

การสังเคราะห์อนุภาคนาโนเล็กพิเศษที่มีการกระจายตัวแบบโมนอดิสเปอร์ส  
ของโคโพลิเมอร์สไตรีนและเมทิลเมทาคริเลต โดยคิสเปอร์ชันโพลิเมอไรเซชัน

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SYNTHESES OF SUPER-FINE MONODISPersed PARTICLES  
OF POLY(STYRENE-*CO*-METHYL METHACRYLATE)  
BY DISPERSION POLYMERIZATION

Miss Arunsri Anantaphiphat

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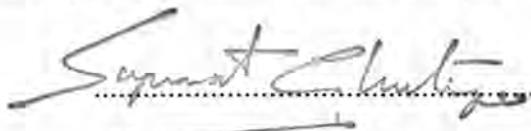
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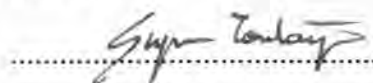
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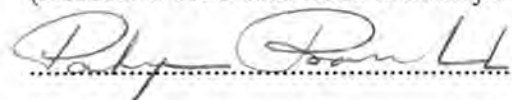
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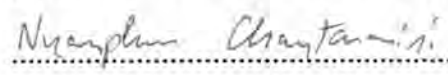
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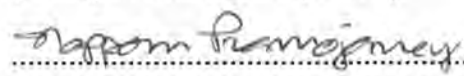
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อรุณศรี อนันตพิพัฒน์ : การสังเคราะห์อนุภาคขนาดเล็กพิเศษที่มีการกระจายตัวแบบโมนอดิสเปอร์สของ โคลิโพลิเมอร์สไตรีนและเมทิลเมทาคริเลต โดยวิธีคอปเปอร์ชัน โพลิเมอไรเซชัน (SYNTHESES OF SUPER-FINE MONODISPERSED PARTICLES OF POLY(STYRENE-CO-METHYL METHACRYLATE) BY DISPERSION POLYMERIZATION) อ.ที่ปรึกษา : รศ. ดร. สุภา เกียรติกำจรวงศ์ ; 278 หน้า. ISBN 974-637-228-9

โพลิ(สไตรีน-โค-เมทิลเมทาคริเลต) ที่มีขนาดอนุภาคระดับไมโครเมตร สามารถเตรียมได้โดยวิธีคอปเปอร์ชัน โพลิเมอไรเซชันในตัวทำละลายผสมระหว่างเอทานอลและน้ำ โพลิเมอร์เมทริกซ์และตัวริเริ่มปฏิกิริยาที่ใช้คือ โพลิไวนิลไพโรลิโดนและเอโซบิสไอโซบิวทิลโรโนไครด ตามลำดับ ได้ศึกษาอิทธิพลของความเข้มข้นของโพลิเมอร์เมทริกซ์ อัตราส่วนผสมระหว่างเอทานอลและน้ำ อุณหภูมิของการเกิดปฏิกิริยา เวลาที่ใช้ในการสังเคราะห์ อัตราการกวน และอัตราส่วนผสมของโพลิเมอร์สไตรีนและเมทิลเมทาคริเลต ที่มีผลต่อขนาดอนุภาค การกระจายขนาดอนุภาค และน้ำหนักโมเลกุลของโพลิเมอร์ที่ได้ นอกจากนี้ได้ศึกษาอิทธิพลของโพลิเมอร์เมทริกซ์โดยเตรียมโพลิเมอร์ที่ปราศจากโพลิเมอร์เมทริกซ์ ศึกษาการเปลี่ยนแปลงของโพลิเมอร์ในปฏิกิริยา และการเติบโตของอนุภาคโพลิเมอร์ภายในระยะเวลา 10 ชั่วโมงด้วย และได้ศึกษานาอนุภาคและการกระจายขนาดอนุภาคของโพลิเมอร์โดยเทคนิค SEM น้ำหนักโมเลกุลเฉลี่ยและการกระจายน้ำหนักโมเลกุลโดย GPC สมบัติทางความร้อนโดย DSC องค์ประกอบของโพลิเมอร์โดย NMR และ EA และสัณฐานของโพลิเมอร์โดย FTIR

การสังเคราะห์โพลิเมอร์ที่ใช้โพลิเมอร์เมทริกซ์ จะได้อนุภาคที่มีรูปร่างทรงกลม พื้นผิวเรียบ ขนาดอนุภาคอยู่ในช่วง 0.3-1.5  $\mu\text{m}$  และมีการกระจายขนาดแคบ ( $CV < 10\%$ ) ส่วนโพลิเมอร์ที่เตรียมโดยปราศจากโพลิเมอร์เมทริกซ์จะได้ลักษณะเป็นแผ่นแทน โพลิเมทิลเมทาคริเลตมีขนาดอนุภาคที่ใหญ่ที่สุด ส่วนโพลิสไตรีนมีขนาดอนุภาคเล็กที่สุด ขนาดอนุภาคของโพลิเมอร์ลดลงเมื่อเพิ่มความเข้มข้นของโพลิเมอร์เมทริกซ์ ความมีขี้ของตัวทำละลาย และอัตราการกวน อุณหภูมิที่เพิ่มขึ้นและระยะเวลาของการเกิดปฏิกิริยานานขึ้น ทำให้อนุภาคเฉลี่ยเพิ่มขึ้น การใช้น้ำและเอทานอลเป็นตัวทำละลาย อนุภาคมีการกระจายขนาดที่กว้าง แต่เมื่อใช้ตัวทำละลายผสมของเอทานอลและน้ำ พบว่าตัวทำละลายเอทานอลที่มีปริมาณน้ำผสมอยู่ในช่วง 10-30 ส่วน อนุภาคจะมีการกระจายขนาดที่แคบ อุณหภูมิที่เหมาะสมในการสังเคราะห์โพลิเมอร์ คือ 70°C สำหรับการกระจายน้ำหนักโมเลกุลของโพลิเมอร์ที่สังเคราะห์ได้ในช่วง 2-3 งานวิจัยนี้ได้อธิบายกลไกการเกิดโพลิเมอร์ขนาดเล็กและการกระจายตัวแคบด้วยทฤษฎีของการละลายและทฤษฎีการดูดซึมเมทริกซ์โพลิเมอร์

สาขาวิชาปิโตรเคมี-โพลิเมอร์  
ภาควิชา.....วิทยาศาสตร์โพลิเมอร์  
สาขาวิชา.....2540.....  
ปีการศึกษา.....

ลายมือชื่อนิติ.....  
ลายมือชื่ออาจารย์ที่ปรึกษา.....  
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม.....

# # C785237 POLYMER SCIENCE  
 # # MAJOR Dispersion polymerization, PVP, STY-co-MMA, Superfine, Particles  
 KEY WORD:

ARUNSRI ANANTAPHIPHAT: SYNTHESIS OF SUPER-FINE MONODISPersed PARTICLES OF POLY(STYRENE-co-METHYL METHACRYLATE) BY DISPERSION POLYMERIZATION.  
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Microsphere particle sizes of poly(styrene-co-methyl methacrylate) were prepared by dispersion polymerization in ethanol/water media. Poly(N-vinyl pyrrolidone) (PVP K-30) and azobisisobutyronitrile were utilized as matrix polymer and initiator, respectively. The effects of matrix polymer concentrations, ethanol/water ratio, reaction temperature, reaction time, agitation rate and feed ratio of styrene/methyl methacrylate monomers on the particle sizes, particle size distribution, average molecular weights and molecular weight distribution of the copolymer were investigated. The effect of the matrix polymer on particle sizes of the copolymer were studied. Monomer conversion and the copolymer particles growth within 10 h were also investigated. The particle sizes and particle size distribution were characterized by SEM; the average molecular weights and molecular weight distribution by GPC; the thermal analysis by DSC; the copolymer compositions by NMR; and EA; and the functional group by FTIR.

Poly(styrene-co-methyl methacrylate) with spherical shape, smooth surface, the size range of 0.3-1.5  $\mu\text{m}$  and narrow size distribution ( $\text{CV} < 10\%$ ) were obtained by dispersion polymerization in the presence of the matrix polymer. The copolymers by solution polymerization (without the matrix polymer) were formed in sheets and lumps. It was found that the biggest and smallest particles were obtained in the homopolymers of poly(methyl methacrylate) and polystyrene, respectively. The average particle size decreased with increasing the matrix polymer concentration, polarity of the dispersion medium, and agitation rate. The increase in the polymerization temperature and reaction time resulted in an increase in the average particle size. When water and ethanol were used as solvent, the copolymer particles have a broad size distribution. When the solvent mixture of ethanol and water was used, the particle size distribution was narrower in a mixture containing 10-30 parts of water. The appropriate temperature for dispersion polymerization was 70°C. The molecular weight distribution range of the copolymer was 2-3. This research has elucidated the mechanisms of super-fine, monodispersed copolymer formation by the theories of solubility parameter and matrix polymer anchoring.

สหสาขาวิชาปิโตรเคมี-โพลีเมอร์

ภาควิชา.....

ลายมือชื่อนิสิต..... *Arungsri Anantaphiphat*

สาขาวิชา.....  
 วิทยาศาสตร์โพลีเมอร์

ลายมือชื่ออาจารย์ที่ปรึกษา..... *Assoc. Prof. Suda Kiatkamjornwong*

ปีการศึกษา.....  
 2540

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## ABBREVIATIONS

STY	styrene
MMA	methyl methacrylate
AIBN	2,2'-azo-bis-isobutyronitrile
PVP	poly(N-vinyl pyrrolidone)
EtOH	ethanol
kg	kilogram
g	gram
ml	milliliter
$\mu$ l	microlitre
$m^3$	cubic metre
$cm^3$	cubic centimetre
b.p.	boiling point
m.p.	melting point
f.p.	freezing point
D	density
$^{\circ}C$	degrees Celcius
$\mu$ m	micrometre
k	Boltzmann's constant
T	absolute temperature
PSD	particle size distribution

MWD	molecular weight distribution
S.D.	standard deviation
CV	coefficient of variation
PDI	polydispersity index
F	copolymer composition
$\bar{d}_n$	number average diameter
$\bar{d}_w$	weight average diameter
wt	weight
$\bar{M}_n$	number average molecular weight
$\bar{M}_w$	weight average molecular weight
$T_g$	glass transition temperature
$T_d$	decomposition temperature
GPC	Gel permeation chromatography
SEM	Scanning electron microscopy
NMR	Nuclear magnetic resonance spectroscopy
DSC	Differential scanning calorimetry
FTIR	Fourier-transform infrared spectroscopy
EA	Elemental analysis
>	more than

<	less than
s	second
min	minute
h	hour
%	percent