

**NANOENCAPSULATION OF n-OCTADECANE USING  
RAFT MINIEMULSION POLYMERIZATION**

**by**

**Sidharth Sirohi**

**Department of Textile Technology**

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**SIDHARTH SIROHI**



**DEPARTMENT OF TEXTILE TECHNOLOGY  
INDIAN INSTITUTE OF TECHNOLOGY DELHI**

**JUNE 2015**

## CERTIFICATE

This is to certify that the thesis titled '*Nanoencapsulation of n-Octadecane using RAFT Miniemulsion Polymerization*' being submitted by **Mr. Sidhharth Sirohi** to the *Indian Institute of Technology Delhi*, for the award of degree *Doctor of Philosophy*, is a record of bonafide research work carried out by him. He has worked under our guidance and supervision and fulfilled the requirements for the submission of thesis which has attained the standard required for a Ph.D. Degree of this Institute. The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for the award of any degree or diploma.

**Dr. Ashwini K. Agrawal**

Professor

Department of Textile Technology  
Indian Institute of Technology Delhi

**Dr. Manjeet Jassal**

Professor

Department of Textile Technology  
Indian Institute of Technology Delhi

Date

New Delhi

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## Abstract

Miniemulsion polymerization technique has been used extensively for preparing small sized uniform particles of polymers, core-shell polymer particles, hybrid particles and encapsulation of liquid core. The technique has achieved limited success in the area of uniform core shell structure with liquid as core, as it requires matching of strict thermodynamic and kinetic factors. The interfacial tension, spreading coefficient, rate of exit and entry of monomer play crucial role in this process. The advent of control radical polymerization, NMP, ATRP and RAFT has made the encapsulation process easier. NMP and ATRP methods have limitations of temperature, limited choice of monomers, and contamination by catalyst etc. On the contrary, RAFT polymerization is free from such anomalies. Interfacial RAFT miniemulsion polymerization with appropriate RAFT agent enables the formation of uniform core shell structures and allows shell thickness of nanocapsules to increase uniformly. Miniemulsion process requires stabilizers and co-stabilizers for stabilization of droplets and these remain present in the system even after the polymerization and affect the final properties of latex. Reactive surfactants or polymeric surfactants based on oligomeric RAFT agents that can play dual role of stabilizer and RAFT initiator/agent are advantageous. The hydrophilicity of these surfactants is either tuned by increasing block length of hydrophobic part or changing monomers. Styrene-maleic anhydride based copolymers (amphiphilic copolymers) are found to be successful, and effect of pH and ammonolysis on their surface activity has been studied by researchers for obtaining core-shell structure. However, the formation of polymer particles was also observed

and a small amount of conventional surfactant was required to be added to suppress the formation of these polymer particles (i.e. suppression of homogenous nucleation).

The distribution of monomers in amphiphilic copolymers may also influence their surface activity, and therefore, stabilization power of miniemulsion. In order to examine the role played by microstructures of styrene-maleic anhydride (SM) co-oligomers in RAFT miniemulsion polymerization, four different co-oligomers of styrene-maleic anhydride (amphi-RAFTs/SM-RAFT) with varied concentration of styrene and maleic anhydride were prepared by RAFT polymerization using non rate retarding RAFT agent (phenylethyl phenyldithioacetate). The polymerization was conducted in controlled conditions with constant time (130 minutes) and conversions were kept < 50% to restrict the formation of homopolymer block as tails. The polymers were characterized for chemical composition, microstructure, molecular weight and thermal properties. The co-oligomers were found to consist of different microstructures. Critical micelle concentration and DLS results confirmed that self assembling behavior of the four co-oligomers was influenced by their composition and microstructure. SM-84 co-oligomer (prepared with 83.4 mol% of styrene in the feed) showed best self assembling behaviour, while co-oligomer with SM-50 (prepared with 50 mol% of styrene in the feed) showed very poor self assembling tendency.

Nanoencapsulation of n-octadecane (OD) (as phase change material, PCM) was investigated via RAFT miniemulsion polymerization of styrene using the four synthesized co-oligomers (SM-RAFT) as surfactants and RAFT agents, without using any additional co-surfactant. The surface activity of co-oligomers was further tuned by varying the extent of ammonolysis of maleic anhydride moieties. The

encapsulation process involved anchoring of surface active initiator radicals to the RAFT agent assembled at the oil /water interface. FE-SEM and TEM were used for investigating the morphology and encapsulation behavior of the prepared nanocapsules. Using SM-50 as a RAFT surfactant, only polymer particles were obtained. This was ascribed to the very low concentration of semi-alternating triads only 7% present in this co-oligomer which were not enough for keeping co-oligomer (SM-RAFT) anchored at oil/water interface in RAFT miniemulsion polymerization of styrene. Therefore, transfer of surfactant into aqueous phase resulted in formation of solid polystyrene particles. Among all the RAFT co-oligomers, SM-84 was observed to exhibit the best encapsulation efficiency (80%) at 1.5 g of ammonia concentration (i.e. ammonia to maleic anhydride mole ratio of 1.5). The presence of reasonably high concentration of semi-alternating triads (31%) in SM-84 helped in demonstrating better surface activity, which could be further, tuned by the ammonia concentration during the ammonolysis of maleic anhydride moieties. The nanocapsules (NC-84/1.5) formed using SM-84 were small (~97 nm), uniform in size and had core content of ~20%.

The composite PVA nanofibres loaded with NC-84/1.5 were electrospun to investigate the feasibility of loading of nanoencapsulated PCMs in PVA nanofibres. The composite nanofibres were hexane washed and characterized by FT-IR and differential scanning calorimetry (DSC) for establishing the presence of nanoencapsulated PCMs inside the nanofibres. The surface morphology was investigated by FE-SEM, S-TEM and HR-TEM. The best results in terms of efficiency of loading and dispersion of nanocapsules were obtained with PVA nanofibres loaded with 15 wt% of n-octadecane (on weight of PVA). The nanofibres

were relatively smooth and uniform with diameter of  $438 \pm 100$  nm (at the bulged regions) and  $292 \pm 49$  nm (at the smooth regions). DSC results showed that these fibres (coded as PVA/NC-84/1.5/15) had the maximum latent heat of fusion indicating that about 65% of the added NC-84/1.5 could be incorporated.

The present study shows that the above approach may be adopted to nanoencapsulate many hydrophobic materials suitable for various applications and successfully entrap them in other nanostructures such as nanofibre webs for easy handling and use.

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## List of Symbols and Abbreviations

PS	Polystyrene
LbL	Layer By Layer
SCK	Shell Cross-Linked Knedel
CRP	Control Radical Polymerization
ATRP	Atom Transfer Radical Polymerization
NMP	Nitroxide Mediated Polymerization
RAFT	Reversible Addition Fragmentation Chain Transfer
M	Monomer
$k_{\text{add}}, k_{-\beta}$	RAFT addition rate constants, $\text{L} \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$
$k_{-\text{add}}, k_{\beta}$	RAFT fragmentation rate constants, $\text{s}^{-1}$
$k_{\text{p}}$	Propagation rate constant, $\text{L} \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$
$k_{\text{d}}$	Rate constant of deactivation
$K_{\text{a}}$	Rate constant of activation
$k_{\text{tr}}$	Rate constant of chain transfer
t	Time
MMA	Methyl Methacrylate
VAc	Vinyl Acetate
AN	Acrylonitrile
MA	Methyl Acrylate
NVP	N-Vinyl Pyrrolidone
AM	Acrylamide
CTAB	Cetyl Trimethyl Ammonium Bromide

SDS	Sodium Dodecyl Sulphate
CDB	Cumyl Ditiobenzoate
PEPDA	Phenylethyl Phenylthioacetate
KPS	Potassium Per Sulphate
PPPTA	Phenyl 2-Propyl Phenylthioacetate
SM	Styrene- Maleic Anhydride co-oligomer
OD	Octadecane
PCM	Phase Change Material
Sty	Styrene
MAn	Maleic Anhydride
SM-50	Styrene- maleic anhydride co-oligomer with 50 mol% of styrene
SM-66	Styrene- maleic anhydride co-oligomer with 66 mol% of styrene
SM-75	Styrene- maleic anhydride co-oligomer with 75.4 mol% of styrene
SM-84	Styrene- maleic anhydride co-oligomer with 83.6 mol% of styrene
p-TSA	p-Toluene Sulphonic Acid
PDA	Phenyl Dithioacetic Acid
AIBN	Azobisisobutyronitrile
CMC	Critical Micelle Concentration
GPC	Gel Permeation Chromatography
FT-IR	Fourier Transform Infrared Spectroscopy
PDI	Poly Dispersity Index

DLS	Dynamic Light Scattering
NC	Nanocapsule
Thick.	Thickness
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	concentration
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NC-84/2.5	Nanocapsules prepared using SM-84 co-oligomer at 2.5 g NH <sub>3</sub> concentration
DSC	Differential Scanning Calorimetry
MDSC	Modulated Differential Scanning Calorimetry
T <sub>g</sub>	Glass Transition Temperature
TGA	Thermal Gravimetric Analysis
FE-SEM	Field-Emission Scanning Electron Microscopy
S-TEM	Scanning Transmission Electron Microscopy
TEM	Transmission Electron Microscopy
μm	Micrometer
nm	Nanometer
Wt%	Weight Percent Concentration
kV	Kilovolt
M <sub>w</sub>	Weight Average Molecular Weight
M <sub>n</sub>	Number Average Molecular Weight\