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FACTORS INFLUENCING PARTICLE SIZE OF POLYAMIDE MICROCAPSULES  
SYNTHESIZED VIA INTERFACIAL POLYCONDENSATION

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พอลิเม ไมค ไมโครแคปซูลทมีขนาดระหวาง 1-100 ไมโครเมตร สามารถสงเคราะทได้โดย กระบวนการพอลิคอนเดนเซนแบอินเตอร์เฟเชียล ระหวาง ไดเอซิคคลอไรด์กับไดเอมีน การสงเคราะท นีกระทาโดยละลายไดเอซิคคลอไรด์ในสารทต้องการบรรจุอในไมโครแคปซูล ทมีลักษณะ เป็นน้ำมัน น้ำ สารละลายผสมกับน้ำ ทมีสารอิมัลซิไฟอิง เอเจนต์ละลายออยแลวนส่วนผสมน ไปบ้นให้เป็นอิมัลชันด้วยเครื่อง โฮโมจิไนเซอร์ทมีความเร็ว ใบพัดตงแต่ 2,000-10,000 รอบต่อนาที และใช้ระยยะเวลาบ้นตงแต่ 30-150 วันาที แลวนอิมัลชันทได้ผสมลงในน้ำ ทมีไดเอมีนและสารรับกรละลายออย ปฏิกริยาพอลิคอนเดน เซชันจะ เกิดขึ้นตรงผิวสัมผัสของหยดน้ำมันทแขวนลอยอในน้ำและชั้นสารละลายในน้ำ ททำให้เกิดเยื่อบางๆ ของพอลิเม ไมค เป็นเปลือกของ ไมโครแคปซูลทหุ้มสารทมีลักษณะ เป็นน้ำมันบรรจุอในใจกลางของแคปซูล

การสงเคราะทพอลิเม ไมค ไมโครแคปซูลนได้ทดลอง เลือก ซิบาโซอิลคลอไรด์, เทเรพทาโลอิล คลอไรด์, เฮกซะ เมทิลลีนไดเอมีน และพาราฟีนลีนไดเอมีน เป็นโมโนเมอร์ในการสงเคราะทเปลือก พอลิเม ไมค โดยทมีบิวทิลอะซิเตทหรือ ไดบิวทิลพทาเลตเป็นสารบรรจุอใน ไมโครแคปซูล สารอิมัลซิไฟอิง เอเจนต์เลือกใช้ 2 ชนิด คือ โซเดียมโคดิซิลเบนซินซัลโฟเนตและ พอลิออกซิเอทิลลนอลริลอีเทอร์เป็น สารอิมัลซิไฟเออร์ และใช้เกลือ โซเดียมของสไตรีนมาลิกเกนไฮโดรด์และพอลิไวนิลแอลกอฮอล์เป็นสาร โปรเทกททพอลลอยด์ เลือก โซเดียมไฮดรอกไซด์เป็นตัวรับกรทเกิดจากปฏิกริยาพอลิคอนเดนเซน

จากผลการทดลองพบว่า พอลิไวนิลแอลกอฮอล์มีประสิทธิภพในการ เป็นสารอิมัลซิไฟอิง เอเจนต์ มากที่สุด โดยทสามารถใช้ โซเดียมโคดิซิลเบนซินซัลโฟเนตเป็นตัวช่วยเสริมประสิทธิภพในการ เกิดอิมัลชัน ของการสงเคราะทไมโครแคปซูลทมีขนาดตางๆกัน โมโนเมอร์ทใช้ในการสงเคราะทพอลิเม ไมคสามารถ สลับคโมโนเมอร์ได้ทกตัว ทมีผลต่อสมบัติทางกายภพและ เคมีของ ไมโครแคปซูลทสงเคราะทได้ ความ เร็ว ใบพัดของเครื่อง โฮโมจิไนเซอร์ ระยยะเวลาที่บ้น ปริมาณของพอลิไวนิลแอลกอฮอล์ และปริมาณของ ไดเอซิคคลอไรด์มีผลต่อลักษณะของ ไมโครแคปซูลทได้ โดยทความเร็ว ใบพัดสงขนจะทาให้เส้นผ่าศูนย์กลาง เฉลี่ยของ ไมโครแคปซูลลดลง ระยยะเวลาที่ใช้บ้นนานมากขึ้นทาให้ขนาดของ ไมโครแคปซูลสมาเสมอมากขึ้น การเพิ่มปริมาณของพอลิไวนิลแอลกอฮอล์จะทาให้ไมโครแคปซูลอในสภาพอิมัลชัน ได้นานและมีผลทาให้เส้น ผ่าศูนย์กลาง เฉลี่ยของ ไมโครแคปซูลลดลง ปริมาณการใช้ซิบาโซอิลคลอไรด์มีผลโดยตรงต่อประสิทธิภพใน การหุ้มจุลภพ ซึ่งจะต้องปรับความเข้มข้นให้เหมาะสม เพื่อจะได้ไมโครแคปซูลทมีเปลือกหุ้มทสมบูรณ์

ภควิชา สนสาขาวิชาปิโตร-โพลีเมอร์  
สาขาวิชา วิทยาศาสตร์โพลีเมอร์  
ปีการศึกษา 2532

ลายมือชอนสิติ   
ลายมือชอนอาจารย์ทปรีชา   
ลายมือชอนคณาจารย์ที่ปรึกษา



พิมพ์ที่ศูนย์ฉบับมหาวิทยาลัยศรีนครินทรวิโรฒ กรุงเทพมหานคร

SUTHON CHUENCHOKESANT : FACTORS INFLUENCING PARTICLE SIZE OF POLYAMIDE MICROCAPSULES SYNTHESIZED VIA INTERFACIAL POLYCONDENSATION. THESIS ADVISOR : ASSOC. PROF. SUDA KIATKAMJORNWONG, Ph.D.; THESIS CO-ADVISOR : SUPON CHOTIWANA, Ph.D., 292 pp. ISBN 974-577-270-4

Synthesis of 1-100 micrometers of polyamide microcapsules by interfacial polycondensation between diacid chlorides and diamines was carried out. The synthesis was started by dissolving the diacid chloride in an oily-like material to be encapsulated in microcapsules and then mixing this solution in an aqueous solution of an emulsifier or a protective colloid. The mixture was agitated into emulsion by a high-speed homogenizer at the propeller speeds between 2,000-10,000 rpm., and the agitation times between 30-150 sec. This emulsion was poured into the aqueous solution of a diamine and an acid-receiver. Polycondensation occurred at the interface between the tiny oil droplets and another liquid layers and produced a thin film of polyamide as a shell of the microcapsules which encapsulated the oily-like material in the core.

Sebacoyl chloride and terephthaloyl chloride as diacid chlorides; and hexamethylene diamine and para-phenylene diamine as diamines were selected as monomer pairs to synthesize the polyamide microcapsule shell. Sodium dodecyl benzene sulfonate and poly(oxyethylene lauryl ether) as surface active agents; and sodium salt of styrene maleic anhydride and poly(vinyl alcohol) as protective colloids were selected to be emulsifying agents. Sodium hydroxide was used as an acid-receiver during polycondensation. Butyl acetate and dibutyl phthalate were used as encapsulated materials in microcapsules.

Based on the experiments, it was found that dibutyl phthalate was more appropriate as encapsulated material than butyl acetate; poly(vinyl alcohol) was the most effective emulsifying agent and sodium dodecyl benzene sulfonate could be used as a co-emulsifying agent with poly(vinyl alcohol) to produce microcapsules with various diameters. Furthermore, it was found that all diacid chloride and diamine combinations could be applied as monomer pairs to synthesize the polyamide microcapsule shell having a wide range of chemical and physical properties; the speed of the propeller, the agitation time of the homogenizer, and the amount of diacid chloride and emulsifying agent affected the characteristics of the microcapsules : increasing the speed of propeller decreased the average diameter of the microcapsules; longer agitation time produced more equal-sized microcapsules; increasing the concentration of emulsifying agent produced more stable emulsion which reduced the average diameter of the microcapsules; the amount of diacid chloride had a direct effect on encapsulation efficiency, the concentration of which must be adjusted properly so as to produce a perfect shell of the microcapsules.

ภาควิชา สหสาขาวิชา โคร-โพลีเมอร์  
สาขาวิชา วิทยาศาสตร์ โพลีเมอร์  
ปีการศึกษา 2532

ลายมือชื่อนิสิต .....  
ลายมือชื่ออาจารย์ที่ปรึกษา .....  
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม .....



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## LIST OF ABBREVIATIONS



ABBREVIATIONS	DEFINITION
gm	gram
ml	milliliter
rpm	revolution per minute
sec	second
°C	degree celcius
SBC	sebacoyl chloride
TPC	terephthaloyl chloride
HMDA	hexamethylene diamine
PNDA	para-phenylene diamine
BAT	butyl acetate
DBP	dibutyl phthalate
PVA	poly(vinyl alcohol)
SMA	sodium salt of styrene maleic anhydride copolymer
POLE	poly(oxyethylene lauryl ether)
SDBS	sodium dodecyl benzene sulfonate
NaOH	sodium hydroxide
DIW	deionized water
S	sebacoyl chloride for assigned name
T	terephthaloyl chloride for assigned name
B	butyl acetate for assigned name
D	dibutyl phthalate for assigned name
PV	poly(vinyl alcohol) for assigned name
SM	sodium salt of styrene maleic anhydride copolymer for assigned name
PO	poly(oxyethylene lauryl ether) for assigned name
SD	sodium dodecyl benzene sulfonate for assigned name
SDPxxx/yyy	S = sebacoyl chloride
(for section 2.9)	D = dibutyl phthalate
	P = poly(vinyl alcohol)
	xxx = value of rpm
	yyy = value of sec

ABBREVIATIONS	DEFINITION
TDPxxx/yyy (for section 2.9)	T = terephthaloyl chloride D = dibutyl phthalate P = poly(vinyl alcohol) xxx = value of rpm yyy = value of sec
SDPxxxE (for section 2.10)	S = sebacoyl chloride D = dibutyl phthalate P = poly(vinyl alcohol) xxx = value of poly(vinyl alcohol) concentration E = emulsifier
TDPxxxE (for section 2.10)	T = terephthaloyl chloride D = dibutyl phthalate P = poly(vinyl alcohol) xxx = value of poly(vinyl alcohol) concentration E = emulsifier
SDPxxx/yyy (for section 2.11)	S = sebacoyl chloride D = dibutyl phthalate P = poly(vinyl alcohol) xxx = amount of sebacoyl chloride ratio yyy = amount of dibutyl phthalate ratio
TDPxxx/yyy (for section 2.11)	T = terephthaloyl chloride D = dibutyl phthalate P = poly(vinyl alcohol) xxx = amount of terephthaloyl chloride ratio yyy = amount of dibutyl phthalate ratio
STC	sebacoyl chloride and terephthaloyl chloride combination
HMPN	hexamethylene diamine and para-phenylene diamine combination