# Aging of cellulose solution

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## 1. Introduction

With an annual production of about the ten billion tons, cellulose, the principal structural polysaccharide of plant cell walls, is definitely the most abundant biopolymer. Cellulose is the isotactic polyacetale of cellobiose or the syndiotactic polyacetale of glucose, the monomers connected by β-1,4-glycosidic linkages. Cellulose is a semirigid chain polymer with high glass transition temperature. Further the hydroxyl groups make cellulose molecule highly polar, leading to inter- and intrahydrogen bonding. Cellulose is not a thermoplastic polymer, its processing requires either a homogeneous chemical modification (viscose process) or its dissolution in a true solvent. In fact, one issue is to replace the viscose process that is derivated cellulose structure. Viscose rayon process is not good for environment because chemical agents are occurred by process likely $CS_2$ ,strong acid and strong alkali. A lot of the attempts have been made to find out direct solvent systems of cellulose. Among them N-methylmorpholine N-oxide (NMMO) proved most versatile and commercially successful. The process of cellulose solution using N-methylmorpholine-N-oxide (NMMO) as a solvent is a new alternative method to the viscose process, as it is wasteless, environmentally friendly, and used in a closed production cycle. Several reports on other effective solvent systems for cellulose were also disclosed.[1-3]

Gagnaire et al.[4] have demonstrated that the cellulose/(NMMO-$H_2O$) system forms a true solution, i.e. it is homogeneous at the molecular level.

The aim of the present study will confirm the change of properties by aging of cellulose solution according to time. Characteristics were measured by Differential scanning calorimetry (DSC), thermogravimetric analyse (TGA), scanning electron microscope (SEM) and wide angle X-ray diffraction (WAXD).

## 2. Experiment

### 2.1 Materials

Pulp(DP=1200) was used as powder types after crushing with a mill and vacuum drying. N-methlymorpholine-N-oxide was used to 87%, in addition to 0.1 wt% propyl gallate (Aldrich Chemical) as an anti-oxidation to avoid and degradation during the cellulose dissolving process.

### 2.2 Preparations of cellulose solution

To prepare a cellulose solution, we mixed NMMO 87% solution, pulp and anti-oxidation in order. Films were made from 7 wt% solution of cellulose in NMMO containing 0.1 wt% PG. Cellulose was dissolved in NMMO using a mechanical stirrer for 2 hours.

### 2.3 Preparations of cellulose film

Cellulose solution was cast onto a glas plate at 90℃ with 0.3mm thickness. The glass plate was immersed immediately into 1L of deionized water at 30℃ and then the films formed was washed thoroughly. The obtained filmed was dried under a vacuum oven at room temperature and remaining cellulose solution kept in vacuum machine at 95℃. Film process was shown in Figure 1.

### 2.4 Analysis

To know thermal property, prepared films were measured through TA Instrument 2010, Du Pont, U.S.A. The samples were sealed in aluminum pans under dry nitrogen from 30 ℃ to 350℃ at the heating rate of 10℃/min. A Thermogravimetric Analyzer(TGA, TA Instrument 2050, Du Pont, U.S.A) was used to investigate the thermal stability of films. TGA experiment was performed at a scanning rate of 20℃/min from 30℃ to 500℃ under nitrogen atmosphere. Wide-angle X-ray diffractometer (WAXD) measurements were carried out, CuKα radiation at room temperature, by a D/MAX-2200H, Rigaku. Co, Japan. Morphology was determined from SEM, CM200, PHILIPS, Netherlands.

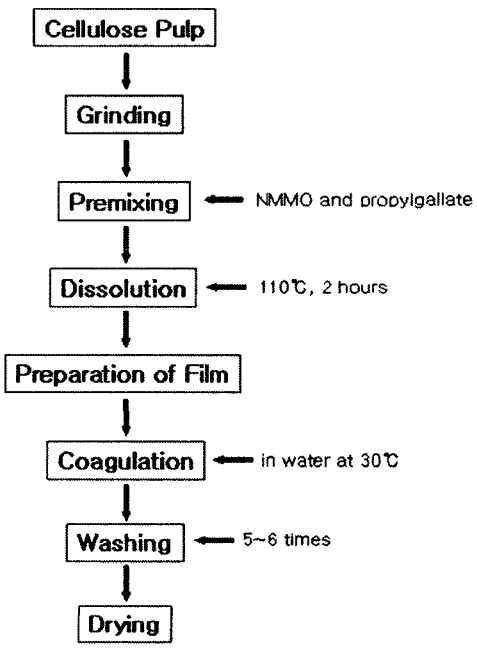


**Fig. 1 Preparation diagram of film preparation**

## 3. Results and Discussion

Thermal studies of the films obtained from cellulose solution with various aging time will be carried out by examining DSC and TGA thermograms of these films. We will confirm crystallinity and crystal size through WAXD. From SEM, we will observe morphology of cellulose films.

## 4. Reference

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