# PREPARATION AND CHARACTERIZATION OF CITRATE-CROSSLINKED CHITOSAN FIBER

หอสมุดกลาง สถาบันวิทยบริการ จุฬาลงกรณ์มหาวิทยาลัย

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A Thesis Submitted in Partial Fulfilment of the Requirements
for the Degree of Master of Science
The Petroleum and Petrochemical College, Chulalongkorn University
in Academic Partnership with
The University of Michigan, The University of Oklahoma,
and Case Western Reserve University
2003

ISBN 974-17-2347-4

**Thesis Title:** Preparation and Characterization of Citrate-Crosslinked Chitosan Fiber

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**Program:** Polymer Science

**Thesis Advisors:** Asst. Prof. Ratana Rujiravanit
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# บทคัดย่อ

วรรณะศิริ วรกิจกาญจนกุล: การเตรียมและทดสอบสมบัติของเส้นใยไคโตแซนที่เชื่อมโยงโมเลกุลด้วยซิเตรต (Preparation and Characterization of Citrate-Crosslinked Chitosan Fiber) อ. ที่ปรึกษา: ผศ.ดร. รัตนา รุจิรวนิช, รศ.ดร. นันทยา ยานุเมศ และ ศ.ดร. เซอิจิ โทคูระ 108 หน้า ISBN 974-17-2347-4

ไคโตแซนซึ่งมีระดับการเปลี่ยนหมู่อะซิติลร้อยละ 94.41 และน้ำหนักโมเลกุลเท่ากับ 4.25 x $10^5$ กรัมต่อโมล ถูกผลิตเป็นเส้นใยด้วยกระบวนการปั่นเส้นใยแบบเปียก โดยการอัดสารละลายไคโตแซนเข้มข้นร้อยละ 7 โดยน้ำหนักในสารละลายกรดอะซิติกเข้มข้นร้อยละ 4 โดยปริมาตร ผ่านหัวรีดซึ่งประกอบด้วยรูฉีดเส้นใยจำนวน 50 รู แต่ละรูมีขนาดเส้นผ่านศูนย์กลาง 0.1 มิลลิเมตร จากการศึกษาพบว่า อ่างน้ำยาเคมีซึ่งประกอบด้วยสารละลายโซเดียมไฮดรอกไซด์ให้เส้นใยซึ่งมีความแข็งแรงในรูปปมและการยืดตัว ณ จุดขาดสูงกว่าอ่างน้ำยาเคมีซึ่งประกอบด้วยสารละลายอิ่มตัวของแคลเซียมในสารละลายเมทานอลเข้มข้นร้อยละ 50 ในขณะที่ไม่มีความแตกต่างที่เห็นได้ชัดเจนระหว่างค่าความทนแรงดึงของเส้นใยที่ได้จากอ่างน้ำยาเคมีทั้งสองประเภท เส้นใยไคโตแซนที่เชื่อมโยงโมเลกุลถูกเตรียมขึ้นโดยการเติมกรดซิตริกลงในสารละลายไคโตแซนหรือเติมลงในอ่างน้ำยาเคมีในระบบโซเดียมไฮดรอกไซด์ จากผลการทดลองพบว่าการเติมกรดซิตริกช่วยปรับปรุงสมบัติความทนแรงดึงของเส้นใยในทั้งสองกรณี ในขณะที่ไม่สามารถปรับปรุงความแข็งแรงในรูปปม นอกจากนี้ยังพบการเพิ่มขึ้นของระยะยืด ณ จุดขาด โดยเฉพาะอย่างยิ่งโดยการเติมกรดซิตริกลงในสารละลายที่ใช้ผลิตเส้นใย การตรวจสอบเส้นใยโดยใช้กล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราดแสดงให้เห็นว่า การใช้กรดซิตริกปริมาณสูงทำให้ได้เส้นใยไคโตแซนที่มีพื้นผิวขรุขระในทั้งสองกรณี ในขณะที่เส้นใยไคโตแซนสูตรอื่นๆที่เตรียมขึ้นในการทดลองเดียวกันนี้มีผิวที่ค่อนข้างเรียบ นอกจากการใช้กรดซิตริกแล้ว ยังได้ทำการศึกษาเพิ่มเติม โดยการใช้กลูตาราลดีไฮด์เติมลงในสารละลายไคโตแซนที่จะผลิตเป็นเส้นใยแทนการใช้กรดซิตริก ซึ่งจากการทดลอง พบว่าการเชื่อมโยงโมเลกุลของเส้นใยไคโตแซนโดยกลูตาราลดีไฮด์ ให้ผลการทดลองที่มีแนวโน้มคล้ายคลึงกับการใช้กรดซิตริก เว้นแต่ความเข้มข้นของกลูตาราลดีไฮด์ที่ใช้ต่ำกว่าในกรณีของกรดซิตริก

# ABSTRACT

4472027063 : POLYMER SCIENCE PROGRAM
Wannasiri Worakitkanchanakul: Preparation and Characterization of Citrate-Crosslinked Chitosan Fiber.
Thesis Advisors: Asst. Prof. Ratana Rujiravanit, Assoc. Prof. Nantaya Yanumet, and Prof. Seiichi Tokura, 108 pp. ISBN 974-17-2347-4

Keywords : Chitosan/ Fiber/ Wet spinning/ Calcium chloride/ Sodium hydroxide/ Citric acid/ Glutaraldehyde/ Crosslinked

Chitosan with a degree of deacetylation of 94.41% and molecular weight of 4.25 x $10^5$ was wet-spun through a 50-hole (0.1-mm diameter) spinneret using a solution of 7% (w/v) chitosan in 4% (v/v) aqueous acetic acid. A sodium hydroxide (NaOH) coagulating system gave higher values of knot strength and elongation at break than a calcium chloride saturated aqueous methanol system. However, the tensile strength of chitosan fibers from both systems was not considerably different. Crosslinked chitosan fibers were prepared by adding citric acid to either a chitosan dope or to the first coagulation bath using NaOH coagulating system. The addition of citric acid in both cases appeared to increase the tensile strength of the fiber, while knot strength was not improved. An increase in elongation at break was found, especially with the addition of citric acid to the spinning dope. Interestingly, SEM micrographs showed rough, instead of smooth, fiber surface for fibers produced using high citric acid content in both chitosan dope and the first coagulation bath. An additional experiment was performed whereby glutaraldehyde was used instead of citric acid in chitosan dope. The glutaraldehyde-crosslinked chitosan fiber obtained showed quite similar trends in mechanical properties as for the citrate-crosslinked fibers except that the concentration range of applied glutaraldehyde was lower.

# ACKNOWLEDGEMENTS

The author is grateful for the partial scholarship and partial funding of the thesis work provided by Postgraduate Education and Research Programs in Petroleum and Petrochemical Technology (PPT Consortium).

The author would like to express her most appreciation to her thesis advisors, Prof. Seiichi Tokura, Assoc. Prof. Nantaya Yanumet, and Asst. Prof. Ratana Rujiravanit, for all supports, kind guidance, helpful suggestions, encouragement, care, and understanding throughout this thesis work. Moreover, she is greatly indebted to Prof. Tokura together with Assoc. Prof. Hiroshi Tamura and their students from Kansai University for all valuable spinning apparatus, the demonstration of the first experimental run, and their kindness.

The author is appreciative to Assoc. Prof. Saowaroj Chuayjuljit and all instructors at the Department of Material Science, Faculty of Science, Chulalongkorn University, her previous academic institute, for not only giving her the basic knowledge in polymers and textiles, but also kindly allowing her to use the Lloyd universal testing machine in this thesis work, together with special thanks to Mr. Sompong Chutikulswadi for technical assistance and kindness. In addition, the author also would like to acknowledge Dr. Sanong Ekgasit and his student at the Department of Chemistry, Faculty of Science, Chulalongkorn University, where partial FTIR spectroscopic measurements were carried out.

Moreover, the author would like to thank Asst. Prof. Pitt Supaphol and Dr. Manit Nithitanakul for being her thesis committee.

Above all, the author is thankful to all instructors, staffs, Ph.D. and first year students, and friends at the Petroleum and Petrochemical College for the valuable knowledge, equipment, great experience, encouragement, helpful suggestions, and support.

Finally, the author is in a great debt of her family for financial support, love, care, and understanding.

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