

CHAPTER 3

EXPERIMENTS

3.1 Purification of Cellulose

The starting materials used in this experiment was waste product obtained from local vegetable oil manufacture. The seed hull, kernel and other contaminants were separated by laboratory mill and by manual screening in order to obtain merely cotton fuzz as much as possible. At this stage, the cotton fuzz contained light to dark brown particles of ground seed hull attached to the short fiber.

In purifying process of cellulose, the several batches of cotton fuzz were first digested for 2 and 3 hours with various percent concentration of sodium hydroxide solution by an automatic temperature and pressure controlled autoclave.

Each batch of treated cotton fuzz called pulp was removed from dark-colored liquor and then rinsed with running-tab water for hours. The tiny pieces of seed hull must be totally removed. The pulp obtained was still light brown because of the partial absorption of the pigment from seed hull. Such pulp was thus bleached with about 0.1 weight-percent calcium hypochlorite for about 30 minutes. The

bleached pulp was washed with water and soaked with warm 0.1 percent sodium bisulfite. This treated pulp was thoroughly washed and air-dried. After drying, the treated pulp was first determined the content of α -cellulose following the improved method described by ASTM standard method (see 2.2). The process conditions used for cellulose purification were then obtained as shown in Table 1.

3.2 Preparation of Sodium Carboxymethylcellulose (Na-CMC)

The aim of this experiment is to increase D.S. of etherification as high as possible leading to the theoretical maximum D.S. of 3.0. A multistep of etherification is a technique commonly referred for the preparation of Na-CMC with high D.S.. Furthermore, a mixed system of reaction medium is also known as one of the process techniques in order to increase D.S. of the etherification product. Alcohol is frequently used to eliminate dough which always occurred in Na-CMC with high D.S.(16). Our intention is, at this stage, to follow such techniques with the use of cellulose source derived from the treatment of the waste product of our local vegetable oil manufacture.

According to a report of a separated work conducting in the same laboratory, D.S. of etherification product of cellulose obtained by using experimental procedure similar to one used in this work are 0.880, 0.707, 0.041 for 7 days of

Table 1 Percentage of α -cellulose obtained from various batches of treated pulp

Sample no.	% NaOH	Pressure (lbs./in ²)	Time (hrs.)	% α -cellulose (unbleached)	% α -cellulose (bleached)
1	1	10	2	69.35±0.74	67.23±0.72
2	3	10	2	73.52±0.78	68.85±0.74
3	5	10	2	69.98±0.73	67.07±0.71
4	8	10	2	65.61±0.70	64.15±0.68
5	10	10	2	64.15±0.68	60.99±0.65
6	1	20	2	71.99±0.67	67.61±0.72
7	3	20	2	78.04±0.84	75.17±0.80
8	5	20	2	70.15±0.75	68.23±0.73
9	8	20	2	67.72±0.72	66.58±0.71
10	10	20	2	65.03±0.69	64.60±0.69
11	1	10	3	67.12±0.72	63.24±0.67
12	3	10	3	71.60±0.76	64.61±0.70
13	5	10	3	70.09±0.75	63.83±0.68
14	8	10	3	65.63±0.70	63.29±0.67
15	10	10	3	63.42±0.68	60.91±0.65
16	1	20	3	67.55±0.72	64.62±0.69
17	3	20	3	71.28±0.79	69.07±0.74
18	5	20	3	70.09±0.75	67.71±0.72
19	8	20	3	67.52±0.72	66.42±0.71
20	10	20	3	65.01±0.69	63.02±0.67

etherification at room temperature and for methyl ethyl ketone, benzene and ethyl alcohol as reaction media respectively. It is, therefore, our decision to perform this experiment with 2 mixed system of reaction media:

- (1) Methyl ethyl ketone and ethyl alcohol
- (2) Benzene and ethyl alcohol

In practice, the etherification was conducted at room temperature for a certain period of time in each step of etherification by individual using of the above mentioned reaction medium. The experiment was performed as follows:

One mole of vacuum dried cellulose (obtained from 3.1) was well stirred with reaction medium in a laboratory blender. Two moles of sodium hydroxide was then added. After removal of heat involved during mixing, a mole of chloroacetic acid was added and stirred for about 5 minutes. The mixture was transformed into a beaker and covered with plastic sheet. It was then left for etherification at room temperature for 7 or 5 days.

After such desired period, the product was filtered and washed by soaking in methyl alcohol for several times. 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. Dried Na-CMC was divided into 2 parts; one for determining its D.S. whereas the other for performing second, third, fourth, fifth, etc. of etherification step.

The multistep of etherification was also followed the same procedure as described above except Na-CMC obtained at the end of each etherification step rather than cellulose obtained from 3.1 was used as starting material for etherification.

3.3 Determination of Degree of Substitution of Sodium Carboxymethylcellulose

In practice, moisture content of Na-CMC must be determined in order to obtain exact weight of Na-CMC used for determining its D.S.. The experiment was then performed as follows:

About m grams of Na-CMC containing in a known weight of nickel crucible (X) was vacuum dried at 110°C in vacuum dryer (Karl Kalb Scientific Technical Supplier) for 5 hours. It was then removed from the dryer and kept in desiccator at room temperature. After cooling, it was again weighed (n).

At this point, grams of sample used and mass loss on drying can be obtained:

$$\begin{array}{lcl} \text{Na-CMC used} & = & m \quad \text{gram} \\ \text{Na-CMC after drying} & = & n - X \quad \text{gram} \\ \text{mass loss on drying} & = & m - (n - X) \quad \text{gram} \end{array}$$

Therefore, the moisture content of Na-CMC can be calculated by using equation (1) in 2.3.2

$$\text{moisture (\%)} = \frac{m - (n - X)}{m} \cdot 100$$

After obtaining the moisture content of the sample, the D.S. of Na-CMC was determined by the following procedure:

About 0.5 gram of vacuum dried sample was ignited in nickel crucible by Bunsen Burner until white-solid residue was obtained after complete combustion. The residue was dissolved by careful dropping the crucible and its cover in 250 cm³ beaker containing hot water. The crucible and its cover were then removed from solution and well washed with distilled water. The solution was then boiled for about 30 minutes. About 10 cm³ of 0.2 M. H₂SO₄ was added in the solution and the solution was boiled until about 100 cm³ of solution remained. The solution was cooled and titrated with standard NaOH (about 0.2 M) and with phenolphthalein as indicator. The D.S. of the Na-CMC can thus be calculated by using equation (2) in 2.3.2