

# On the Densities of Various Fibres and the Widths of the Intermicellar Spaces

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## Abstract

Intermicellar spaces of various textile fibres were examined by measuring their densities and their swellings in water, in various primary normal alcohols, and in liquid paraffin. It was observed that the density of a fibre which was measured in methyl alcohol was greater than those measured in the other liquids, and that the density decreased with the number of carbon atoms of the alcohols and the liquid paraffin.

## Introduction

The density of a fibre has frequently been measured in water. But according to the recent research made by the writer<sup>1</sup> the water molecules penetrate, as molecular groups, into the intermicellar space of the fibre which is constructed with the micellar structure. And the water molecules, even when the fibre is swollen perfectly in it, cannot completely penetrate into the intermicellar space. This means that the density of the fibre measured in water may be considered as giving an apparent density somewhat smaller than its net density. Thus the density of the fibre measured in methyl alcohol, the chain length of whose molecules are short, is greater than that measured in water, as has already been reported in connection with silk and ramie.

According to G. W. Stewart and R. M. Morrow,<sup>2</sup> the lateral thickness of the molecules of the primary normal alcohols is considered to be approximately constant, and their chain length increases with the number of the carbon atoms.

When the density of the fibre is measured in various primary normal alcohols, the molecules of the alcohols penetrate into the intermicellar space to different degrees according to the chain length of the alcoholic molecules. Consequently the variation of the densities measured in various primary normal alcohols shows the proportion of the intermicellar spaces having different widths.

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1. Y. Matsunaga: These Memoirs, **20**, 157 (1937).

2. G. W. Stewart & R. M. Morrow: Phys. Rev., **30**, 232 (1927).

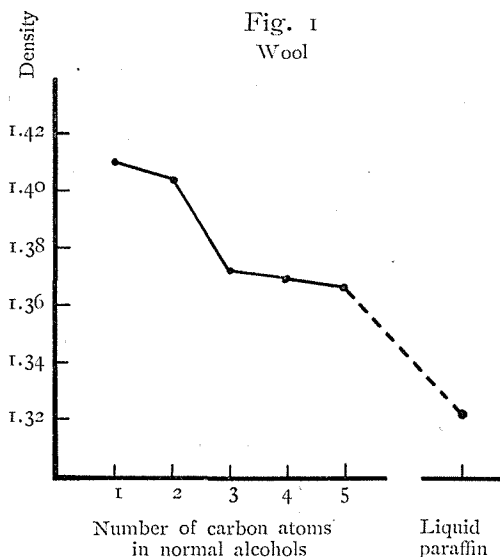
In the present investigation the writer measured the density of wool, silk, reworked silk, ramie, cotton wool, and viscose rayon in water, in various primary normal alcohols and in liquid paraffin by the evacuation method employed by U. Yoshida and B. Takei,<sup>1</sup> which is especially suitable for obtaining the net densities of porous and powdery substances; and at the same time the mean lateral swelling of the fibres in the above mentioned liquids were measured.

### Consideration from the Density

The densities of wool, silk, reworked silk, ramie, cotton wool, and viscose rayon were measured in water, in primary normal alcohols

Table I

	Wool	Silk (Fibroin)	Reworked silk	Ramie	Cotton wool	Viscose rayon
Water	1.399	1.426	1.425	1.614	1.604	1.614
Methyl alcohol	1.410	1.483	1.440	1.629	1.641	1.614
Ethyl alcohol	1.404	1.445	1.409	1.591	1.637	1.580
n. Propyl alcohol	1.372	1.382	1.361	1.589	1.597	1.580
n. Butyl alcohol	1.367	1.380	1.364	1.584	1.597	1.563
n. Amyl alcohol	1.366	1.382	1.366	1.580	1.600	1.550
Liquid paraffin	1.322	1.363	1.329	1.519	1.558	1.522



from methyl to amyl and in liquid paraffin by the evacuation method devised by U. Yoshida and B. Takei. The results are tabulated in Table I, and are shown in Figs. from 1 to 6.

As will be seen in the Table, and by the figures the densities of these fibres, except viscose rayon, are greater in methyl alcohol than when measured in water and they decrease with the size of the mole-

1. U. Yoshida & B. Takei: These Memoirs, 15, 1 (1932).

Fig. 2  
Silk

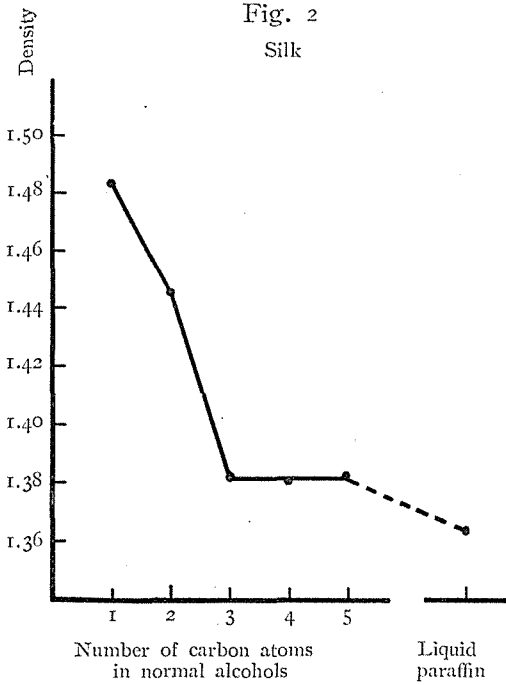


Fig. 4  
Ramie

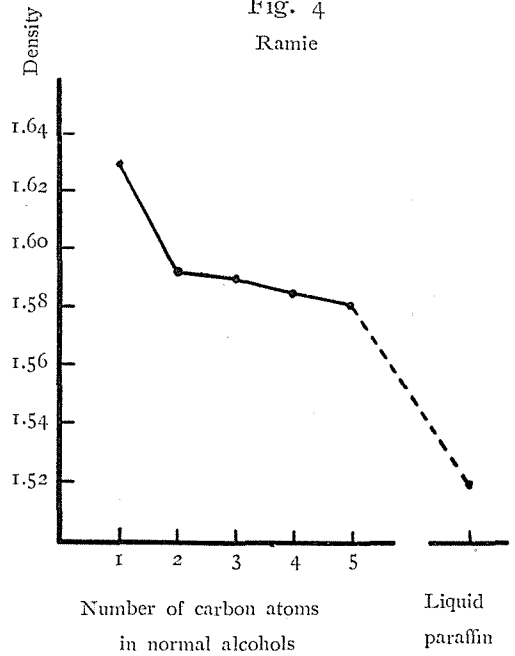


Fig. 3  
Reworked silk

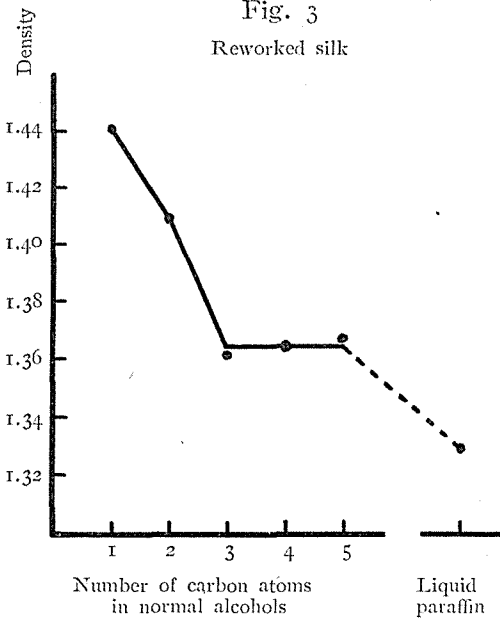
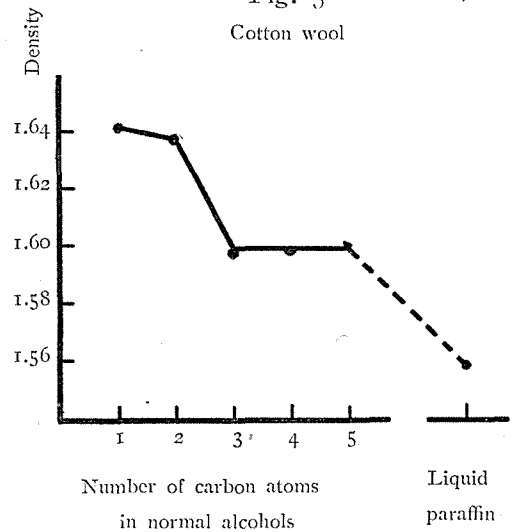
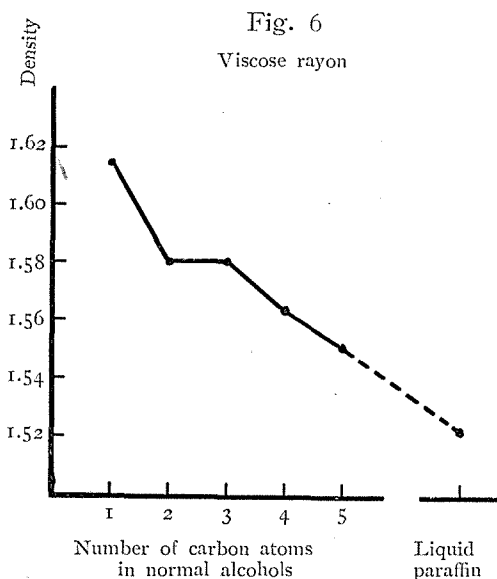


Fig. 5  
Cotton wool





cules of various kinds of alcohols; but the density of viscose rayon is the same in methyl alcohol as in water and decreases with the size of the molecules of various kinds of alcohol.

A thorough investigation with the X-ray diffraction of the primary normal alcohols from methyl to lauryl, has already been carried out by G. W. Stewart and R. M. Morrow.<sup>1</sup> Each alcohol gave rise to two diffraction

peaks. One of these peaks was practically independent of the carbon content of the alcohol and appeared to be associated with the lateral thickness of the molecule. The other is associated with the chain length and gives an increase of 1.3 A. U. per one carbon atom. Next B. E. Warren<sup>2</sup> has given a more correct interpretation of the inner peaks observed with alcohols and fatty acids. The corrected lengths of the chain of primary normal alcohols are tabulated in Table II.

Table II

Number of carbon atoms	Length of chain in A. U.
1	4.3
2	5.7
3	6.9
4	7.9
5	9.0

The normal paraffins were studied by C. H. Sogani<sup>3</sup> and G. W. Stewart.<sup>4</sup> The formula proposed by Stewart for their chain length  $L$  is

$$L = \frac{\text{Molecular weight} \times \text{Mass of H atom}}{(4.64 \times 10^{-8})^2 \times \text{Density}}$$

When the values  $L$  obtained from the above relation are plotted against the number  $n$  of the carbon atoms in the chain molecules of the

1. G. W. Stewart & R. M. Morrow: *loc. cit.*

2. B. E. Warren: *Phy. Rev.*, **44**, 969 (1933).

3. C. H. Sogani: *Indian J. Phys.*, **2**, 97 (1928).

4. G. W. Stewart: *Phys. Rev.*, **31**, 174 (1928) & **32**, 153 (1928).

paraffins, it is found that the above formula is reduced to the following linear relation :

$$L = 1.24 n + 2.70.$$

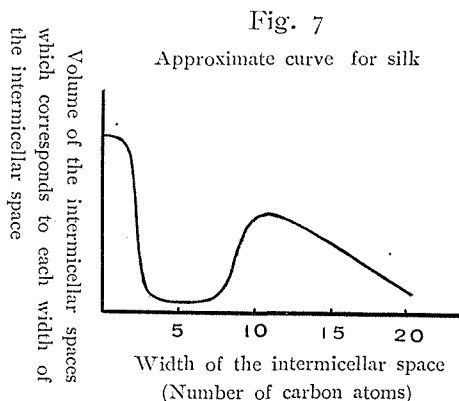
Thus the distance 1.24 A. U. represents the distance between two adjacent carbon atoms. By assuming as is reasonable, that the liquid paraffin used in the present experiment consists only of normal paraffins, the number of the carbon atoms is estimated to be 10-15; which give the values of 15-20 A. U. to the chain lengths of the paraffin molecules by the above formula.

The reason why the densities of the fibres decrease with the length of the molecular chain of the alcohols and liquid paraffin seems to be the increasing difficulty of penetrating into the narrow intermicellar space of the fibres with increasing chain lengths of the molecules.

### Consideration on the Intermicellar Space

In Figs. from 1 to 6, the densities of the fibres are taken as the ordinates and the number of the carbon atoms of various alcohols and liquid paraffin are taken as abscissae. As is evident from the curves in these figures, the density of every fibre decreases at first as the number of the carbon atoms of the alcohols increases, then it becomes constant for a few of the following carbon atoms, and lastly it decreases again with the increasing number of the carbon atoms. This behaviour of the density of the fibres is due to the uneven proportion of the intermicellar spaces having different widths. The fact that the density of a fibre is constant for a time in alcohols containing a successively increasing number of carbon atoms indicates that the volume of the intermicellar spaces of a fibre into which the molecules of these alcohols can and cannot penetrate are respectively the same for all of these alcohols.

Consequently it may be inferred that the intermicellar spaces having a range of widths somewhat narrower than the chain lengths of these alcohols are missing. Thus the intermicellar spaces of a fibre may be said to consist of two parts: one which is narrower than and the other which is wider than



the missing portion. The proportion of the volume of the intermicellar spaces having different widths is shown schematically by the curve in Fig. 7 for the case of silk as an example.

In the case of wool fibre, silk fibre, reworked silk fibre and cotton wool fibre the first intermicellar space is narrower than the chain length of the molecule of propyl alcohol, and the second one is wider than the chain length of the molecule of amyl alcohol. In the case of ramie fibre, the first intermicellar space is narrower than the chain length of the molecule of ethyl alcohol and the second one is wider than the chain length of the molecule of amyl alcohol. Lastly in the case of viscose rayon the first intermicellar space is narrower than the chain length of the molecule of ethyl alcohol and the second one is wider than the chain length of the molecule of propyl alcohol.

N. Matsumoto<sup>1</sup> estimated the width of the intermicellar space of viscose rayon by means of X-ray patterns. The specimen was at first immersed in a solution of about 0.5%  $\text{HAuCl}_4$ , and then the  $\text{HAuCl}_4$  in the fibre was reduced to Au in the vapour of Hydrazine Hydrate. The size of the microcrystals of Au thus deposited in the intermicellar space of the fibre was estimated from the microphotometer curve of Debye-Scherrer X-ray patterns, and he concluded, from the size of Au-crystals thus estimated, that the mean width of the intermicellar space of viscose rayon is 65-69 A. U. Recently Frey-Wissling<sup>2</sup> measured, by the same method, the widths of the intermicellar space of various fibres. The results are as follows: natural cellulose 85-135 A. U., acetyl cellulose 60 A. U., viscose rayon 50 A. U., silk fibre 50 A. U. and wool fibre 60 A. U.

On the other hand Y. Hiratsuka and J. Hori<sup>3</sup> estimated the width of the intermicellar space of ramie fibre as 17-16 A. U., by assuming that the volume of water which was adsorbed from the perfectly dry state to the normal state is equal to the total volume of the intermicellar spaces, and also that the size of the micell is  $200 \times 200 \times 500$  (A. U.)<sup>3</sup> or  $200 \times 200 \times 1000$  (A. U.)<sup>3</sup>.

More recently S. Iwasaki and T. Miyamoto<sup>4</sup> investigated the serigraph curve of viscose rayon immersed in paraffins, in normal al-

1. N. Matsumoto: Soc. Chem. Industry Japan, **37**, 827 (1934).

2. Frey-Wissling: *Protoplasma*, **27**, 372 (1937).

3. Y. Hiratsuka & J. Hori: *Oyo Butsuri*, **4**, 9 (1935).

4. S. Iwasaki & T. Miyamoto: Soc. Chem. Industry Japan, **41**, 440 (1938).

cohols, and in normal fatty acids; and concluded that two kinds of intermicellar spaces exist, having different widths.

Thus the widths of the intermicellar spaces of the fibres are different according to the methods of measurement. This seems to indicate that the intermicellar spaces of a fibre are not uniform, but their widths extend unevenly over a pretty wide range in accordance with the present investigation, and that as the experimental method is different, different portions of the intermicellar spaces of different width are detected.

Next the writer measured with a microscope the lateral swellings of the fibres in water, in various primary normal alcohols, and in liquid paraffin. After the diameter of a fibre was measured in the air, the gases and water adsorbed by the fibre were taken off by evacuating with a Cenco Hyvac pump the glass tube containing the fibre at 100°C, and the glass tube was sealed. Then the glass tube was broken in water, in an alcohol or in liquid paraffin, and the diameter of the fibre was again measured after it had stood for 48 hours in it. The results are tabulated in Table III. The figures given in Table III refer to the ordinary state in air, which is swollen by a certain quantity of water. Therefore the positive sign indicates the expansion and the negative sign the contraction of the fibres in reference to the ordinary state in air.

Table III  
Lateral swelling in % in reference to the ordinary state

	Wool	Silk (Fibroin)	Reworked silk	Ramie	Cotton wool	Viscose rayon
Water	+18.0	+19.7	+19.7	+13.0	+17.4	+31.5
Methyl alcohol	+ 3.8	+ 2.6	- 0.3	- 0.9	+ 4.4	+20.6
Ethyl alcohol	- 0.4	+ 3.8	- 0.5	+ 0.6	+ 3.8	+18.1
n. Propyl alcohol	+ 0.1	+ 2.6	- 2.6	+ 3.6	+ 3.4	- 2.7
n. Butyl alcohol	+ 1.6	+ 2.1	- 3.1	+ 0.6	+ 3.0	+ 0.2
n. Amyl alcohol	- 0.9	+ 1.6	- 3.0	- 0.2	+ 0.5	- 0.1
Liquid paraffin	- 0.9	- 3.1	- 4.1	- 5.2	- 1.8	- 4.8

Generally the lateral swelling of every kind of fibre is especially large in water, and in the alcohols and in liquid paraffin it is only a small expansion or contraction, less than a few per-cent, except with the viscose rayon in methyl and ethyl alcohols where the lateral swelling rises to about 20%. The prominent swelling in water seems to

be due to its surface tension which is remarkably strong as compared with that of the alcohols and the liquid paraffin; and the general tendency for the swelling to decrease with the number of carbon atoms in the alcohols and the liquid paraffin is probably caused by the increasing difficulty in penetrating into the narrow intermicellar spaces with the increasing chain length of the molecules.

### Discussion

In Table III the figures of the lateral swellings of the fibres are respectively calculated in reference to the lateral dimensions of the fibres at an ordinary state. But under ordinary atmospheric conditions in Japan, namely a relative humidity of 70-80%, the fibres are already swollen to some extent as compared with the perfectly dry state.

The moisture thus adsorbed by the fibres at an ordinary state is taken off together with adsorbed gases by evacuation in measuring the lateral swelling; and a small contraction of the fibre will of course accompany this process. The apparent negative values of the lateral swelling as given in Table III have their origin in this fact; and the minimum diameters of the fibres measured in the liquid paraffin, corresponding respectively to the maximum apparent negative swellings, are taken approximately to be the same as those in the perfectly dry state.

Table IV shows the increase of the volume, expressed in per-cent, of the fibres swollen in various liquids in reference to their volume in liquid paraffin. Here it must be noted that the longitudinal swellings of the fibres are disregarded in calculating the increase of the volume by swelling since it is actually very small.

Table IV  
Increase of volume by swelling in %

	Wool	Silk	Reworked silk	Ramie	Cotton wool	Viscose rayon
Water	41.8	52.5	55.8	42.1	43.0	86.9
Methyl alcohol	9.6	12.1	8.0	9.2	13.0	57.3
Ethyl alcohol	1.0	14.7	7.7	12.6	11.7	50.8
n. Propyl alcohol	2.0	12.1	3.2	19.5	10.9	2.2
n. Butyl alcohol	5.1	11.1	2.0	12.6	10.0	8.6
n. Amyl alcohol	0.0	9.8	2.2	10.9	4.7	7.7
Liquid paraffin	0.0	0.0	0.0	0.0	0.0	0.0



Table V  
Values of  $\frac{V_2''}{V_1}$

	Wool	Silk (Fibroin)	Reworked silk	Ramie	Cotton wool	Viscose rayon
Water	0.513	—	—	0.523	0.506	0.981
Methyl alcohol	0.170	0.220	0.171	0.171	0.190	0.667
Ethyl alcohol	0.078	0.248	0.167	0.207	0.176	0.598
n. Propyl alcohol	0.088	0.220	0.119	0.281	0.168	0.083
n. Butyl alcohol	0.126	0.209	0.106	0.207	0.158	0.151
n. Amyl alcohol	0.067	0.195	0.108	0.189	0.102	0.142
Liquid paraffin	0.067	0.088	0.084	0.072	0.053	0.060

As was reported in a previous paper,<sup>1</sup> silk fibroin swells in water both in inter- and intramicellar manner. But in the alcohols and in liquid paraffin intermicellar swelling only occurs with silk; and with all other fibres examined in the present experiment no intramicellar swelling was detected with any kind of liquid examined. So in the following all calculations made are assumed to apply to intermicellar swelling, omitting the exceptional case of silk and reproduced silk in water.

Now let us denote by

$D$  : the net density of a fibrous material

$V_1$  : the net volume of a fibrous material

$M$  : the net mass of a fibrous material

$v$  : the volume of intermicellar space of a fibrous material in perfectly dried state

$V_2''$  : the volume of intermicellar space of a fibrous material when it is swollen entirely in a certain liquid

$v'$  : the volume of the intermicellar space into which the liquid molecules cannot intrude when fully swollen in a certain liquid

$d$  : the apparent density of a fibre measured in a liquid

$s$  : the increased volume percentage of a fibre when swollen in a certain liquid as compared with perfectly dried state.

Though it is conceivable that there are some very tiny intermicellar spaces of a fibre into which even the molecules of methyl alcohol cannot intrude, in the following we take approximately the density of a fibre measured in methyl alcohol as  $D$ , i. e. the net density of the

1. Y. Matsunaga: *loc. cit.*

fibre. Next let  $d_p$  be the density of a fibre measured in liquid paraffin, and if we assume roughly that the liquid paraffin molecules cannot intrude into the intermicellar space of a fibre entirely, as the density of a fibre is minimum in liquid paraffin so far as the present experiment is concerned, then

$$\frac{M}{V_1} = D, \text{ or } M = DV_1, \quad (1)$$

$$\frac{M}{V_1 + v} = \frac{DV_1}{V_1 + v} = \frac{D}{1 + \frac{v}{V_1}} = d_p. \quad (2)$$

By putting the values of  $D$  and  $d_p$  as tabulated in Table I in the equation (2), we get the numerical values of  $\frac{v}{V_1}$  for various fibres.

Now, from the density of a fibre measured in a liquid, we have the following equation :

$$\frac{M}{V_1 + v'} = \frac{DV_1}{V_1 + v'} = \frac{D}{1 + \frac{v'}{V_1}} = d, \quad (3)$$

and we obtained the numerical values of  $\frac{v'}{V_1}$  as given in Table VI.

On the other hand, from the increase of volume of a fibre by swelling we have the relation :

$$\frac{V_1 + V_2'' - (V_1 + v)}{V_1 + v} = \frac{1 + \frac{V_2''}{V_1} - \left(1 + \frac{v}{V_1}\right)}{1 + \frac{v}{V_1}} = s. \quad (4)$$

Table VI

Values of  $\frac{v'}{V_1}$ 

	Wool	Silk (Fibroin)	Reworked silk	Ramie	Cotton wool	Viscose rayon
Water	0.008	—	—	0.009	0.023	0.000
Methyl alcohol	0.000	0.000	0.000	0.000	0.000	0.000
Ethyl alcohol	0.003	0.027	0.022	0.022	0.003	0.022
n. Propyl alcohol	0.027	0.072	0.058	0.024	0.028	0.022
n. Butyl alcohol	0.031	0.073	0.054	0.028	0.028	0.032
n. Amyl alcohol	0.032	0.072	0.053	0.030	0.025	0.041
Liquid paraffin	0.067	0.088	0.084	0.072	0.053	0.060

By substituting to  $s$  and  $\frac{v'}{V_1}$  the values given in Table IV and those obtained from equation (2), we get the values of  $\frac{V_2''}{V_1}$  as given in Table V. In calculating thus the values of  $\frac{V_2''}{V_1}$ , we have assumed that the molecules of liquid paraffin cannot intrude into the intermicellar space of a fibre entirely. But this may not be so actually, and we can of course imagine the presence of still wider intermicellar spaces into which the molecules of the liquid paraffin can penetrate with ease. Consequently the values of  $\frac{V_2''}{V_1}$  calculated in the above ought to be regarded as a minimum estimate.

Next the value of  $\frac{V_2''}{V_1}$  can be checked by using the following relation :

$$\frac{V_2'' - v'}{V_1 D} = x, \text{ i. e., } \frac{V_2''}{V_1} = xD + \frac{v'}{V_1}, \quad (5)$$

where  $V_1$ ,  $V_2''$ ,  $v'$  and  $D$  denote the same meanings as before, and  $x$  the value of weight per-cent of the water adsorbed by a fibre in reference to the perfectly dried state when the fibre is put for 49 days in an enclosed space saturated with water vapour.

By putting in the equation (5), the value of the density of a fibre measured in methyl alcohol as  $D$ , the value of the water content  $x$  of a fibre which was measured by the writer and  $\frac{v'}{V_1}$  as is given in Table VI for the case of water, we get the values of  $\frac{V_2''}{V_1}$  as shown in Table VII. For the sake of comparison the values of  $\frac{V_2''}{V_1}$  obtained from the expansion by swelling are tabulated in the last horizontal row of the table. Here it must be noticed that silk and reworked silk are omitted in the table as they are considered to swell in an intramicellar manner too. As is evident from Table VII, the values of  $\frac{V_2''}{V_1}$  obtained by two different methods may be considered to be in rather good agreement, for such sort of experiments, though the discrepancy is a little more noticeable with viscose rayon than with the others. It has been stated before that the values of  $\frac{V_2''}{V_1}$  obtained

Table VII

	Wool	Ramie	Cotton wool	Viscose rayon
$D$	1.410	1.629	1.641	1.614
$x$ in weight per-cent	33.2	32	32	48.3
$Dx$	0.468	0.521	0.525	0.778
$\frac{v'}{V_1}$	0.008	0.009	0.023	0.000
$\frac{V_2''}{V_1} (= Dx + \frac{v'}{V_1})$	0.48	0.53	0.55	0.78
$\frac{V_2''}{V_1}$ from Table V	0.513	0.523	0.506	0.981

from the expansion by swelling are a minimum estimate. But considering the fact that such minimum values agree respectively with those obtained from the increase of weight by swelling, the actual values of  $\frac{V_2''}{V_1}$  seem to be well expressed by such a minimum estimate.

Next the writer examined how the lateral swellings of wool, silk, reworked silk, ramie, cotton wool, and viscose rayon in water proceed with time. The results are tabulated in Table VIII. The figures in Table VIII give the percentage of lateral swelling in reference to the diameter of the fibre in liquid paraffin, by considering the diameter of the fibre in it as approximately equal to that in vacuum, namely that of perfectly dried state.

Table VIII

Lateral swelling of fibre in % in reference to perfectly dry state

Time of immersion	Wool	Silk	Reworked silk	Ramie	Cotton wool	Viscose rayon
Within 1 hour	19.8	21.0	12.0	20.3	17.8	25.3
1 day	20.1	23.9	19.1	19.7	19.1	35.6
2 days	18.8	25.5	46.6	19.2	19.5	41.7
3 "	—	25.1	52.4	21.1	18.0	46.7
4 "	20.2	25.5	56.9	—	—	50.3
5 "	—	—	56.2	21.5	18.0	51.1
6 "	19.7	25.3	56.2	—	—	50.7

As seen in Table VIII, the lateral swellings in water of wool, silk, ramie and cotton wool attain their maximum values immediately after the immersion. On the other hand, the lateral swellings of reworked

silk and viscose rayon increase gradually as the time goes on, and become very great after several days. This fact seems to suggest that the micellar structure of these artificial fibres is looser than that of natural fibres.

Next by assuming that viscose rayon is reduced to hydratecellulose as  $2C_{12}H_{19}O_9OH$  and by taking the dimension of its unit cell which is determined by W. T. Astbury and T. C. Marwick<sup>1</sup> as:

$$a=8.14 \text{ A. U.}, b=10.3 \text{ A. U.}, c=9.14 \text{ A. U.}, \beta=62^\circ,$$

the volume of a unit cell is equal to:

$$V=abc\sin\beta=676.7 \times 10^{-24} \text{ c.c.}$$

Then the theoretical density  $\rho_0$  is equal to:

$$\rho_0 = \frac{4 \times 162 \times 1.649 \times 10^{-24}}{676.7 \times 10^{-24}} = 1.574.$$

The value of the density of the hydratecellulose as calculated above is much smaller than 1.614, the value of the net density of viscose rayon measured by the writer in water and in methyl alcohol. The net densities of wool, ramie and cotton wool which were measured in methyl alcohol are respectively greater too than their theoretical value calculated from their crystal structures determined by the X-ray diffraction, as is shown in Table IX.

As to the cause of such discrepancy between the densities obtained from the X-ray data and from the measurement in methyl alcohol, the presence of some impurities is of course conceivable. But at the same time reexamination of the crystal structures of such fibres seems to be also desirable.

Table IX

	<i>a</i>	<i>b</i>	<i>c</i>	$\beta$	Theoretical density	Observed density in methyl alcohol
Wool <sup>2</sup> (Residue)	3.38	4.65	9.8	—	1.231	1.410
Cellulose <sup>3</sup>	8.22	10.33	7.84	84°	1.609	1.627 (ramie) 1.641 (cotton)
	8.3	10.3	7.9	84°	1.571	
Viscose rayon <sup>4</sup>	8.14	10.3	9.14	62°	1.574	1.614

1. W. T. Astbury & T. C. Marwick: *Nature*, **127**, 12 (1931).
2. W. T. Astbury & H. J. Woods: *Phil. Trans. Roy. Soc. A* **232**, 333 (1933).
3. U. Yoshida & C. Park: *These Memoirs*, **17**, 443 (1934);  
K. H. Meyer & H. Mark: *Ber. D. chem. Gesells.*, **61**, 593 (1928);  
" " *Z. S. physik. Chem.*, **2B**, 115 (1929).
4. W. T. Astbury & T. C. Marwick: *Nature*, **127**, 12 (1931).

### Summary

The main results obtained from the present experiment are summarized below :

(1) The densities of various fibres measured in methyl alcohol are greater than those measured in water, in the other kinds of alcohols and in liquid paraffin. Therefore the net density of fibrous substances which consist of micellar structure must be measured in methyl alcohol.

(2) The difference of the densities of a fibre measured in various primary normal alcohols and in liquid paraffin reveals to some extent the variation of the widths of its intermicellar spaces.

In conclusion the writer wishes to express his sincere gratitude to Prof. U. Yoshida for invaluable suggestions he has given to the present research.

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