

Preparations of Chemical Cellulose from Ascidian Tunic and Effect of Spinning Conditions on the Properties of New Regenerated Cellulose Fiber

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Abstract

Chemical cellulose from an ascidian tunic is isolated by extraction, digestion and bleaching steps. The content of α -cellulose was above 98 wt%, and its DPw was about 918. A new regenerated cellulose fiber from the chemical cellulose obtained in this study was made using NMMO/water(87/13 wt%) as a solvent by dry jet-wet spinning. The effects of spinning speed and cellulose content of spinning dope on the properties were investigated.

1. Introduction

Cellulose, as the most abundant organic polymer, has served mankind for thousands of years as an indispensable material for clothing and housing. We have been using wood cellulose as a main component of paper, regenerated cellulosic fiber, and woody building materials.

Tunicate in Protochordata, a kind of sea animal, produces highly crystalline cellulose I, which is called tunicin. Also algal and bacterial cellulose consist of highly crystalline cellulose I. These celluloses form large microfibrils 15~30 nm in width, and electron diffractometry and lattice imaging techniques using TEM showed that these microfibrils are single crystals of cellulose I.

The cellulose from woody plants has been isolated by chemical digestion processes for more than a century, dissolving the lignin and the hemicelluloses via a combined chemical transformation and cleavage, and leaving the cellulose component as a solid in a somewhat degraded state. The whole process comprises a so-called digestion, removed most of the lignin as liginosulfonic acid (sulfite process), alkali or thiolignin (graft and sulfate process respectively), and subsequent bleaching steps for eliminating nearly all of the residual lignin.[1,2,3]

Purity and average chain length of the wood pulp obtained are controlled by the parameters of the digestion and bleaching steps, and have to be adapted to the

end-use intended. The degree of polymerization of cellulose, which is polydispersed, seems to vary with its source and method of isolation. Special purity requirements have to be met frequently for chemical celluloses (dissolving pulps), i.e. products employed for subsequent chemical conversion. Generally, there are many impurities such as hemicellulose, lignin, resin/fats and mineral in woody cellulose. Cellulose is versatile starting material for subsequent chemical conversion, aiming at the production of artificial cellulose-based thread and films as well as of a variety of stable soluble cellulose derivatives to be used in many areas of industry and domestic life. The higher content of α -cellulose is needed for chemical cellulose.[1,2,3]

In contrast to cotton all regenerated cellulosic fibres lose a certain portion of their tensile strength in the water swollen state. However, currently the viscose process produces the largest amounts of rayon staple and filament fibres. Apart from various advantages, such as the extensive variability of the process and of the fibre properties, the viscose process holds a number of disadvantages for the environment. For this reason, research activities are underway with the aim to further improve the viscose process, with regard to reducing environmental pollution in particular, and develop carbon disulphide (CS_2)-free cellulose shaping process. Although there were several patent applications prior to this date, intensive investigation of new solvent systems for cellulose and new ways of shaping cellulose without the use of carbon disulphide generally commenced in the 1970s. A comparison of the various new shaping processes shows that the amine oxide (N-methylmorpholine-N-oxide, NMMO) process has reached the highest level of development and is employed in one production plants. There have been many researches on the effect of spinning conditions for properties of regenerated cellulose fibers.[4]

There are a good deal of cultured ascidian in Korea. An ascidian tunic has been blamed for problems of costal environmental pollution when discharged into the sea after being used as a slices of raw fish. We isolated chemical cellulose from an ascidian by treating with acid, acetone, alkali and bleaching agent. The composition of the chemical cellulose prepared in this study was investigated. A new regenerated cellulose fiber was made from ascidian tunic cellulose using NMMO/water as a solvent by dry jet-wet spinning. The effect of spinning conditions on the properties of the new regenerated cellulose fiber was investigated.

2. Experimental

Materials : The ascidian tunic cellulose (α -cellulose content : 98%, DP_w : 918) was used. Reagent grade of acetone, sodium hydroxide, sulfuric acid and sodium

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hypochlorite were used without further purification. NMMO/water (87/13 wt%) was prepared from NMMO/water (5/50) (Aldrich) by drying process. An n-propylgalate (Aldrich) as antioxidant was used without further purification.

Preparation of spinning dope : Spinning dope was made by standing at 120°C for 30min after making solid-dope of cellulose-NMMO/water-antioxidant mixture. The dope concentration of cellulose was 6 wt% and antioxidant was 0.5 wt% (per cellulose weight) respectively.

Spinning of regenerated cellulose fiber : Regenerated cellulose fibers were made from spinning dope described above by dry jet-wet spinning using pressure type spinneret equipped with pressure gauge, temperature and winding controllers, and coagulation bath. The spinneret had 3 holes, the nozzle diameter was 0.2mm and the length/diameter was 2. The air gap was 10cm. The coagulation bath contains water of 30°C. The dope temperature was 110°C and winding speeds were in the range of 0 - 100 m/min.

3. Results and Discussion

Chemical cellulose from an ascidian tunic is isolated by extraction, digestion and bleaching steps. The composition of the chemical cellulose obtained in this study were investigated. The content of α -cellulose was above 98 wt%, and it's DPw was about 918. A new regenerated cellulose fiber from the chemical cellulose obtained in this study is made using NMMO/water as a solvent by dry jet-wet spinning. The effects of winding speed and cellulose content of dope on the properties were investigated. The winding speed was in the range of 0 - 100 m/min, and the cellulose content of spinning dope was 3 - 9 wt%. As the cellulose content of dope increased, initial modulus and denier of fiber increased. However, the tensile tenacity decreased. As the winding speed increased, the tensile tenacity and initial modulus of fiber increased and the denier of fiber decreased. The new regenerated cellulose fiber has the distinguishing feature of higher wet strength compared to the commercial regenerated cellulose fibers. These results are shown in Table 1.

Table 1. Effect of spinning conditions on the properties of regenerated cellulose

Sample	Cellulose content (%)	Winding speed (m/min)	Denier		Mechanical Properties					
					Initial modulus (g/d)		Tenacity (g/d)		Elongation at break(%)	
			dry	wet	dry	wet	dry	wet	dry	wet
C-3	3	60	2.23	2.10	102.14	83.62	3.46	3.23	5.44	5.40
C-6	6	60	3.10	3.27	106.95	78.00	2.56	2.36	4.96	5.76
C-9	9	60	3.87	3.73	112.41	83.03	2.51	2.43	4.70	5.02
C6-W0	6	0	41.66	36.65	33.27	5.95	0.87	1.09	11.21	47.16
C6-W20	6	20	6.89	6.85	89.15	78.80	2.59	2.72	6.81	9.02
C6-W40	6	40	5.10	5.04	106.35	77.82	2.66	2.59	5.20	6.35
C6-W60	6	60	3.10	3.27	109.95	78.00	2.56	2.36	4.96	5.76
C6-W80	6	80	2.94	3.04	111.80	96.02	2.75	2.89	5.38	4.88

4. References

1. K. P. Lim, S. P. Moon and M. G. Lee, *Polymer Science and Technology*, **8**, 5 (1997)
2. James P. Casey, "Pulp and Paper"
3. D. Klemm, B. Philipp, T. Heinze, U. Heinze, W. Wagenknecht, "Comprehensive Cellulose Chemistry"
4. Lenzinger Berichte, 1994, p.11-12