

**STUDY ON SOLUTION PROPERTIES OF HEXANOYL CHITOSAN  
AND EFFECT OF SOLVENT ON ITS BLEND FILMS AND  
ELECTROSPUN FIBERS**

Manisara Peesan

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**By:** Manisara Peesan

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**Thesis Advisors:** Assist. Prof. Ratana Rujiravanit

Prof. John Blackwell

Assist. Prof. Pitt Supaphol

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Accepted by the Petroleum and Petrochemical College, Chulalongkorn  
University, in partial fulfilment of the requirements for the Degree of Doctor of  
Philosophy.

*Nantaya Yanumet*  
..... College Director  
(Assoc. Prof. Nantaya Yanumet)

**Thesis Committee:**

*Nantaya Yanumet*  
.....  
(Assoc. Prof. Nantaya Yanumet)

*J. Blackwell*  
.....  
(Prof. John Blackwell)

*Ratana Rujiravanit*  
.....  
(Assist. Prof. Ratana Rujiravanit)

*Pitt Supaphol*  
.....  
(Assist. Prof. Pitt Supaphol)

*Anuvat Sirivat*  
.....  
(Assoc. Prof. Anuvat Sirivat)

*Jatuphorn*  
.....  
(Assist. Prof. Jatuphorn Wootthikanokkhan)

## ABSTRACT

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To obtain the organic solvent-soluble chitosan derivative, hexanoyl chitosan (H-chitosan) was synthesized by repeatedly reacting chitosan with hexanoyl chloride in the mixture of anhydrous pyridine and chloroform. H-chitosan was dissolved in selective organic solvents such as chloroform, dichloromethane and tetrahydrofuran in order to study the effect of solvent type on hexanoyl chitosan solution properties. The solution properties of hexanoyl chitosan were determined by dilute solution viscometry, dynamic light scattering, and surface tension technique. In addition, blend of hexanoyl chitosan (H-chitosan) and polylactide (PLA) were fabricated to two forms, films and nanofibers, by solution-casting and electrospinning, respectively. In case of blend films, the effects of blend composition and casting solvent (chloroform, dichloromethane, or tetrahydrofuran) on miscibility, morphology, thermal properties, and mechanical properties were investigated. In case of electrospun fibers, the as-spun fibers of H-chitosan appeared to be flat with ribbon-like morphology, while the as-spun PLA fibers appeared to be circular in cross-section with a regular pore structure on their surface. Morphology of H-chitosan/PLA blend fibers was varied depending on solvent type and blend ratio.

## บทคัดย่อ

มณิสรา ปีสาร : การศึกษาสมบัติของสารละลายเฮกซะโนอิลโคโตซานและผลของตัวทำละลายต่อแผ่นฟิล์มและเส้นใยผสมของเฮกซะโนอิลโคโตซาน (Study on Solution Properties of Hexanoyl Chitosan and Effect of Solvent on Its Blend Films and Electrospun Fibers) อ. ที่ปรึกษา : ผศ. ดร. รัตนา รุจิรวนิช ผศ. ดร. พิชญ์ ศุภผล และศ. จอห์น แบลคเวลล์ 173 หน้า ISBN 974-965-180-4

งานวิจัยนี้ศึกษาการเตรียมเฮกซะโนอิลโคโตซาน โดยการทำปฏิกิริยาของโคโตซานกับเฮกซะโนอิลคลอไรด์ จากการศึกษาพบว่าเฮกซะโนอิลโคโตซานที่เตรียมได้สามารถละลายได้ดีในตัวทำละลายอินทรีย์ เช่นคลอโรฟอร์ม ไคคลอโรมีเทนและเตตราไฮโดรฟูแรน เนื่องจากสมบัติในด้านการละลายในตัวทำละลายอินทรีย์ของเฮกซะโนอิลโคโตซาน ทำให้มีความน่าสนใจในการศึกษาสมบัติของสารละลายเฮกซะโนอิลโคโตซานในตัวทำละลายอินทรีย์ต่างๆ เช่นคลอโรฟอร์ม ไคคลอโรมีเทนและเตตราไฮโดรฟูแรน โดยใช้เทคนิคต่างๆ ในการวิเคราะห์คือการวัดความหนืดของสารละลายเจือจาง (Dilute Solution Viscometry) การวัดการกระเจิงของแสงแบบไดนามิกส์ (Dynamic Light Scattering) และการวัดแรงตึงผิวของสารละลาย (Surface Tension) นอกจากนี้ในงานวิจัยนี้ยังได้ศึกษาพอลิเมอร์ผสมของเฮกซะโนอิลโคโตซานกับพอลิแลคไทด์โดยการขึ้นรูปสองแบบคือ การขึ้นรูปเป็นแผ่นฟิล์ม โดยวิธีเทสารละลายลงในแม่แบบ (Solvent Casting) และการขึ้นรูปเป็นเส้นใยระดับนาโนเมตรโดยวิธีการปั่นด้วยไฟฟ้าสถิต (Electrospinning) โดยมีวัตถุประสงค์ในการศึกษาผลของปริมาณเฮกซะโนอิลโคโตซานและชนิดของตัวทำละลายที่มีต่อสมบัติต่างๆของแผ่นฟิล์มและเส้นใยระดับนาโนเมตรของพอลิเมอร์ผสมระหว่างเฮกซะโนอิลโคโตซานกับพอลิแลคไทด์ ที่เตรียมได้จากการละลายในคลอโรฟอร์ม ไคคลอโรมีเทนหรือเตตราไฮโดรฟูแรน โดยการเปรียบเทียบสมบัติทางความร้อน, สมบัติทางกายภาพ โครงสร้างทางเคมี โครงสร้างทางจุลภาค โครงสร้างทางผลึก และสมบัติทางกล

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## ABBREVIATIONS

$\mu\text{l}$	Microliter
$\mu\text{m}$	Micrometer
$^1\text{H-NMR}$	Proton Nuclear Magnetic Resonance Spectroscopy
Al	Aluminium
$\text{C}_6\text{H}_5\text{CH}_3$	Toluene
$\text{C}_6\text{H}_6$	Benzene
$\text{CDCl}_3$	Deuterium Chloroform
$\text{CH}_2\text{Cl}_2$	Dichloromethane
$\text{CH}_3(\text{CH}_2)_4\text{COCl}$	Hexanoyl Chloride
$\text{CH}_3\text{COOH}$	Acetic Acid
$\text{CH}_3\text{COONa}$	Sodium Acetate
$\text{CHCl}_3$	Chloroform
cm	Centimeter
cP	Centipoise
$\text{CS}_2$	Carbon Disulfide
DC	Direct Current
DD	Degree of Deacetylation
deg	Degree
dl	Deciliter
DMAc	Dimethylacetamide
DMF	<i>N,N</i> -dimethylformamide
DMSO	Dimethylsiloxane
DS	Degree of Substitution
DSC	Differential Scanning Calorimetry
FT-IR	Fourier Transform Infrared Spectroscopy
g	Gram
h	Hour
H-chitosan	Hexanoyl Chitosan
HCl	Hydrochloric Acid

He	Helium
J	Joule
KBr	Potassium Bromide
kV	Kilovolt
LiCl	Lithium Chloride
M	Molar
mg	Milligram
MHz	Megahertz
min	Minute
ml	Milliliter
mm	Millimeter
mmol	Millimole
mN	Millinewton
N	Newton
NaBH <sub>4</sub>	Sodium Borohydride
NaOH	Sodium Hydroxide
nm	Nanometer
NR	Natural Rubber
P(DLLA-GA)	Poly(DL-lactide-co-glycolide)
PEO	Poly(ethylene oxide)
PEVA	Poly(ethylene-co-vinyl acetate)
PLA	Polylactide
PLLA	Poly(L-lactide)
PMMA	Poly(methyl methacrylate)
PS	Polystyrene
PTFE	Polytetrafluoroethylene
PVA	Poly(vinyl acetate)
PVME	Poly(vinyl methyl ether)
PVOH	Poly(vinyl alcohol)
sec	Second
SEM	Scanning Electron Microscope
TGA	Thermal Gravimetric Analysis

THF	Tetrahydrofuran
TMS	Tetramethylsilane
w/v	Weight by Volume
w/w	Weight by Weight
WAXD	Wide-angle X-ray Diffraction
XRD	X-ray Diffraction Spectroscopy
Zn-Se	Zinc Selenide

## List of Symbols

$\overline{M}_v$	Viscosity-average Molecular Weight
$\theta$	Scattering Angle of Incident Light
$\lambda$	Wavelength of Incident Light
$\beta$	Coherence factor
$\delta$	Hildebrand Solubility Parameter
$\Gamma(q)$	Decay Rate
$\zeta_l$	Hydrodynamic Friction Coefficient
$\chi_c$	Apparent Degree of Crystallinity
$\Delta H_f$	Area under the Melting Endotherm
$\Delta H_f^0$	Equilibrium Enthalpy of Fusion for PLA
$\eta_o$	Solvent Viscosity
$\eta_{red}$	Reduced Viscosity
$\eta_{rel}$	Relative Viscosity
$\eta_{sp}$	Specific Viscosity
$v_{sp}$	Reciprocal of Density of Polymer
$[\eta]$	Intrinsic Viscosity
$a$	Mark-Houwink Exponent
$A_2$	Second Virial Coefficient
$C$	Concentration in g/100 ml
$D_{cm}$	Diffusion Coefficient of the Particle's Center of Mass
$D_o$	Diffusion Coefficient at Infinite Dilution
$I(\tau, \theta)$	Scattering Intensity
$K$	Mark-Houwink Constant
$k_B$	Boltzmann Constant
$k_D$	First-order Concentration Coefficient
$M$	Molecular Weight of Polymer
$n_o$	Refractive Index
$^{\circ}C$	Degree Celcius

$q$	Wavevector
$R_H$	Hydrodynamic Radius
$T$	Absolute Temperature
$t$	Running Time of Sample Solution
$T_b$	Boiling Point Temperature
$T_d$	Thermal Decomposition Temperature
$T_g$	Glass Transition Temperature
$T_{mh}$	High-temperature Melting Endotherm
$T_{ml}$	Low-temperature Melting Endotherm
$t_s$	Running Time of Solvent
$w_{PLA}$	Weight Fraction of PLA in the Sample
wt.%	Percent by Weight