

CHAPTER 3

EXPERIMENTS

3.1 Reaction - media

The reactivity of sodium ions in etherification reaction of cellulose was studied by using various common organic solvents as reaction media. The solvents were chosen according to their characteristic natures as follows :

3.1.1 polar and non-polar properties

3.1.2 boiling point and rate of evaporation

3.1.3 extent of harmfulness on contact

They were also chosen with respect to availability in local market, economics and reusability.

In etherification, alcohols are frequently used as reaction media for most chemical transformation both in laboratories and in industrial processes. To search for more scientific information, this work was thus performed by using alcohols and other organic solvents including both polar and non-polar. Some typical reaction media used in this experiment were as follows :

<u>polar</u>	methyl ethyl ketone
	acetone
	iso-propyl alcohol
	n-propyl alcohol
	ethyl alcohol

non-polar benzene
toluene
cyclohexane
n-hexane

3.2 Experimental Procedure

Throughout this work, the experiments were carried out in the same manner but with various reaction media, different reaction times and at different temperatures. Treated cotton fuzz was used as source of cellulose. Juice mixer (blender) was used for pulverizer as recommended by K. Watanabe and M. Nakamura (22). Two sets of samples were prepared in this experiment. One set was allowed the etherification to proceed at $26 \pm 2^{\circ}\text{C}$ for 1, 3, 5 and 7 days. The other was conducted at higher temperature ($44 \pm 2^{\circ}\text{C}$) in an automatic incubator when temperature fluctuated within a narrow range.

In practice, a mole of cellulose was well mixed with each individual reaction medium as listed in 3.1. With continuous stirring, 2 moles of sodium hydroxide were added. The mixture was cooled by using ice-water if heat evolved during mixing. Then one mole of monochloroacetic acid was added and well mixed for about 15 minutes. The mixture was transferred to a beaker which was covered by thin plastic sheet and kept at $26 \pm 2^{\circ}\text{C}$ for 1, 3, 5 and 7 days. After each period, the product was sampled and filtered through the Buchner funnel and, then, soaked in methyl alcohol. At this point, the acidity or alkalinity of soaked solution (methyl alcohol) was inspected by using Bromothymol blue indicator.

If the solution was acid (yellow color), it was neutralized by sodium hydroxide solution. If the solution was basic (blue color), it was thus neutralized by hydrochloric acid. After obtaining neutral solution (green color), the mixture was filtered and washed for several times with methyl alcohol to remove sodium chloride obtained as reaction by-product. The product was, then, air dried and kept in desiccator.

Another set of samples was prepared in the same manner as mentioned above but the etherification was allowed to proceed at $44 \pm 2^{\circ}\text{C}$ in automatic controlled incubator (Baird & Tadlock Ltd).

3.3 Reactivity of Sodium Ions in the System

At present, direct- and indirect-methods are two available methods for determination of bound alkali involved in alkali treatment of cellulose and for determination the composition of alkali cellulose (6). The disadvantage of the two methods is the difficulty in performing the experiment and an understanding of the chemical implications of the structure of alkali cellulose.

Since our main purpose of this study is one concentrated to the etherification reaction of cellulose. Therefore, further etherification after alkali treatment of cellulose was carried out by using monochloroacetic acid. Neglect the mechanism from which alkali cellulose formed (either $\text{C}_6\text{H}_7\text{O}_2(\text{OH})_2\cdot\text{ONa}$ or $\text{C}_6\text{H}_7\text{O}_2(\text{OH})_2\cdot\text{OH}\cdot\text{NaOH}$), and of the errors due to experimental complication as previously mentioned, the degree of substitution (D.S.) of sodium carboxymethyl group ($-\text{CH}_2\text{COONa}$) in alkali cellulose at the end of the first day of each sample is used to predict

the extent of the reactivity of sodium ions or alkali in forming alkali cellulose. The D.S.'s of samples with the longer reaction times are also used to predict the inducing tendency or promotion for further etherification of cellulose in the system. In other words, D.S. is assumed to be a scale for justifying the reactivity of sodium ions in etherification reaction of cellulose.

In practice, about 0.5 g of product in nickel crucible of known weight was dried at 110°C by using vacuum dryer (Karl Kolb-Scientific Technical Supplies). After 5 hours, the nickel crucible and its cover were removed from dryer and kept in desiccator until they became cool. The crucible containing sample and cover were then weighed. The moisture content of the sample can be calculated by using equation (4).

The vacuum dried sample in nickel crucible was ignited with Bunsen burner until white solid appeared. The crucible and cover were carefully placed into a beaker containing hot water and were then boiled. After white solid was completely dissolved the crucible and its cover were removed from solution and well washed with distilled water. Add 10 cm^3 of $0.2\text{ M H}_2\text{SO}_4$ in the solution then it was evaporated to about 100 cm^3 . After cooling, the solution was titrated with standard NaOH (about 0.2M) and with phenolphthalein as indicator. The D.S. of the product can thus be calculated by using equation (6).