to control the size of ZSM-5 catalyst into nanoscale (27-70 nm). On the other hand, the nanosphere and microsphere of carbon xerogels were obtained by using cationic (hexadecyltrimethylammonium bromide) and non-ionic (polyethylene glycol nonylphenyl ether) surfactants, respectively. In additon, high specific surface (518-899 m<sup>2</sup>/g) area and extremely high total pore volume  $(1.34-6.05 \text{ cm}^3/\text{g})$  of carbon xerogels could be obtained by varying the amounts of silica loading as a hard template. Mesopore diameter of carbon xerogels was also controlled by varying either the concentrations of cationic surfactant or particle sizes of the silica hard template.

iii

# บทคัดย่อ

อุเทน ทับทรวง : การสังเคราะห์คาร์บอนซีโรเจลจากโพลีเบนซอกซาซีนและการพิสูจน์ คุณลักษณะ (Synthesis and Characterizations of Polybenzoxazine-based Carbon Xerogels) อ. ที่ ปรึกษา: ผู้ช่วยศาสตราจารย์ คร. ธัญญลักษณ์ ฉายสุวรรณ์ ศาสตราจารย์ คร. ฮัทซุโอะ อิชิคะ และ รองศาสตราจารย์ คร. สุจิตรา วงศ์เกษมจิตต์ 146 หน้า

งานวิจัยนี้มุ่งเน้นการศึกษาและการพัฒนาคาร์บอนซีโรเจลจากโพลีเบนซอกซาซีนโดย ้สังเคราะห์ผ่านกระบวนการโซลเจลและเปลี่ยนเป็นคาร์บอนโคยการเผาที่อุณหภูมิสูงภายใต้ ้บรรยากาศเฉื่อย ในระหว่างกระบวนการโซลเจลหากมีการใช้ตัวทำละลายที่ต่างชนิคกัน พฤติกรรมการเกิดเจลและการขยายตัวของคลัสเตอร์ของโพลีเบนซอกซาซีนจะแตกต่างกันอัน เนื่องมาจากค่าดัชนีการละลาย (Solubility parameter) ที่แตกต่างกันระหว่างตัวทำละลายและโพลี เบนซอกซาซีน อัตราการขยายตัวของกลัสเตอร์จะช้าและมีขนาดกลัสเตอร์ที่เล็กในตัวทำละลายที่มี ้ ค่าคงที่ใดอิเล็กทริกต่ำเช่นใดออกเซน ดังนั้นหลังจากการเผาจะทำให้เกิดการ์บอนที่มีขนาดคลัส เตอร์เล็กและมีรูพรุนในระคับนาโนเมตร (40-200 นาโนเมตร) ปรากฏการณ์นี้สามารถอธิบายได้ ้โดยพฤติกรรมการเปิดวงออกซาซีน ซึ่งถูกเปิดได้ง่ายในตัวทำละลายที่มีค่าคงที่ไดอิเล็กทริกสง เช่น ใคเมทิลฟอร์มาไมค์ มากกว่าตัวทำละลายที่มีค่าคงที่ใคอิเล็กทริกต่ำ เช่น ใคออกเซน คาร์บอน ซีโรเจลที่ได้จากระบบของไดออกเซนถูกนำมาใช้เป็นแม่แบบที่มีประสิทธิภาพในการสังเคราะห์ ตัวเร่งปฏิกิริยา ZSM-5 ที่มีขนาดอนุภาคระดับนาโน นอกจากนี้ขนาดคลัสเตอร์ของคาร์บอนซีโร เจลยังสามารถถูกควบคุมให้เป็นทรงกลมที่มีขนาคเล็กในระดับนาโนและระดับไมครอนได้โคยใช้ สารถดแรงตึงผิวที่มีประจุลบ (Hexadecyltrimethylammonium bromide) และสารถดแรงตึงผิวที่ไม่ มีประจุ (polyethylene glycol nonylphenyl ether) ตามลำคับ ปริมาตรของรูพรุนและพื้นที่ผิวของ ้คาร์บอนซีโรเจลยังสามารถถูกพัฒนาให้มีค่าสูงขึ้นได้ถึง 1.34-6.05 ถูกบาศก์เซนติเมตรต่อกรับ และ 518-899 ตารางเมตรต่อกรัม ตามลำคับโคยการใส่อนุภาคซิลิกาเป็นแม่แบบในการสร้างรูพรุน อีกทั้งขนาครูพรุนในระดับเมโซ (2-50 นาโนเมตร) ของคาร์บอนซีโรเจลยังสามารถถูกควบคุมได้ ้โดยการเปลี่ยนแปลงความเข้มข้นของสารลดแรงตึงผิวที่มีประจุลบและขนาดอนุภาคของแม่แบบซิ ລີ້ຄາ

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## **TABLE OF CONTENTS**

		PAGE
Tit	le Page	i
Ab	stract (in English)	iii
Ab	stract (in Thai)	iv
Ac	knowledgements	v
Tal	ble of Contents	vi
Lis	t of Tables	х
Lis	t of Figures	xii -
Lis	t of Schemes	xviii
CHAPT	ER	
Ι	INTRODUCTION	1
II	LITERATURE REVIEW	-
	2.1 ZSM-5	5
	2.1.1 Structure and Properties	5
	2.1.2 Synthesis of ZSM-5 via Microwave Irradiation	6
	2.1.3 Mesoporous ZSM-5	7
	2.1.4 Nanosized ZSM-5	8
	2.2 Polybenzoxazine	10
	2.2.1 Synthesis of Polybenzoxazine	10
	2.2.2 Properties of Polybenzoxazine	11
	2.3 Porous Carbon Derived via the Sol-Gel Process	12
	2.3.1 Types of Porous Carbon Derived via	
	the Sol-Gel Process	12

-

-

1.2

<b>CHA</b>	PTER
------------	------

.

Π

2.4 Porous Carbon Derived by Using Template	16		
2.4.1 Porous Carbon Derived by Using Surf	actant		
as Soft Template	16		
2.4.2 Porous Carbon Derived by Using Silic	а		
as Hard Template	17		
2.5 Points of Study	18		
	-		
SELF-FORMATION OF 3D INTERCONNEG	CTED		
MACROPOROUS CARBON XEROGELS DERIVED FROM			
POLYBENZOXAZINE BY SELECTIVE SOLVENT DURING			
THE SOL-GEL PROCESS			
3.1 Abstract	19		
2.2. Introduction	20		

3.2 Introduction	20
3.3 Experimental	23
3.4 Results and Discussion	26
3.5 Conclusions	47
3.6 Acknowledgements	49
3.7 References	50

# IV NOVEL TEMPLATE CONFINEMENT DERIVED FROM POLYBENZOXAZINE-BASED CARBON XEROGELS FOR SYNTHESIS OF ZSM-5 NANOPARTICLES VIA MICROWAVE IRRADIATION

4.1 Abstract	56
4.2 Introduction	57
4.3 Experimental	58
4.4 Results and Discussion	61
4.5 Conclusions	74

	4.6 Acknowledgements	75
	4.7 References	76
V	EFFECTS OF NON-IONIC AND CATIONIC SURFA	CTANT
	ON POROUS STRUCTURE OF POLYBENZOXAZI	NE
	-BASED CARBON XEROGELS	
	5.1 Abstract -	79
	5.2 Introduction	80
	5.3 Experimental	82
	5.4 Results and Discussion	84
	5.5 Conclusions	96
	5.6 Acknowledgements	97
	5.7 References	98
	÷.	
VI	IMPROVEMENT IN THE PORE STRUCTURE OF	
	POLYBENZOXAZINE-BASED CARBON XEROGE	LS -
	THROUGH A SILICA TEMPLATING METHOD	
	6.1 Abstract	103
	6.2 Introduction	104
	6.3 Experimental	105
	6.4 Results and Discussion	110
	6.5 Conclusions	124
	6.6 Acknowledgements	125
	6.7 References	126
VII	CONCLUSIONS AND RECOMMENDATIONS	129
	REFERENCES	131

•

4

CHAPTER

•

PAGE

viii

.

-

-

APPENDICES		142
Appendix A	Synthesis of Polybenzoxazine	142
Appendix B Peak Positions and Assignments in the FTIR		
	Spectrum of Benzoxazine	143
Appendix G	Peak Positions and Assignments in the <sup>1</sup> H-NMR	
	Spectrum of Benzoxazine	144

## **CURRICULUM VITAE**

.

-

145

## LIST OF TABLES

	TABL	E	PAGE
8		CHAPTER II	
	2.1	Characteristics of ZSM-5 derived by various methods	9
	2.2	Characteristics of porous carbon derived from various	
		starting materials and preparation methods	15
		CHAPTER III	
-	3.1	Pore characteristics of carbon xerogels using different	
		solvents by ambient pressure drying at 25% w/w of solid	
		contents	32
	3.2	Pore characteristics of carbon xerogels using DMF as a	
		solvent by ambient pressure drying at different	
		concentrations	41
	3.3	Pore characteristics of carbon xerogels using DMF as a	
		solvent at 35% w/w of solid contents by ambient pressure	
		drying and supercritical CO <sub>2</sub> drying	45
		CHAPTER IV	
	4.1	Pore structure of PBZ-based carbon xerogels using DMF and	
		dioxane as solvents via ambient pressure drying at	
		concentration of 45% w/w	63
	4.2	Pore structure of ZSM-5 formed at 150 °C for 6 h after 5 h	
		of aging time	73

-

.

.

.

.

-

## PAGE

## **CHAPTER V**

5.1	Pore structure of PBZ-based carbon xerogels prepared from	
	10%w/w of benzoxazine precursor using different	
	concentrations of CTAB	87
5.2	Pore structure of PBZ-based carbon xerogels prepared from	
	10%w/w of benzoxazine precursor using different	
	concentrations of Synperonic NP30	94

### **CHAPTER VI**

6.1	Pore structure of PBZ-based carbon xerogels obtained after	
	removal of silica nanoparticle templates	114
6.2	Comparison between porous organics/carbons derived from	
	polybenzoxazine and resorcinol-formaldehyde polymer by	
	various synthesis methods	121

### APPENDICES

Bl	Peak positions and assignments in the	FTIR spectrum of	
	resulting benzoxazine precursors		143
C1 Peak positions and assignments in the <sup>1</sup> H-NMR spectrum of			
	resulting benzoxazine precursors		144

#### **LIST OF FIGURES**

#### **FIGURE** PAGE **CHAPTER II** 2.1 Structure of ZSM-5. 6 2.2 Synthesis reaction of polybenzoxazine. 10 **CHAPTER III** 3.1 Densities and gelation times of carbon xerogels at 25 % w/w using DMF (C-DX-25), dioxane (C-DiX-25), and isopropanol (C-IX-25) as solvents by ambient pressure drying. 27 3.2 Size of cluster during gelation process at 25 %w/w using DMF (C-DX-25), dioxane (C-DiX-25), and isopropanol (C-IX-25) as solvents. 28 3.3 SEM micrographs of carbon xerogels using DMF, isopropanol, and dioxane as solvents by ambient pressure drying (inset: high magnification) (a) C-DX-25 (b) C-IX-25 (c) C-DiX-25 (d) self-micelle-like formation model. 30 3.4 (a) N<sub>2</sub> adsorption-desorption isotherms of carbon xerogels using DMF (C-DX-25), dioxane (C-DiX-25), and isopropanol (C-IX-25) as solvents by ambient pressure drying (b) pore size distribution. 32 3.5 TEM images of carbon xerogels at 25% w/w (a) C-DX-25 (b) C-DiX-25 (c) C-IX-25; upper: low magnification; lower: high magnification. 33 3.6 Cluster formation during phase separation process (a) C-DX-25 (b) C-DiX-25. 35

		-	-	-	
٦	,	ъ.	1	1	
	c	×		1	
۰	•	٠		۰	

PAGE

-

3.7	Densities and gelation times of carbon xerogels at different		
	concentrations using DMF as a solvent by ambient pressure		
	drying.	38	
3.8	SEM micrographs of carbon xerogels using DMF as a		
	solvent by ambient pressure drying (a) C-DX-25 (b) C-DX-		
	35 (c) C-DX-45.	39	
3.9	(a) $N_2$ adsorption-desorption isotherms of carbon xerogels		
	using DMF as a solvent at different concentrations (b) pore	-	
	size distribution.	40	
3.10	Size of cluster during gelation process at different		
	concentrations using DMF as a solvent.	42	
3.11	(a) $N_2$ adsorption-desorption isotherms of carbon xerogels		
	using DMF as a solvent by (a) ambient pressure drying (C-		
	DX-35) and supercritical CO <sub>2</sub> drying (C-DA-35) (b) pore		
	size distribution.	44	
3.12	SEM micrographs of porous carbon using DMF as a solvent		
	by (a) ambient pressure drying (C-DX-35) and (b)		
	supercritical CO <sub>2</sub> drying (C-DA-35); inset: high		
	magnification.	46	
3.13	Structure of MCBP(BA-teta) and cross linked MCBP(BA-		
	teta).	46	

### FIGURE

-

÷.

.

•

-

•

## **CHAPTER IV**

4.1	Adsorption isotherms of PBZ-based carbon xerogels	
	prepared from 45% w/w of benzoxazine precursor using ( $\blacktriangle$ )	
	DMF (CXDM) [19] and ( $\bigcirc$ ) Dioxane (CXDI) as solvents	
	via ambient pressure drying.	63
4.2	Pore structure of PBZ-based carbon xerogels prepared from	
	45% w/w of benzoxazine precursor using (a) DMF (CXDM)	
	[19] and (b) Dioxane (CXDI) as solvents via ambient	
	pressure drying.	64
4.3	XRD patterns of ZSM-5 formed at 150 °C for 6 h after 5 h	
	aging time using (a) CXDI, (b) CXDM as hard template, (c)	
	reference ZSM-5 without hard template carbon, FCP =	
	fraction of crystalline phase.	67
4.4	SEM micrographs of ZSM-5 formed at 150 °C for 6 h after 5	
	h aging time; (a) microsized ZSM-5 formed in the opened	
	structure of CXDM, (b) microsized ZSM-5 with monolithic	
	pore after calcinations, (c)-(d) nanosized ZSM-5 formed in	
	the pores of CXDI.	67
4.5	TEM images of ZSM-5 formed at 150 °C for 6 h after 5 h	
	aging time, using carbon xerogels as hard template; (a)-(b)	
	ZSM-5-CXDI (c) ZSM-5-CXDM (d) Particle size	
	distribution of ZSM-5-CXDI obtained from dynamic light	
	scattering.	68
4.6	Crystal lattice of nanosized ZSM-5 formed at 150 °C for 6 h	
	after 5 h aging time, using CXDI as hard template.	69
4.7	FTIR spectra of ZSM-5 formed at 150 °C for 6 h after 5 h	
	aging time; (a) ZSM-5 reference (b) ZSM-5-CXDM (c)	
	ZSM-5-CXDI.	70

xiv

### **GHAPTER V**

5.1	SEM micrographs of PBZ-based carbon xerogels prepared	
	from 10% w/w of benzoxazine precursor using different	
-	concentrations of CTAB; (a) no added CTAB, (b) 0.003 M,	
	(c) 0.009 M, (d) 0.030 M, (e) 0.090 M, and (f) 0.180 M; 1:	
	low magnification; 2: high magnification.	84
5.2	Scheme of the benzoxazine-micelle formation; (a)	
	nanospheres for CTAB system, (b) microspheres for	
	Synperonic NP30 system.	86
- 5.3	N2 adsorption-desorption isotherms of polybenzoxazine-	
	based carbon xerogels using different concentrations of	
	CTAB.	89
5.4	Mesopore size distributions of polybenzoxazine-based	
	carbon xerogels using different concentrations of CTAB,	
	determined by BJH method.	89
5.5	TEM images of PBZ-based carbon xerogels prepared from	
	10% w/w of benzoxazine precursor using 0.090 molar of	
	CTAB; (a) low magnification, (b) high magnification.	90
5.6	SEM micrographs of PBZ-based carbon xerogels prepared	
	from 10% w/w of benzoxazine precursor using different	
	concentrations of Synperonic NP30; (a) no added CTAB, (b)	
	0.003 M, (c) 0.009 M, (d) 0.030 M, (e) 0.090 M, and (f)	
	0.180 M; 1: low magnification; 2: high magnification.	92

.

-

 $-\pi$ 

PAGE

## FIGURE

4

xvi

5.7	N <sub>2</sub> adsorption-desorption isotherms of polybenzoxazine-	
	based carbon xerogels using different concentrations of	
	Synperonic NP30.	95
5.8	Mesopore size distributions of polybenzoxazine-based	
	carbon xerogels using different concentrations of Synperonic	
	NP30, determined by BJH method.	95
5.9	Micropore size distributions of polybenzoxazine-based	
	carbon xerogels using different concentrations of Synperonic	
-	NP30, determined by MP method.	96

## **CHAPTER VI**

6.1	Polycondensation reaction and ring-opening polymerization	
	of benzoxazine.	106
6.2	Schematic illustration of synthesis process.	108
6.3	Percentage of composite yield of polybenzoxazine xerogel	
	synthesized by using various amounts of silica loading as	
	template.	111
6.4	EDX spectrum of polybenzoxazine-based carbon xerogel	
	assembled with 30% w/w of silica nanoparticles template.	112
6.5	Morphology of polybenzoxazine-based carbon xerogel: (a)	
	carbon xerogel without silica nanoparticles template	
	(reference carbon xerogel), (b) carbon xerogel with silica	
	nanoparticles loading of 50% w/w, and (c) carbon xerogel	
	after removal of silica nanoparticles loading of 50% w/w	
	(upper: low magnification, lower: high magnification).	113
6.6	Adsorption isotherm of polybenzoxazine-based carbon	
	xerogel obtained after removal of silica nanoparticles.	115

FIGURE		PAGE
6.7	Mesopore size distributions of polybenzoxazine-based carbon xerogels obtained after removal of silica	
	nanoparticles.	116
6.8	The effects of percentage of silica loading on a) specific	
	surface area and b) pore volume of polybenzoxazine-based	
	carbon xerogel obtained after removal of silica nanoparticles.	
		117

## **APPENDICES**

Al	Synthesis reaction of polybenzoxazine.	142
----	--	-----

### **LIST OF SCHEMES**

## SCHEME

÷ .

## PAGE

## CHAPTER II

2.1	Scope of research works.	18
-		
	CHAPTER IV	
	-	

4.1	Synthesis diagram of PBZ-based carbon xerogels.	60
4.2	Formation of benzoxazine wet gel during the sol-gel process.	66

