

CHAPTER 4

Experimental Work

4.1 General

The purpose of the experimental work described here was to study the rate of mercerization, effective diffusivity of sodium ion through alkali-cellulose, percent conversion of mercerization. Experimental data were obtained from reaction between cotton fibre and sodium hydroxide solution by varying temperature, concentration of sodium hydroxide solution, tension of cotton fibre and speed of stirrer so that effective diffusivity can be found in terms of temperature, concentration and tension of cotton fibre.

4.2 Experimental Apparatus

Fig. (4.1) shows the schematic fitting of laboratorial mercerizing apparatus including stirring system, temperature controllable heater, weight and stainless steel cylinder containing sodium hydroxide solution.

4.2.1 Stirring system

The cylindrical magnetic bar coated with teflon is 30 m.m. long and 10 m.m. in diameter which was used in circulating sodium hydroxide solution. The speed of this stirrer was controlled by a variable speed motor.

4.2.2 Temperature controllable heater

The temperature of sodium hydroxide solution was maintained at a certain temperature by a thermostat which has accuracy of $\pm 0.2^\circ\text{K}$.

4.2.3 Reaction vessel

Sodium hydroxide solution, sulfuric acid solution and distilled water were contained in the stainless steel cylinders which have dimensions of 200 mm. in height and 90 mm. in diameter.

4.2.4 Weight

Weights which were used for producing the tensile force on the mercerizing yarn were made of stainless steel. Their dimensions are 26 mm. in height and 16 mm. in diameter. They are 44.5 gm. in weight and 5.5 ml. in volume.

4.3 Preparation of Solution

4.3.1 Sodium hydroxide solution

Sodium hydroxide flakes of analytical reagent grade were weighted with the accuracy of ± 0.01 gm., dissolved in distilled water in a volumetric cylinder, cooled to the room temperature. The concentration of prepared solution were 4.80×10^{-3} , 5.92×10^{-3} , 7.96×10^{-3} , 1.06×10^{-2} and 1.27×10^{-2} gm-mole/ml.

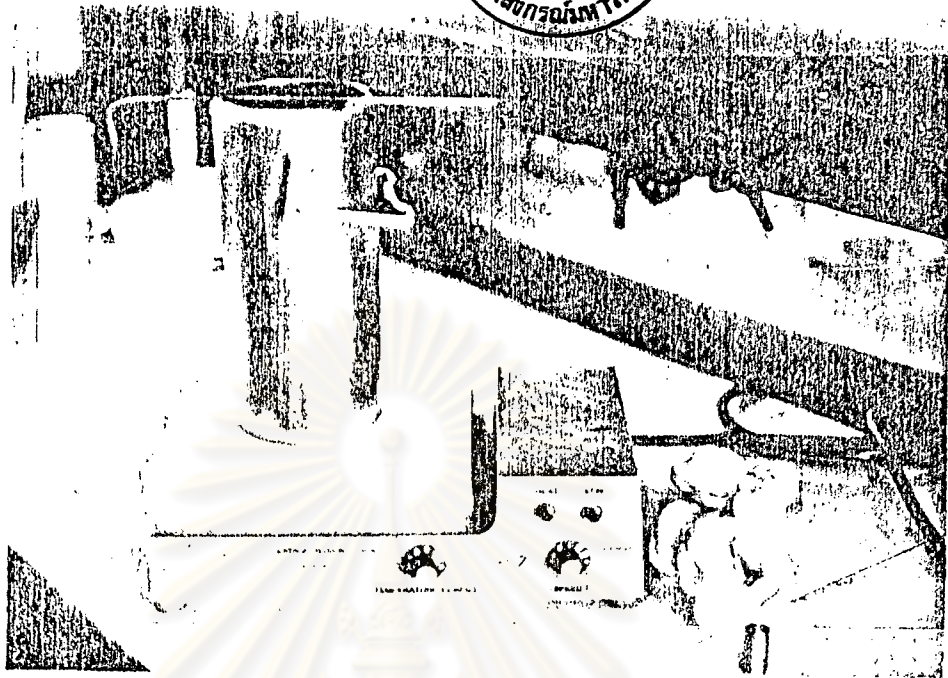


Fig 4.1 Laboratorial cotton yarn mercerizing apparatus

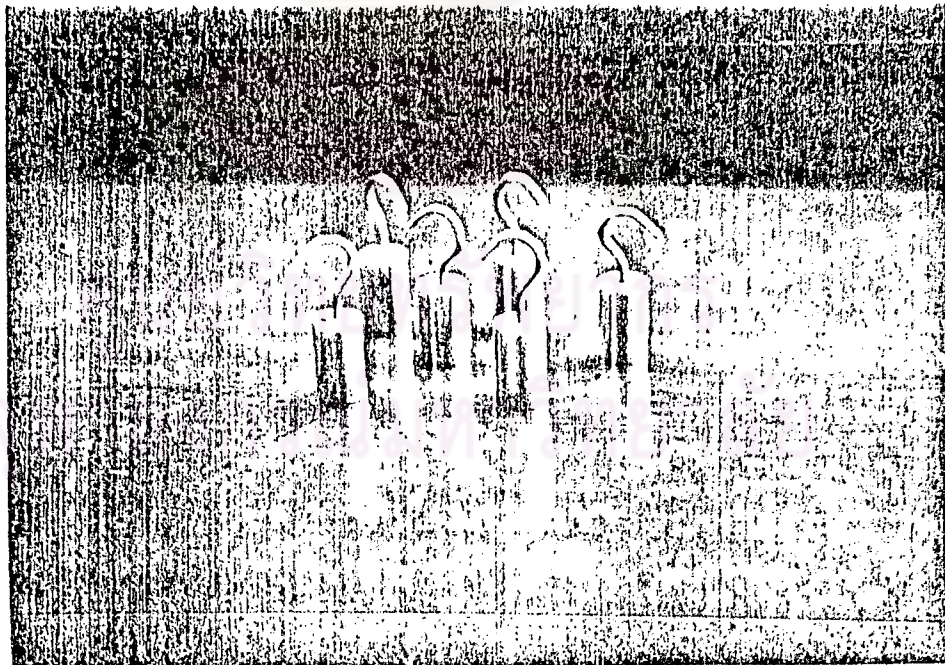


Fig 4.2 Weights

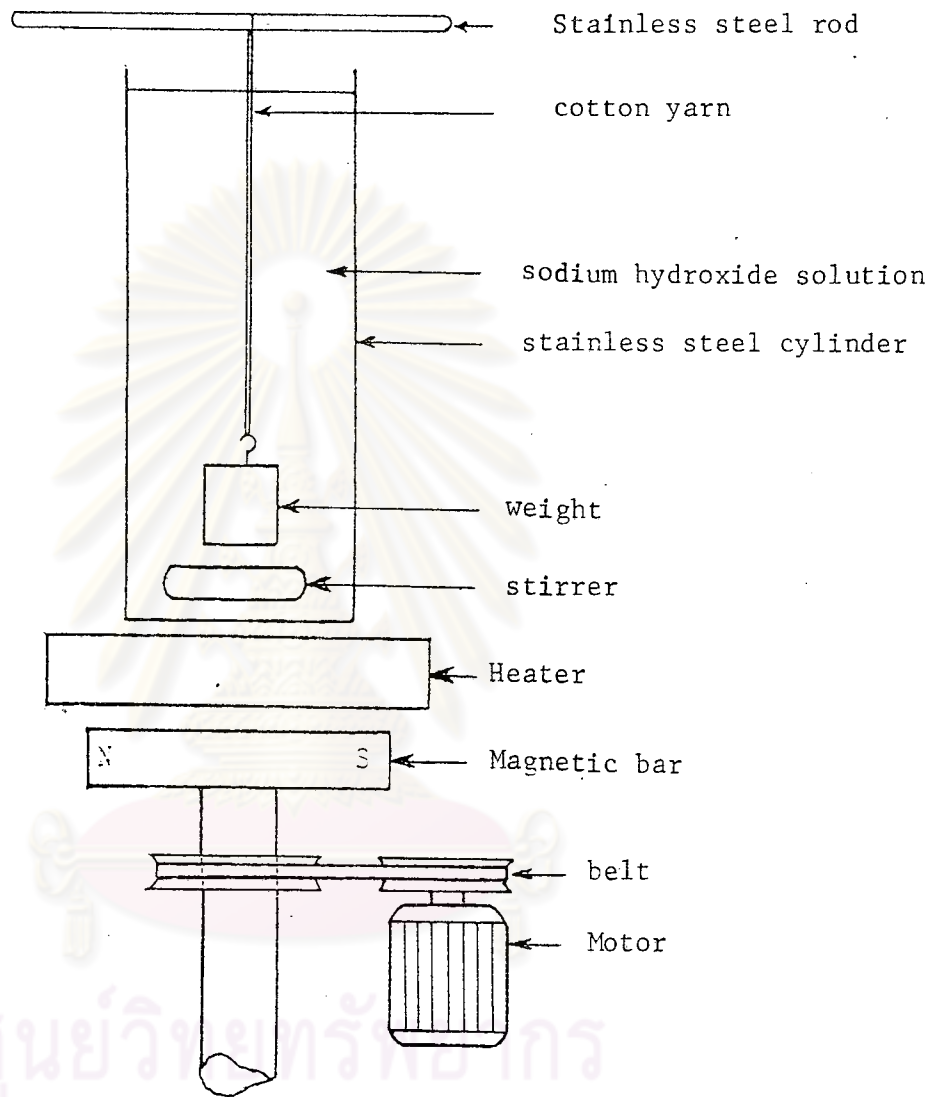


Fig 4.3 Explanation of apparatus

4.3.2 Sulfuric acid solution

Volume of concentrated sulfuric acid, analytical reagent grade, was measured with a graduate pipette of ± 0.05 ml and then poured into distilled water in a volumetric flask, filled the flask with distilled water to the marking line. The concentration of the solution was 1.0 % (w/w).

4.4 Preparation of Cotton Yarn

Cotton yarn which was used in this work is number 20. Before mercerization, cotton yarn had to be cleaned by scouring and bleaching and then it was prepared into a loop form as the following.

4.4.1 Scouring

Cotton yarn was boiled for 30 minutes with sodium hydroxide solution, concentration 1 gm/litre, and soap, concentration 0.5 gm/litre. Solution : cotton yarn ratio was 30 : 1 (w/w).

4.4.2 Bleaching

After scouring, it was bleached with hydrogen peroxide for 30 minutes at 343°K, concentration of 0.12 gm/ml. Solution : cotton yarn ratio was 30 : 1 (w/w).

4.4.3 Looping

The bleached yarn was cut in to short yarns, each of them was 15 cm. long and both ends were tied together into a loop form. It was dried at the room temperature for one week. It was then ready to be mercerized.

4.5 Experimentation

The experimental apparatus was prepared as shown in Fig. 4.1. The experiment was performed at the sodium hydroxide concentration of 4.80×10^{-3} , 5.92×10^{-3} , 7.96×10^{-3} , 1.06×10^{-2} and 1.27×10^{-2} gm-mole/ml, temperature of 303, 313, 323, 333 and 343 °K, tension of 112,380, 168,560, 224,755, 280,940 and 337,130 gm/cm² and speed of stirrer of 80, 200 and 750 rpm. The mercerization time for each run of the experiment was varied from 1-15 minutes. The plan of the experiment is shown in Table 4.1.

4.5.1 Method of experiment

The yarn loop which was prepared in 4.4.2 was put into the sodium hydroxide solution tank as shown in Fig. 4.1, the mercerization time was recorded by a stop-watch immediately as the yarn contacted with the sodium hydroxide solution. After a certain period of time, then the yarn was taken from the sodium hydroxide tank and put into the sulfuric acid tank for neutralization. It took about 20 minutes and then it was taken from the sulfuric acid tank and washed in the distilled water tank for removing the excess sulfuric acid after neutralization. The time for washing was 20 minutes, and the yarn was dried at the room temperature for one week.

4.6 Measurement of Mercerization

After washing, each yarn was cut cross sectionally in to slices, then they were taken photographs under a microscope with 1,000 times magnification. From the photographs of cross section of cotton fibre, external and internal radii of the cotton fibre were determined by measuring their circumferences and divided by 2π . Sixty radii were measured to find the average radius of each run of the experiment. From these radii, the rate, conversion and the appropriate model were determined with the aid of the theory derived in chapter 3. Fig 4.4 shows the cross sections of cotton fibre at various stages of mercerization (scale 1,000 : 1).



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157seconds



290 seconds



360 seconds



600 seconds

Fig 4.4 Cross-section of cotton fibre at various stages of mercerization. (scale 1000 : 1)



Table 4.1 Plan of the Experiment

Experimental number	$(C_{Na^+})_b$	T	S	V_e	Remark
1	4.8×10^{-3}	303	112,380	80	
2	4.8×10^{-3}	303	112,380	200	
3	4.8×10^{-3}	303	112,380	750	
4	4.8×10^{-3}	303	168,560	200	
5	4.8×10^{-3}	303	224,755	200	
6	4.8×10^{-3}	303	280,940	200	
7	4.8×10^{-3}	303	337,130	200	
8	4.76×10^{-3}	313	168,560	200	
9	4.74×10^{-3}	323	168,560	200	
10	4.72×10^{-3}	333	168,560	200	
11	4.69×10^{-3}	343	168,560	200	
12	1.27×10^{-2}	303	168,560	200	
13	1.06×10^{-2}	303	168,560	200	
14	7.96×10^{-3}	303	168,560	200	
15	5.92×10^{-3}	303	168,560	200	

$(C_{Na^+})_b$ = Bulk concentration of sodium ion in mercerizing solution, (gm-mole/ml.)

T = Mercerizing temperature. ($^{\circ}$ K)

S = Tension of cotton fibre. (gm/cm²)

V_e = Speed of stirrer. (rpm)