DEVELOPMENT OF RAYON STAPLE PRODUCED BY VISCOSE HAVING HIGH DEGREE OF POLYMERIZATION AND SOME RHEOLOGICAL PROPERTIES OF SUCH VISCOSE

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INTRODUCTION

We have been engaged for more than 20 years into the investigation of the viscose fibres having a high degree of polymerization. The original viscose fibre developed by us was named Toramomen. Thereafter, T-51, T-61, etc. were made public successively by improving the original Toramomen fibre.

The speciality of the Toramomen process lies in that a high viscosity viscose having highly polymerized cellulose is spun into a low acid and low salt concentration bath. (Hereafter, such a viscose is abbreviated to Polynosic viscose.) Its spinning feature is fairly different from that of conventional viscoses to which the Müller's bath is applied.

At first, the Toramomen process was established through comprehensive experimentation. However, to make further progress in this process, it will be quite important to make clear its theoretical meaning. With this aim we are studying the rheological properties of the Polynosic viscose. These properties are also important for the engineering, because the behaviours of a viscose in the spinnerets, viscose distributors, viscose pipes, etc. are controlled by such properties.

FLOW CURVES OF POLYNOSIC VISCOSE

The flow behaviours of a Polynosic viscose were studied by using various capillary tube viscometers of different lengths and diameters. A viscose to be tested is extruded from a capillary under various pressures (P) at 20°C, and the extruded volume (Q) of the viscose per unit time is measured at each pressure.

The flow curve is indicated as the log-log plot of the shear stress $(\tau_{w'} = PR/2L)$ at the capillary wall versus the apparent shear rate $(D_{w'} = 4Q/\pi R^3)$ at the capillary wall, where L is the length of capillary tube, and R is its diameter. The flow curves measured by a series of capillaries having same diameter but different lengths are shown in *Figure 1*. The corrections for both end effects, i.e. the flow-in effect at the inlet and the kinetic energy effect at the outlet, are not applied.

Various values concerning the rheological properties of a viscose are known from a linear part of the curves obtained with a capillary having



Figure 1. Flow curves of a Polynosic viscose measured by a series of capillaries having same diameter but different lengths

a fairly high length-to-diameter ratio. In this experiment, the flow behaviour index n, which corresponds to the slope of a flow curve, is 0.29-0.33for the Polynosic viscoses.

In addition, we would like to point out that both high and low shear stress parts of these curves seem to behave more Newtonian. We think that



Figure 2. Flow curves of a conventional viscose measured by a series of capillaries having same diameter but different lengths

this phenomenon originates probably from the characteristics of a viscose itself.

The flow behaviours of a conventional viscose are shown in Figure 2. The flow behaviour index n of this conventional viscose is 0.45. It is clear that the Polynosic viscose has fairly higher structural viscosity in comparison with the conventional viscose.

The flow curves of a Polynosic viscose, measured by a series of capillaries having the same length-to-diameter ratio but different diameters, are shown in *Figure 3*. So far as this result is concerned, the existence of the so-called wall effect cannot be proved.



Figure 3. Flow curves of a Polynosic viscose measured by a series of capillaries having same length-to-diameter ratio but different diameters

The flow behaviours of a viscose, being extruded from the spinneret, will be prescribed naturally by the rheological properties of that viscose. In general, the effective hole-length of a conventional spinneret is about twice the spinneret hole-diameter. On the other hand, the length-todiameter ratio of a glass spinneret is 20 to 30. Therefore, the capillaries having low length-to-diameter ratios will correspond to the metal spinneret, and those of high ratios to the glass or ceramic spinneret.

BARUS EFFECT OF POLYNOSIC VISCOSE

Certain kinds of liquids having high viscosity expand when extruded from a capillary. This phenomenon is known as the Barus effect. As a parameter representing the Barus effect, the flow-broadening effect D/D_0 has been adopted in this study.

The flow-broadening effect of a Polynosic viscose was observed by a microscope simultaneously with the measurement of flow curves mentioned above, where D is the maximum diameter of a viscose flow being extruded from a capillary, and D_0 is the inner diameter of that capillary. In *Figure 4*, the flow-broadening effect of a Polynosic viscose is plotted to the logarithm of apparent shear rate.

It is noteworthy that each flow-broadening curve has maximum value at certain shear rate and, furthermore, the flow-broadening effect has some relationship with the flow curve mentioned above.

The flow-broadening effect of a conventional viscose is shown in Figure 5.



Figure 4. Flow-broadening effect of a Polynosic viscose measured by a series of capillaries having same diameter but different lengths



Figure 5. Flow-broadening effect of a conventional viscose measured by a series of capillaries having same diameter but different lengths

It seems that the maximum effect of various capillaries once decreases to a certain constant value and then increases, as the length-to-diameter ratio of the capillary increases. Such tendency seems more distinguished in conventional viscose, although the maximum value of the flow-broadening effect of the conventional viscose is generally lower than that of the Polynosic viscose.

Now, let the flow rate of a viscose in a spinneret hole be V, then the rate of viscose filaments extruded from the spinneret will decrease to $V/(D/D_0)^2$, owing to the flow-broadening effect of the viscose. This decreased speed is regarded practically as the initial spinning speed. Therefore, the length-to-diameter ratio of a spinneret hole, which has some relation to the flow-broadening effect, will have an influence upon the initial spinning speed of the viscose filaments.

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We think that this is at least one of the reasons why the spinning speed of Polynosic viscoses is relatively low and the glass spinneret is appreciated for the spinning of Polynosic viscoses.

As the dimensions of the capillary tube viscometers used in this experiment are fairly larger in comparison with those of the spinneret hole, it is difficult to presume the exact value of the flow-broadening effect in the actual spinning directly from the above-mentioned experiment.

However, this effect will be estimated roughly from the spinning experiment as follows. We looked for the lowest draw off speed of viscose filaments by reducing the revolution of draw off godet until the tension of those filaments disappeared. This lowest speed is to correspond approximately to the initial spinning speed. As such lowest speed of a Polynosic viscose, 3.5 m/min for a metal spinneret, and 6.5 m/min for a glass spinneret were obtained. In this case, the flow-rate of viscose in the spinneret hole was 13.8 m/min in either spinneret. The flow-broadening effects calculated from these values are c. 2 for the metal spinneret and c. 1.5 for the glass spinneret, respectively.

MEASUREMENT OF STRUCTURAL VISCOSITY BY USING A ROTATIONAL VISCOMETER

On the other hand, we have discovered that the structural viscosity is conveniently obtained by using a rotational viscometer of the Brookfield type. Plotting the logarithm of viscosities, obtained in various revolutions of same rotor of the viscometer, to the square root of r.p.m. of that rotor, a straight line is obtained. It is thought that the slope of the line corresponds to the structural viscosity $(\Delta \eta)$. This measurement is so simple that we are able to use it as a routine method. The relationship between cellulose concentration of a Polynosic viscose and $\Delta \eta$ of that viscose is shown in *Figure 6*.



Figure 6. Relationship between cellulose concentration and $\Delta \eta$ of a Polynosic viscose, and influence of temperatures upon it

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This experiment indicates that the cellulose concentration of Polynosic viscoses is adjusted on the whole to such a point that the change of $\Delta \eta$ accompanying the variation of cellulose concentration becomes smallest.

The effects of the ripening time and the ripening temperature upon $\Delta \eta$ of a Polynosic viscose are shown in *Figure 7* in which it is indicated that the



Figure 7. Progress of $\Delta \eta$ during viscose ripening, and influence of temperature upon it

 $\Delta \eta$ curve passes through a minimum value at ripening of viscose and the lowest value of $\Delta \eta$ as well as its position are influenced by the ripening temperature.

THEORETICAL CONSIDERATIONS OF POLYNOSIC VISCOSE SPINNING

The structural viscosity is related to the degree of entanglement among cellulose molecules in a viscose. Such entanglement will dominate the reactivity of viscose, i.e. the coagulation rate of the viscose, the regeneration rate of cellulose, and the crystallization rate of cellulose. The degree of coagulation is presumed by the measurement of gel swelling value of spun thread, and the regeneration rate by the measurement of residual γ -value of spun thread.

On the other hand, it is not unreasonable to assume that the degree of progress of the fibre structure during the spinning stage (crystallization) roughly corresponds to the swelling value of the thread after being treated in distilled water for a few minutes.

With these considerations, we are able to presume the progress of inner structure of the Polynosic fibre, and it has been found that the rate of structure formation at Polynosic fibres is fairly low and its slow rate is due, to a considerable degree, to the fine structure of Polynosic viscose.

INTRODUCTION OF T-61 ADVANCED

Next, we would like to introduce briefly a new Polynosic fibre "T-61 Advanced" recently developed by us.

As mentioned above, we could presume the process of fibre-structure-

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formation during the spinning stage, and the T-61 Advanced process has been developed by the application of such knowledge.

T-61 Advanced process is characterized by the fact that the spun thread is treated by the so-called relaxation liquor being selected in conformity with the degree of development of inner structure of the thread. This means that such liquor must be selected in accordance with the period or the place of the treatment as well as with the history of the thread.

By such treatment the loop tenacity of the fibre has been remarkably improved, without any material deterioration of its so-called Polynosic properties. Furthermore, the treated fibre has fine crimps which have been looked forward to for a long time in the Polynosics. In *Table 1*, the standard characteristics of T-61 Advanced are given in comparison with the original T-61 fibre. These tests were done according to the Japanese Industrial Standard (JIS) and the Japanese Polynosic Testing Method.

	T61 Advanced	<i>T–61</i>
Denier (den.)	1.5	1.5
Wet tenacity (g/den.)	3.5-3.8	$3 \cdot 5 - 3 \cdot 8$
Wet elongation (%)	11-13	9-10
Loop tenacity (KM)	9-11	5–6
Wet elongation at 0.5 g/den. (%)	<3.0	< 3.0
After 5% NaOH-treatment		
Wet elongation at 0.5 g/den. (%)	< 6.0	<6.0
Wet tenacity (g/den.)	>3-0	>3.0
Degree of dyestuff absorption (%)	76	58
Degree of swelling (%)	68	67
Degree of polymerization	600-650	600-650
Appearance	curled	straight

Table 1. Standard characteristics of T-61 Advanced

It is clear that the loop tenacity of T-61 Advanced is almost doubled compared with the value of the original T-61 fibre. Nevertheless, the other mechanical properties are equal in both fibres.

RELATIONSHIP BETWEEN FLEXING ABRASION RESISTANCE OF FABRICS AND LOOP TENACITY OF FIBRES

In Table 2 is shown the abrasion resistance of various fabrics.

Material	T–61 Advanced	T-61	T-51	Cotton	Conventional rayon staple
Abrasion Resistance (cycle) Surface Abrasion Flexing Abrasion Folding Abrasion	57 756 43	56 268 23	50 241 27	40 1319 32	$\frac{46}{20}$

Table 2. Comparison of abrasion resistance of various fabrics

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As seen in *Table 2*, the serious defect of rayon fabrics lies in their abrasion resistance, especially flexing abrasion resistance; this is considerably low in comparison with cotton fabrics, and, in this point, T-61 Advanced has been fairly improved as compared with T-61 or T-51.

A key to increase the flexing abrasion resistance of rayon fabrics, is found in *Figure 8* which indicates that the loop tenacity of fibres has a close correlation with the flexing abrasion resistance of fabrics.



Figure 8. Relationships between loop tenacity of fibres and flexing abrasion resistance of those fabrics

TWISTABILITY OF T-61 ADVANCED

In the spinning test of 60 counts yarn, it was proved that the number of breakages of T-61 Advanced at the ring spinning, was exceedingly small as compared with T-51 or HWM fibre. The loss of yarn strength in the twisting test is also smallest at T-61 Advanced. From these facts, we can expect the stability of T-61 Advanced yarn against high twisting. To confirm this the influence of 2nd twist upon the tenacity of various yarns was exa-



Figure 9. Effect of 2nd twist on single yarns strength spun from various fibres

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mined. The results are shown in Figure 9. It is obvious that the tenacitytwisting behaviour of T-61 Advanced yarn is entirely different from other Polynosic yarns or HWM yarn, and approaches that of cotton. The number of yarn-breakages during the additional twisting is shown in Table 3.

Material fibre	Additional turns per metre				
	500	1000	1250	1500	
Cotton	0	0	1	5	
T–61 Advanced	0	2	8	84	
T–61	4	68	524	2836	
T-51	0	56	656	3512	
HWM Fibre	12	700	2496	7360	

Table 3. Number of breakages per pound during second twist

That of T-61 Advanced is remarkably reduced and is nearly on a level with cotton. The conspicuous improvement of T-61 Advance concerning the twist behaviour is attributed to its high loop tenacity.

With the results available at present it is not unreasonable to expect the development of the crepe yarns, for which a strong twist is indispensable, by using T-61 Advanced.

As seen in Figure 9, although the loop tenacity of T-61 Advanced has been remarkably increased, it is not yet sufficient as compared with cotton. The final goal of T-61 Advanced will be to increase its loop tenacity up to a level with cotton, i.e. from 11 KM to 14 KM. We believe that such goal will be realized before long.

We think that T-61 Advanced is one of the exceedingly interesting fibres at present and its development is an important contribution for the advancement of the viscose rayon fibres in the future.