On Continuous Ageing Apparatus for Alkali-Cellulose

By Naozo Fukaya, Shimpei Chikaraishi, Kiyoshi Ikemoto,
Takeshi Ninomiya and Tadamasa Kakehi
Member, Mitsubishi Rayon Co., Ltd.

Abstract

The purpose of this study is to make the conventional ageing process for alkali-cellulose into a continuous process and to reduce manufacturing cost. Several test machines have been made and studied, and the last of them has been found to satisfy all the conditions required of a continuous ageing machine. The total length of time for ageing by this machine is about 3.5 hours, and the products obtained are superior to those made by conventional processes.

Introduction

Extensive studies on the continuous preparation of viscose have been made recently with a view to lowering the production cost and to improving the quality of viscose, and the batch process has now been partially replaced by a continuous method. For example, in the steeping, pressing and shredding processes for alkali-cellulose, continuous methods are now adopted in place of the conventional batch process. However, the subsequent process—the ageing process—still is carried on in cans, as attempts at continuous ageing have been unsuccessful in spite of great efforts made to that end. The present article is a report on a machine which we have successfully perfected for continuous ageing of alkali-cellulose.

Several test machines have been made and studied, and the last of them, as we have said, fulfils all the conditions required of a continuous ageing machine. It consists of three parts: (1) the heating part, (2) the reacting part, and (3) the cooling part. The first and third parts are equipped with mixing blades; the second part has none. The discharge zone of each part is provided with takeout apparatus designed after careful study, and the number of revolutions of this apparatus controls the transportation of alkali-cellulose linearly. The total time for ageing by this machine is about 3.5 hours, and the products obtained are of a very high quality.

1. Past Attempts at Continuous Ageing

The ageing of alkali-cellulose consists in storing shredded crumbs for a sufficient length of time to allow the accompanying air to oxidize the cellulose to a desired degree. The conventional method of ageing is to stuff alkali-cellulose into bins, or ageing cans, which are

stored at various room temperatures for periods of between 20 and 60 hours. A demand for ever greater production has stimulated attempts in Germany to speed up the ageing process and make it continuous. These attempts are reported on in detail in reports of C.I.O.S., B.I.O.S.⁽¹⁾ and F.I.A.T. The continuous methods of ageing referred to in these reports are:

- (1) Conveyor band type (Figure 1 a)
- (2) Rotary drum type (ex. Rotary kiln)
- (3) Büttner dryer type (Figure 1 b)

These types of agers are large in volume

Fig. 1. a Conveyor type

Fig. 1. b Büttner type

because of large void, and are undesirable in many other respects, so they are not used in Japan. Specific reasons why they are not used can be understood from the following conditions required of an ager.

2. Conditions Required of a Continuous Ager

A continuous ager should make good viscose at low cost, and this requirement demands the following mechanical conditions:

(1) Easy and correct temperature control for alkali-cellulose (during heating, reacting and cooling).

(2) Shortness of time during which alkali-cellulose should stay in an ager.

(3) Machine should be simple to construct and easy to maintain and repair.

(4) Smallness of void and the smallness of the machine in volume.

(5) Good enlargeability.

(6) Good controllability and absence of need for a watchman.

(7) (From a production quality standpoint) Output of Na_2CO_3 should be limited and cellulose should not dry or stick.

3. Results of Experiments Discussed

(A) Horizontal Mixing Blade Type

An experiment was made in which several horizontal cylinders were piled and inclined slightly and miscellaneous mixing blades used, but it was found that the void should be more than 80 percent in order to obtain alkali-cellulose equal in quality to that obtainable by the conventional batch type (Figure 2).

Cylinder:

Inclination 4°
Steps 6

Fig. 2 Continuous Ager (Old type)

Revolutions 3.1 rev./min.
Void 75%
Cylinder diameter 200 mm
" length 3000 mm
Top cylinder for heating
Bottom cylinder for cooling
Ageing time: 6 hrs. $\pm$ 30 mins.
Standard deviation $\sigma = 11.8$ min.
Ageing time fluctuation

$$= \frac{\sigma}{\text{mean temperature } \theta m} = \frac{11.8}{360} \times 100 = 3.28\%$$

The alkali-cellulose produced by this experiment had the composition shown in Table 1, the data being mean value every four hours.

Table 1. Cellulose & NaOH Content

	Before ageing							
Cell. (%)	31.86	32.42	32.10	31.60	32.22	31.34	31.99	31.93
NaOH (%)	15.33	15.32	15.25	15.29	15.11	15.28	15.17	15.25
	Per hour after ageing							
Cell. (%)	31.83	32.25	32.02	32.21	32.57	32.05	32.08	32.22
NaOH (%)	15.32	15.29	15.12	15.25	15.25	15.13	15.32	15.33
Cell. (%)	32.51	32.24						
NaOH (%)	15.18	15.19						

The mean degree of polymerization $\bar{P}$ was equal to 700 before ageing, and as shown in Table 2 after ageing. (Samples per 2 hours.)

Table 2. Variation of Mean Degree of Polymerization

260	270	250	260	290	270	260	300
Total mean				270			

The quality of yarn made from this alkali-cellulose was not particularly good, but its spinnability was excellent.

Table 3. Property of Yarn

Denier	Dry strength (g/d)	Wet strength (g/d)	Dry elongation (%)	Wet elongation (%)
2.08~2.28	2.24~2.44	1.32~1.54	14.8~16.7	19.9~24.1

In view of these findings, we abandoned the experimental ager as unsuitable for industrial purposes, and made researches looking to improved type of machine.

(B) Improved Type

(a) Construction

In researching for an improved ager, we directed our attention to the development of a better temperature control, the reducing of fluctuations in ageing time and the lessening of void. We succeeded in devising a splendid ager. This was the last of the several machines made and studied.

It is very important that continuous apparatus be so constructed that its reaction can be separated into unit processes and its phenomenon simplified. This is true of all types of continuous apparatus. In fact, we have divided the improved ager into three parts:

- (1) Alkali-cellulose heating part
- (2) Alkali-cellulose reacting part
- (3) Alkali-cellulose cooling part

The heating and cooling parts are differently constructed from the reacting part.

The cooling part is made larger than heating, by reason of the difference in holding time, but the two are the same in construction and have each a single bar type blade and special take-out apparatus⁽²⁾ (see Figure 3.) Mixing may be done with the blade, conveying may be done by inclination, and taking out may be done with the take-up.

Fig. 3 Continuous ager improved type

Thanks to the bridgeability of alkali-cellulose, the amount of alkali-cellulose to be delivered can be proportionally adjusted by the number of rotations of the blade. The mixing performance is very good, and fluctuations in ageing time are as small as $\sigma/\theta m$ when the void is 20~40%.

The overall heat transfer coefficient of $U = 30 \text{ Kcal/m}^2\text{hr } ^\circ\text{C}$ is the basis for fixing the dimensions of the apparatus. The dimensions of the improved ager under discussion are:

Capacity	Pulp 100 kgs./day
Inclination of heating and cooling cylinder	3°
Length of heating cylinder	1385 mm
Reacting cylinder	$400 \times (1650 + 300)$
Length of cooling cylinder	2085 mm
Total height	400 mm
Number of revolutions:	
Heating cylinder	13.6 r.p.m.
Cooling cylinder	12 r.p.m.

Motor (with P.I.V.) 1HP 1, $\frac{1}{2}$ HP 2

Cooling temperature 40°C , 35°C , 30°C

The reacting part is a jacketed vertical cylinder with numerous parallel blades at the bottom to take off alkali-cellulose linearly by gravity⁽³⁾ (void is 10~20%). The results of the experiment with this ager are shown by the following examples:

(c) Results of experiment discussed

(1) Ageing time

We measured fluctuations in ageing time in the ager by inserting red-colored alkali-cellulose between the testing materials.

Mean ageing time: heating time 10 mins.
reacting time 3 hrs.
cooling time 20 mins.

Standard deviation of fluctuations $= 5 \text{ mins.}$

Ageing time fluctuations $\frac{\sigma}{\theta m} = \frac{5}{210} = 2.4\%$

(2) Fluctuations in taken-off quantity

This is within the range of 150 g/min (mean value) $\pm 7\%$, as shown in Figure 4.

(3) Cellulose temperature

Fig. 4. Take-out quantity of alkali-cellulose

Heating cylinder 47°C
Reacting cylinder 45°C
○ Upper part of reacting cylinder
□ Lower part of reacting cylinder
Fig. 5 Cellulose temperature

Fig. 6 Composition of alkali-cellulose

This is almost constant in the heating and cooling parts, but in the reacting part it differs by about $1\sim 2^{\circ}\text{C}$ (Figure 5) due to the heat of depolymerization. Therefore, we should separate the jacket into upper and lower parts, and make the temperature in the lower part $1\sim 2^{\circ}\text{C}$ higher than in the upper (Figure 5).

As we have said earlier, the cooling part has to be cooled gradually to prevent alkali-cellulose from sticking. Therefore, the jacket is divided into three parts with a temperature differential of 5°C among them. The smaller the temperature differential is, the better the quality of the product.

(4) Fluctuations in the composition

The composition of alkali-cellulose is almost constant in the testing period, and is free from phenomena (drying, for instance) that make the quality worse.

In the inlet and outlet of the ager, rotary valves and cellulose beds are constructed to prevent contact with air. Therefore, hardly any change in the amount of Na_2CO_3 is found

before or after ageing.

Content of Na_2CO_3 before ageing $1.70\sim 1.81\%$
after ageing $1.75\sim 1.78\%$

(5) Mean degree of polymerization

After shredding $\bar{P} = 600$

After ageing $\bar{P} = 320\sim 350$

showing nothing abnormal.

(6) Property of viscose

We xanthated and dissolved one kilogram of alkali-cellulose in a small kneader, and obtained the same viscose as by the conventional process.

Conclusion

(1) We have explained the construction of a new continuous ager for alkali-cellulose and our experiments with it. The new ager reduces to very limited proportions the defects of the conventional ager—fluctuations in ageing time and in quality.

(2) We have separated the reaction into moderate units, e.g., mixing, transportation, heating and cooling, and have adopted most suitable apparatus for each unit. We use a horizontal cylinder for heating and cooling and vertical cylinder for ageing. Each outlet is equipped with special take-out apparatus and the amount of alkali-cellulose to be delivered can be proportionally adjusted by the number of rotations of the blade.

(3) This ager is simple in construction and can be made at low cost. Its enlargeability and controllability are excellent. For instance, dimensions of apparatus on a scale of 20 t/day, S.F are shown below:

Reacting part $2\text{ m}\phi \times 4\text{ m}$ (height)
Heating part $1\text{ m (breadth)} \times$
 $0.8\text{ m (height)} \times 3.5\text{ m (length)}$
Cooling part $1\text{ m (breadth)} \times$
 $1.2\text{ m (height)} \times 5\text{ m (length)}$

(4) The products obtained are superior to those made by the conventional process.

References:

- [1] B.I.O.S., No. 33, The German Rayon Industry during the Period (1939~1945).
- [2] Japanese Patent, Showa 30 (1955) No.6095
- [3] Japanese Patent, Showa 28 (1953) No. 2810

Table 4 Property of Viscose

Alkali-cellulose (after shredding)		Viscose		Viscosity (Poise)	Degree of Xanth- ation	By- product	Non-dissolving fiber				KW	Heating temp. °C	Agent temp. °C
Cell. (%)	NaOH (%)	Cell. (%)	NaOH (%)				Ball	Rod	FG	G			
32.85	15.15	8.01	6.11	33	48	0.63	0	0	0	20	294	47	47
32.85	15.15	7.91	6.03	33	48	0.74	0	10	0	30	206	47	47
32.85	15.15	8.11	6.14	38	46	0.81	0	30	20	50	250	47	45