

**NOVEL POLYMER FOAM VIA POLYMERIZED HIGH INTERNAL PHASE  
EMULSION (POLYHIPES)**



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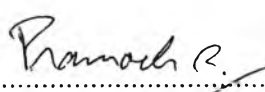
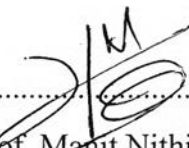



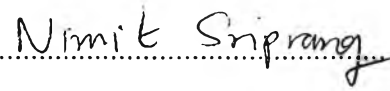
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## ABSTRACT

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Key words: High internal phase emulsion/ PolyHIPE/ Bentonite/ Organo-modified clay/ Porous clay heterostructure/ Gas adsorption

In order to obtain polyHIPE polymeric foam with improved properties for performance in ever-broadening applications i.e. adsorbent for CO<sub>2</sub> adsorption and scaffold for tissue engineering application, the present work focuses on how to elevate the overall properties of polymerized high internal phase emulsion (PolyHIPE porous foam) to above their inherent values including studying the effect of surfactant system, Soxhlet extraction time, addition of organoclay as inorganic filler and also plasma surface modification technique.

Plasma surface modification technique was used to improve the surface properties of polyHIPE scaffold in tissue engineering applications. After surface modification, poly(S/EGDMA)polyHIPE scaffold prepared from styrene and ethylene glycol dimethacrylate monomers with greater hydrophilic properties, were obtained leading to improve the interaction between the living cells and the polyHIPE substrate. The amount of cell adhesion and proliferation was further increased with the utilization of the surface modification technique via atmospheric pressure plasma treatment that would impart the hydrophilic improvement to the polyHIPE scaffold surface due to the polar-like property of the biofluid cell medium.

With the aim of designing suitable adsorbent materials i.e. high surface area with superior mechanical properties and also good adsorption capacity that would adsorb such gases before being liberated into the environment, polymerized high internal phase emulsion of divinylbenzene; poly(DVB)polyHIPE foam was successfully prepared by using two different systems of three-component surfactants

and toluene as porogenic solvent (S20M\_T and S80M\_T). Samples prepared using S20M\_T and S80M\_T showed relatively similar characteristics which indicated the effectiveness of the two three-component surfactants for use in preparation of poly(DVB)polyHIPE foam. Moreover, it was also demonstrated that the usage of Soxhlet extraction technique for poly(DVB)polyHIPE foam improved surface area of the obtained materials by 107% as compared with the unextracted polyHIPE porous foam. The optimum Soxhlet extraction time to achieve the highest surface area with the best mechanical properties for S20M\_T systems was around 6 h whereas S80M\_T system, composed of span80 as non-ionic surfactant that had longer alkyl chain length in the structure, needed around 12–24 h to remove nearly all the residue materials from the obtained polyHIPE porous structure. However, polyHIPE foam without any reinforcement phase also exhibited poor mechanical properties i.e. low crush strength and brittleness. Thus, to further improve the overall properties of polyHIPE porous foam, poly(DVB)polyHIPE filled with organoclay was prepared. Three types of organoclay including hybrid organic-inorganic porous clay heterostructure (HPCH), organo-modified bentonite (MOD) and acid treated organo-modified bentonite (AC-MOD) were used as inorganic reinforcement for polyHIPE foam. The effect of different type of organoclay on physical properties and CO<sub>2</sub> adsorption capacity of poly(DVB)polyHIPE nanocomposites foam was investigated. In all system, the addition of organoclay into polyHIPE matrix resulted in the improvement of the overall properties of the resulting polyHIPE foam. The surface area and the decomposition temperatures ( $T_d$ ) for the series of poly(DVB)polyHIPE filled with organoclay increased with increasing the clay content from 0 to 10 wt% whereas the maximum improvement for mechanical properties was observed at 5 wt% organoclay. The adsorption of CO<sub>2</sub> gas by poly(DVB)polyHIPE foam filled with organoclay was found to increase as well when compared with neat poly(DVB)polyHIPE foam. Additionally, it has been demonstrated in this study that the CO<sub>2</sub> gas adsorption of poly(DVB)polyHIPE nanocomposites foam was increased in the following order: neat poly(DVB)polyHIPE foam < poly(DVB)polyHIPE foam filled with MOD  $\leq$  poly(DVB)polyHIPE foam filled with HPCH < poly(DVB)polyHIPE foam filled with AC-MOD.

## บทคัดย่อ

พรศรี เพศขางกูร : พอลิเมอร์โฟมรูพรุนสูงจากกระบวนการไฮอินเทอร์นอลเฟสอิมัลชันพอลิเมอร์ไรเซชัน (Novel Polymer Foam via Polymerized High Internal Phase Emulsion) อาจารย์ที่ปรึกษา: ศศ.ดร. มานิตย์ นิธิธนากุล รศ.ดร. รัตนาวรรณ มกรพันธุ์ และ ศศ.ดร. ปมทอง มาลากุล ณ อยุธยา 176 หน้า

พอลิเมอร์โฟมรูพรุนสูง หรือที่เรียกว่า พอลิฮิฟ (PolyHIPE porous foam) เป็นวัสดุพอลิเมอร์โฟมที่มีโครงสร้างรูพรุนสูง ซึ่งสามารถเตรียมได้จากกระบวนการไฮอินเทอร์นอลเฟสอิมัลชันพอลิเมอร์ไรเซชันที่เกิดขึ้นรอบๆ emulsion droplet (Polymerized High Internal Phase Emulsions) ทำให้ได้พอลิเมอร์โฟมที่มีอัตราส่วนของความพรุนสูงมากถึงระดับ 0.99 นอกจากนี้ยังมีการเชื่อมต่อระหว่างรูพรุนที่เกิดขึ้นทำให้โครงสร้างของพอลิเมอร์โฟมนี้เป็นแบบ 3 มิติ ซึ่งเหมาะสมกับการนำมาใช้ในหลากหลายอุตสาหกรรม อาทิเช่น วิศวกรรมเนื้อเยื่อ กระบวนการแยก กระบวนการเร่งปฏิกิริยา และการดูดซับและบำบัดมลพิษทางอากาศและในน้ำ วัตถุประสงค์หลักของโครงการวิจัยนี้มุ่งพัฒนากระบวนการผลิต และสังเคราะห์พอลิเมอร์โฟมจากกระบวนการไฮอินเทอร์นอลเฟสอิมัลชันพอลิเมอร์ไรเซชัน และโคไวนิลเบนซินมอนอเมอร์ให้มีประสิทธิภาพสูงขึ้น เพื่อให้เหมาะสมกับการนำมาใช้เป็นตัวดูดซับในการบำบัดมลพิษทางอากาศ อาทิเช่น การบอนด์ออกไซด์ (CO<sub>2</sub>) โดยทำการศึกษาผลของชนิด และอัตราส่วนของสารลดแรงตึงผิว เวลาที่ใช้ในการสกัดสารตกค้างในรูพรุน และการใช้แรดดินเหนียวเป็นสารเติมแต่ง พอลิเมอร์โฟมที่สังเคราะห์ได้ถูกนำมาขึ้นรูปและศึกษาสมบัติทางกายภาพ อาทิเช่น สมบัติเชิงกล สมบัติทางความร้อน และประสิทธิภาพในการดูดซับสารต่างๆ ผลการศึกษาพบว่าพอลิเมอร์โฟมรูพรุนสูงที่เตรียมจากสารลดแรงตึงผิวผสมสามชนิด และใช้เวลาในการสกัดสารตกค้างในรูพรุนประมาณ 6-12 ชม. มีประสิทธิภาพในการดูดซับสูงที่สุดเมื่อเปรียบเทียบกับอัตราส่วนอื่นๆ อย่างไรก็ตาม โครงสร้างพอลิเมอร์โฟมที่สังเคราะห์ได้ยังคงมีสมบัติเชิงกลที่ค่อนข้างต่ำ เพราะแตกหักได้ง่าย ดังนั้นจึงมีการใช้แรดดินเหนียวสามชนิดเป็นสารเสริมแต่งได้แก่ แรดดินเหนียวที่มีการดัดแปลงโครงสร้างให้มีรูพรุน แรดดินเหนียวที่ทำปฏิกิริยาแลกเปลี่ยนไอออนกับสารลดแรงตึงผิว และแรดดินเหนียวที่ผ่านการปรับปรุงคุณภาพโดยการนำไปต้มด้วยกรดไฮโดรคลอริก และทำปฏิกิริยาแลกเปลี่ยนไอออนกับสารลดแรงตึงผิว เพื่อเพิ่มสมบัติทางกายภาพให้แก่พอลิเมอร์โฟม ผลการศึกษาพบว่าด้วยวิธีดังกล่าว สามารถเตรียมพอลิเมอร์โฟมที่มีความคงทนแข็งแรงในระดับดีมาก อีกทั้งยังสามารถเพิ่มพื้นที่ผิวในการดูดซับ และสมบัติทางความร้อนได้อีกด้วย สำหรับประสิทธิภาพในการดูดซับก๊าซ

คาร์บอน ไดออกไซด์ของพอลิเมอร์โพรพอร์สูงที่ผสมสารเติมแต่งทั้งสามชนิดมีคุณภาพสูงขึ้นตามลำดับ พอลิเมอร์โพรพอร์ที่ไม่มีสารเติมแต่ง มีค่าการดูดซับน้อยกว่า แร่ดินเหนียวที่ทำปฏิกิริยาแลกเปลี่ยนไอออนกับสารลดแรงตึงผิว น้อยกว่า แร่ดินเหนียวที่มีการดัดแปลงโครงสร้างให้มีรูพรุน และน้อยกว่า แร่ดินเหนียวที่ผ่านการปรับปรุงคุณภาพแล้วโดยการนำไปต้มด้วยกรดไฮโดรคลอริก และทำปฏิกิริยาแลกเปลี่ยนไอออนกับสารลดแรงตึงผิวตามลำดับ

นอกจากนำไปใช้เป็นสารดูดซับแล้ว ในงานวิจัยนี้ยังสามารถนำพอลิเมอร์โพรพอร์สูงที่สังเคราะห์จากสไตรีน และเอทิลีน ไกคอลไดเมทาซิเลต มอนอเมอร์ไปประยุกต์ใช้ในวิศวกรรมเนื้อเยื่อ อีกทั้งยังศึกษาวิธีการปรับปรุงพื้นผิวพอลิเมอร์โพรพอร์ โดยใช้การดัดแปรพื้นผิวด้วยเทคนิคพลาสมาเพื่อให้เกิดหมู่ฟังก์ชันบนพื้นผิวซึ่งทำให้พื้นผิวมีความมีขรุขระมากขึ้น ทำให้สิ่งมีชีวิตสามารถยึดเกาะ และเจริญเติบโตได้ดีขึ้นบนพอลิเมอร์โพรพอร์ชนิดดังกล่าว

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## TABLE OF CONTENTS

	<b>PAGE</b>
Title Page	i
Abstract (in English)	iii
Abstract (in Thai)	v
Acknowledgements	vii
Table of Contents	viii
List of Tables	xi
List of Figures	xiii
Abbreviations	xix
List of Symbols	xx
 <b>CHAPTER</b>	
<b>I INTRODUCTION</b>	<b>1</b>
 <b>II THEORETICAL BACKGROUND AND LITERATURE REVIEW</b>	 <b>6</b>
2.1 Air Pollution	6
2.2 Adsorption Process and Adsorbent Materials	7
2.3 High Internal Phase Emulsion Polymer (PolyHIPEs)	10
2.4 Clay Mineral and Their Modified Products	15
2.5 Scaffold and Tissue Engineering	21
2.6 Plasma surface modification	25
2.7 MTT Assay	26
 <b>III EXPERIMENTAL</b>	 <b>27</b>
3.1 Materials	27
3.2 Methodology	29
3.3 Equipment	31



<b>CHAPTER</b>		<b>PAGE</b>
<b>IV</b>	<b>EFFECT OF SOXHLET EXTRACTION AND SURFACTANT SYSTEM ON MORPHOLOGY AND PROPERTIES OF POLY(DVB)POLYHIPE</b>	<b>39</b>
	4.1 Abstract	39
	4.2 Introduction	40
	4.3 Experimental	42
	4.4 Results and Discussion	44
	4.5 Conclusions	47
	4.6 Acknowledgements	48
	4.7 References	48
<b>V</b>	<b>POLYMERIC FOAM VIA POLYMERIZED HIGH INTERNAL PHASE EMULSION (HIPE) FILLED WITH ORGANO-MODIFIED BENTONITE</b>	<b>60</b>
	5.1 Abstract	60
	5.2 Introduction	61
	5.3 Experimental	63
	5.4 Results and Discussion	66
	5.5 Conclusions	70
	5.6 Acknowledgements	71
	5.7 References	71
<b>VI</b>	<b>HIGHLY POROUS MATERIALS VIA POLYMERIZED HIGH INTERNAL PHASE EMULSION FILLED WITH ORGANO-BENTONITE: SYNTHESIS, CHARACTERIZATION, AND CO<sub>2</sub> ADSORPTION</b>	<b>84</b>
	6.1 Abstract	84
	6.2 Introduction	85

<b>CHAPTER</b>	<b>PAGE</b>
6.3 Experimental	88
6.4 Results and Discussion	93
6.5 Conclusions	98
6.6 Acknowledgements	99
6.7 References	99
<b>VII SURFACE MODIFICATION OF HIGH INTERNAL PHASE EMULSION FOAM AS A SCAFFOLD FOR TISSUE ENGINEERING APPLICATIONS VIA ATMOSPHERIC PRESSURE PLASMA TREATMENT</b>	118
7.1 Abstract	118
7.2 Introduction	119
7.3 Experimental	122
7.4 Results and Discussion	128
7.5 Conclusions	133
7.6 Acknowledgements	134
7.7 References	134
<b>VIII CONCLUSIONS AND RECOMMENDATIONS</b>	150
<b>REFERENCES</b>	152
<b>APPENDIX</b>	160
<b>Appendix A</b> Experimental Data	160
<b>Appendix B</b> XRD Spectra	169
<b>Appendix C</b> TPD pulse titration curve	170
<b>CURRICULUM VITAE</b>	174

## LIST OF TABLES

<b>TABLE</b>		<b>PAGE</b>
<b>CHAPTER II</b>		
2.1	Typical characteristics of adsorption processes.	8
2.2	Characteristics of different adsorbents.	9
2.3	Chemical formula and characteristic parameter of commor used 2:1 Phyllosilicates.	16
<b>CHAPTER IV</b>		
4.1	Surface properties of poly(DVB)polyHIPE prepare with and without toluene as porogenic solvent.	58
4.2	Surface properties of poly(DVB)polyHIPE with various Soxhlet extraction times.	59
<b>CHAPTER V</b>		
5.1	Composition of polyHIPE with hybrid organic–inorganic porous clay heterostructures (HPCH).	81
5.2	Properties of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).	82
5.3	Elemental percentages of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).	83
<b>CHAPTER VI</b>		
6.1	Chemical compositions of clay i.e. untreated and treated with acid treatment. (Oct: cations in octahedral position: Al, Mg, Fe and Ti).	115

<b>TABLE</b>	<b>PAGE</b>
6.2 Surface areas and decomposition temperature ( $T_d$ ) of poly(DVB)polyHIPE filled with different type of organoclay i.e. HPCH, MOD and AC-MOD.	116
6.3 Mechanical properties of poly(DVB)polyHIPE filled with different type of organoclay i.e. HPCH, MOD and AC-MOD.	117
<b>CHAPTER VII</b>	
7.1 Physical properties of the poly(S/EGDMA)polyHIPE porous foam.	148
7.2 Elemental percentages of the poly(S/EGDMA)polyHIPE foam before and after atmospheric pressure plasma treatment.	149

## LIST OF FIGURES

FIGURE	PAGE
<b>CHAPTER II</b>	
2.1 The schematic of the formation of polyHIPE porous polymer foam.	4
2.2 Secondary pore structure in polyHIPE foams; a) polyHIPE without porogenic solvent and b) polyHIPE with porogenic solvent.	7
2.3 Structure of Montmorillonite.	11
<b>CHAPTER IV</b>	
4.1 Selected scanning electron micrographs of poly(DVB)polyHIPE; a) Span80 (single surfactant), b) S80M (three-component surfactant) and c) S20M (three-component surfactant).	50
4.2 Scanning electron micrographs of poly(DVB)polyHIPE; a-b S20M was used as surfactant (2a is the sample without toluene as porogenic solvent and 2b is the sample with toluene as porogenic solvent) whereas c-d S80M was used as surfactant (2c is the sample without toluene as porogenic solvent and 2d is the sample with toluene as porogenic solvent).	51
4.3 N <sub>2</sub> adsorption/desorption isotherm of poly(DVB)polyHIPE.	52
4.4 Scanning electron micrographs of poly(DVB)polyHIPE; a) S20M_T without Soxhlet extraction, b) S20M_T with Soxhlet extraction.	53

<b>FIGURE</b>	<b>PAGE</b>
4.5 TEM micrographs of poly(DVB)polyHIPE; a) without Soxhlet extraction, b) S20M_T with Soxhlet extraction 6 h, c) S80M_T with Soxhlet extraction 6 h.	54
4.6 Effect of Soxhlet time on surface area of Poly(DVB)PolyHIPE.	55
4.7 Effect of Soxhlet extraction time on compression stress of poly(DVB)polyHIPE.	56
4.8 Effect of Soxhlet extraction time on Young modulus of poly(DVB)polyHIPE.	57

## **CHAPTER V**

5.1 Scanning electron micrographs of poly(DVB)polyHIPE: (a–e) HIPE1–HIPE5 (1500×); (f–j) HIPE1–HIPE5 (15000×).	73
5.2 Scanning electron micrographs of poly(DVB)polyHIPE: (a–c) reference, HIPE1, and HIPE5, respectively (1500×); (d–f) reference, HIPE1, and HIPE5, respectively (15000×).	74
5.3 EDX spectra of poly(DVB)polyHIPE: a) with porous clay (HPCH) and b) without porous clay (HPCH).	75
5.4 Spectra Si mapping of scanning electron micrographs of poly(DVB)polyHIPE: a) 1 wt% HPCH; b) 3 wt% HPCH; c) 5 wt% HPCH; and d) 10 wt% HPCH.	76
5.5 Specific surface area and pore volume of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).	77
5.6 DTG curves of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).	78

<b>FIGURE</b>	<b>PAGE</b>
5.7 Compressive stress and percentage strain of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).	79
5.8 Young’s modulus of poly(DVB)polyHIPE with various weight percents of hybrid organic–inorganic porous clay heterostructures (HPCH).	80

## CHAPTER VI

6.1 FTIR spectra of (a) Bentonite (BTN), (b) Organo-modified clay (MOD), and (c) Acid treated organo-modified clay (AC-MOD).	102
6.2 XRD pattern of (a) Bentonite (BTN), (b) Organo-modified clay (MOD), and (c) Acid treated organo-modified clay (AC-MOD).	103
6.3 SEM micrographs of poly(DVB)polyHIPE nanocomposite foam filled with organoclay (1 wt%): a) without organoclay; b) HPCH; c) MOD; d) AC-MOD.	104
6.4 Scanning electron micrographs of poly(DVB)polyHIPE filled with various weight percentage of organoclay at high magnification ( $\times 15000$ ): a) reference (0 wt% organoclay), b) 1 wt% organoclay; c) 3 wt% organoclay; d) 5 wt% organoclay; and e) 10 wt% organoclay.	105
6.5 EDX spectra of poly(DVB)polyHIPE: a) Pure poly(DVB)polyHIPE foam and b) Poly(DVB)polyHIPE foam nanocomposite foam.	106
6.6 Typical example of $N_2$ adsorption-desorption isotherm for poly(DVB)polyHIPE nanocomposite foam.	107

<b>FIGURE</b>		<b>PAGE</b>
6.7	Surface area of poly(DVB)polyHIPE nanocomposite foam filled with three type of organoclay i.e. HPCH, MOD, and AC-MOD.	108
6.8	DSC thermograms of the poly(DVB)polyHIPE foam with and with out organoclay. Dashed line denotes the thermogram of neat poly(DVB)polyHIPE foam in the absence of organoclay and solid line denotes the thermogram of poly(DVB)polyHIPE nanocomposite foam.	109
6.9	Compressive stress of poly(DVB)polyHIPE nanocomposite foam filled with three type of organoclay i.e. HPCH, MOD, and AC-MOD. HAB/MSi-treated cotton fabrics using 1.5 mM HAB, 0.6 mM DBSA and 0.15 M NaCl	110
6.10	Young's modulus of poly(DVB)polyHIPE nanocomposite foam filled with three type of organoclay i.e. HPCH, MOD, and AC-MOD.	111
6.11	High-resolution TEM images of poly(DVB)polyHIPE nanocomposite foam.	112
6.12	Spectra Si mapping of scanning electron micrographs of poly(DVB)polyHIPE: a) 1 wt% organoclay; b) 3 wt% organoclay; c) 5 wt% organoclay; and d) 10 wt% organoclay.	113
6.13	CO <sub>2</sub> gas adsorption of poly(DVB)polyHIPE nanocomposite foam filled with three type of organoclay i.e., HPCH, MOD and AC-MOD.	114



<b>FIGURE</b>	<b>PAGE</b>
<b>CHAPTER VII</b>	
7.1 Schematic of DBD treatment of polyHIPE foam.	136
7.2 SEM images of poly(S/EGDMA)polyHIPE foam; a) with 1000× magnification and b) with 3500× magnification.	137
7.3 Effect of atmospheric pressure plasma treatment on the phase morphology of the poly(S/EGDMA)polyHIPE foam; a,b) untreated, c,d) treated 5 min, and e,f) treated 30 min.	138
7.4 Images of the poly(S/EGDMA)polyHIPE foam specimen a) without and b) with atmospheric pressure plasma treatment (30 min), and SEM images of the poly(S/EGDMA)polyHIPE foam with atmospheric pressure plasma treatment (30 min) at c) low magnification and d) high magnification.	139
7.5 FT-IR spectra of the poly(S/EGDMA)polyHIPE foam; a) untreated and b) treated with atmospheric pressure plasma treatment.	140
7.6 Static water sessile drops on the surface of the poly(S/EGDMA)polyHIPE foam; a) before (untreated) and b) after (treated) atmospheric pressure plasma treatment.	141
7.7 Cytotoxicity of the poly(S/EGDMA)polyHIPE foam before (untreated) and after (treated) atmospheric pressure plasma treatment.	142
7.8 L-929 mouse fibroblast cell adhesion on the poly(S/EGDMA)polyHIPE foam in treated and untreated with plasma technique condition. *Significant at $p < 0.05$ with respect to the untreated sample.	143

<b>FIGURE</b>	<b>PAGE</b>
7.9 Effect of treatment time on the L929 mouse fibroblast cell adhesion on the poly(S/EGDMA)polyHIPE foam in treated and untreated with plasma technique condition. *Significant at $p < 0.05$ with respect to the untreated sample.	144
7.10 L929 mouse fibroblast cell adhesion on the poly(S/EGDMA)polyHIPE foam in treated and untreated with plasma technique condition, as well as the TCPS as control after 1, 4, and 24 h of cell culture. *Significant at $p < 0.05$ with respect to the untreated sample.	145
7.11 L929 mouse fibroblast cell proliferation on the poly(S/EGDMA)polyHIPE foam in treated and untreated with atmospheric pressure plasma technique condition, as well as the TCPS as control after 4 h, and 1, 3 and 7 days of cell culture. *Significant at $p < 0.05$ with respect to the untreated sample.	146
7.12 SEM images of the L929 fibroblast-like cells on the poly(S/EGDMA)polyHIPE foam with glass, and treated and untreated with atmospheric pressure plasma at different times in culture.	147

**ABBREVIATIONS**

AC-MOD	Acid Treated Organo-modified Clay
ASTM	American Standard Testing Method
CaCl <sub>2</sub>	Calcium chloride
CTAB	Cetyltrimethylammonium bromide
DBD	Dielectric barrier discharge
DDBSS	Dodecylbenzenesulfonic acid, sodium salt
DRIFT	Diffuse Reflectance Infra-red Fourier Transform
DVB	Divinylbenzene monomer
EGDMA	Ethylene Glycol Dimethacrylate monomer
FTIR	Fourier Transform Infrared Spectrometer
HIPE	High Internal Phase Emulsion
HLB	Hydrophilic-Lipophilic Balance
HPCH	Hybrid Organic-inorganic Porous Clay Heterostructure
K <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	Potassium persulfate
MOD	Organo-modified Clay
PolyHIPE	Polymerized High Internal Phase Emulsion
SEM	Scanning Electron Microscope
S20	Sorbitant monolaurate (Span 20)
S80	Sorbitant monooleate (Span 80)
S	Styrene monomer
T	Toluene
TEM	Transmission Electron Microscope
THF	Tetrahydrofuran

**LIST OF SYMBOLS**

$A_{cs}$	the cross sectional area for N <sub>2</sub> at 77 K (-196°C) = 16.2 Å <sup>2</sup>
$C$	a constant that is related to the heat of adsorption
$M$	molecular weight of the adsorbate
$N$	Avogadro's number ( $6.023 \times 10^{23}$ molecules/mol)
$P/P_0$	relative pressure
$S$	total surface
$W$	weight of gas adsorbed at a relative pressure
$W_m$	weight of adsorbate constituting a monolayer of surface coverage