

APPENDIX

Oral Presentation

At the

26th Congress on Science and Technology of Thailand

Queen Sirikit National Convention Center

Bangkok

18 – 20 October 2000

การผลิตเส้นใยไคโตซานโดยวิธีการปั่นแบบเปียกและการหาลักษณะเฉพาะ
WET SPINNING OF CHITOSAN FIBRES AND THEIR CHARACTERISATION

รุ่งทิwa ชิดทอง, โรเบิร์ต มอลลอย และ นิภาพันท์ มอลลอย
Rungtiwa Chidthong, Robert Molloy and Nipapan Molloy

Biomedical Polymers Research Unit, Department of Chemistry, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand; e-mail address : rungtiwach@hotmail.com

บทคัดย่อ : เส้นใยไคโตซานถูกเตรียมขึ้นโดยวิธีการปั่นแบบเปียกจากสารละลาย 3% โดยน้ำหนักของไคโตซานใน 1% โดยปริมาตรของสารละลายกรดอะซิติกและใช้ 5% สารละลายโซเดียมไฮดรอกไซด์เป็นตัวทำให้ตกตะกอน เส้นใยไคโตซานที่ได้ถูกล้างด้วยน้ำและทำให้แห้งโดยการอบ ภายใต้สภาวะสูญญากาศที่อุณหภูมิ 60 องศาเซลเซียสเป็นเวลา 4 ชั่วโมง คุณสมบัติของเส้นใยไคโตซานที่ได้ขึ้นกับความเข้มข้นของไคโตซาน ความเข้มข้นของอ่างตกตะกอนและสภาวะในการทำเส้นใยให้แห้งและทำการศึกษาสมบัติทางกายภาพและสมบัติเชิงกลของเส้นใยไคโตซาน พบว่า เส้นใยที่ได้มีผิวเรียบและมีเส้นผ่านศูนย์กลางสม่ำเสมอ โดยมีแรงเค้นและการยืดที่จุดขาดเป็น 77.8 เมกกะปาสกาลและ 39.2%ตามลำดับ

Abstract : Chitosan fibres were made by wet spinning from a 3% by weight chitosan in 1% by volume aqueous acetic acid solution and using 5% aqueous sodium hydroxide as coagulant. The fibres were washed by water and then dried at 60°C for 4 hours. The final fibre properties were affected by chitosan concentration, coagulant bath concentration and drying conditions and were studied in terms of their physical and mechanical properties. It was found that the fibres had smooth surfaces and regular diameters with stress and extension at break of 77.8 MPa and 39.2% respectively.

Experimental Procedure : The chitosan used was supplied by Seafresh Chitosan Co., Ltd. Its degree of deacetylation was determined by a titration method and also by nuclear magnetic resonance spectroscopy. The molecular weight was measured by dilute-solution viscometry. The viscosity of the chitosan solution was measured by a Haake Falling Ball Viscometer. After dissolving to give a 3% w/v chitosan solution in 1% acetic acid as spin dope, the solution was stirred for 4 hours and then degassed under vacuum. The spin dope was filtered and wet spun by a small-scale wet spinning apparatus (Bradford University Research Ltd., UK.) into 5% NaOH in 90 water : 10 ethanol as coagulating agent. The fibres were washed with water and dried under vacuum at 60°C for 4 hours. The tensile properties of the fibres were measured on an Universal Mechanical Testing Machine (Lloyds LRX+) using a cross-head speed of 20 mm/min and 40 mm as the initial gauge length. The chitosan fibres showed improved tensile strength after using epichlorohydrin as crosslinking agent and glycerol as plasticiser.

Results, Discussion and Conclusion : The spin dope used in this study contained 3% w/v chitosan in 1%v/v acetic acid. The degree of deacetylation of the chitosan from the titration method was 91.2% compared with 91.8% from the nuclear magnetic resonance method. The viscosity of the spin dope was 298.5 cSt. Dilute-solution viscometry was used to measure the viscosity-average molecular weight of the chitosan and gave the molecular weight of about

246,000. In the wet spinning process, the coagulation bath contained 5% NaOH in 90 water : 10 ethanol as dehydrating agent at room temperature. The fibres were washed with water and then dried at 60°C 4 hours under vacuum. The fibres obtained had a diameter of 0.24 mm with smooth and regular surfaces. The mechanical properties were shown as tensile strength. The resultant fibres had a stress at break of 77.8 MPa and an extension at break of 39.2%. The chitosan fibres were brittle, easy to break and had a low stress at break. However the strength could be improved by using epichlorohydrin as a crosslinking agent for the polymer chain. Glycerol was used as a plasticiser to increase the pliability of the fibres and make them softer.

- Reference :**
- (1) G. C. East and Y. Qin, *J. Appl. Polym. Sci.*, 50 : 1773-1779, 1993.
 - (2) G. C. East, J. McIntyre and Y. Qin, in: *Chitin and Chitosan*, G. Skjak-Braek, ed., Elsevier, New York, USA, 1989.
 - (3) J. Knaul et al., *J. Appl. Polym. Sci.*, 69: 1435-1444, 1998.

Keywords : chitosan fibres, wet spinning

VITA

Name Rungtiwa Chidthong

Date of Birth 9 November 1974

Academic Status High School Certificate holder from
Wattanothai Payap, Chiang Mai, 1993

B.Sc. (Chemistry) degree holder from
Chiang Mai University, Chiang Mai, 1997

Scholarship Awarded National Science and Technology Development
Agency (NSTDA) Foundation Scholarship, Thailand,
1998-1999