Chemical and Physical Nature of Insolubles: Acetone Solutions of Cellulose Acetate*

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INTRODUCTION

The relationship of haze and filterability to the gel content of cellulose acetate solutions is well known, yet relatively little is known about the gels themselves.

Investigation of the chemical composition of the gels has shown[1,2] that more than one fraction of gels can be obtained, and that hemicelluloses, principally xylan and mannan, are associated with the gels.

It was, therefore, decided to carry out an investigation of the chemical and physical properties of these gels with special reference to light-scattering and electron microscopy. The results are related to filterability of the parent acetates. The present work is concerned with gel size and distribution, as well as chemical nature.

EXPERIMENTAL PROCEDURES

Samples

The sources of the cellulose acetates used (*DS* = 2.5) are shown in Table I.

TABLE I
Sample Sources

Source	Sample designation
Wood pulp	
Sulfate process	Acetate A
Sulfite process I	Acetate B
Sulfite process II	Acetate C
Linters I	Acetate D
Linters II	Acetate E

Purification and Fractionation

Three procedures were used for the isolation of the gels from the soluble cellulose acetate; (*1*) centrifugation of 12% solution of cellulose acetate in acetone–water (361:7.3 by volume) mixtures at 21,500 *g* for 30 min.; (*2*) filtration of 1% cellulose acetate solutions in dry acetone through medium (15 micron) sintered glass filters and separation of the remaining gels by centrifugation at 30,000 *g* for 30 min. (see Fig. 1); (*3*) use of dialysis techniques to free the gels from the soluble cellulose acetate.

In the case of procedures (*1*) and (*2*), the gels were carefully washed with acetone and prepared for chemical and microscopic studies. The gels prepared by procedure (*2*) were redispersed in fresh acetone with the aid of a Waring Blendor. Centrifugation and redispersion were repeated three times to remove soluble material. Suitable concentrations of the pure gels were prepared for light-scattering and electron micrograph studies. Separate purified fractions were prepared for carbohydrate and x-ray analyses.

The dialyzed gels were filtered through a 15-micron filter to remove the large gels, and the solutions diluted for light-scattering measurements and electron microscopy. Gels under 15 microns in size (small gels) are commonly known as "haze."

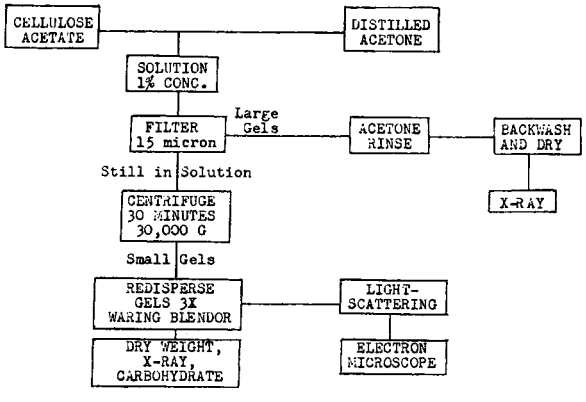

Fig. 1. Fractionation and purification procedure.

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Chemical Analysis

Carbohydrate analyses were performed by standard chromatographic techniques.[3] The results were compared to x-ray diffraction patterns obtained on a Norelco unit. The resulting patterns were compared to x-ray patterns obtained from purified standard samples of cellulose I, xylan, and cellulose triacetate.

Light Scattering

Light-scattering techniques can be used to measure both the size and particle molecular weight of the gels. Measurements were made at 436 and 546 mμ at several angles between 20° and 135° for the various concentrations with the use of a Brice-Phoenix Model 1000D light-scattering machine.[4] The cylindrical cells used (C-101 and C-105), were lined up and calibrated with Debye's Cornell polystyrene, the excess turbidity (τ) and dissymmetry (I_{45}/I_{135}) of the sample (0.5% polystyrene in toluene) being assumed to be 3.51×10^{-3} cm.$^{-1}$ and 1.17 ± 0.01, respectively, at 436 mμ.

Values of dn/dc were obtained on a Brice-Phoenix differential refractometer for soluble cellulose acetate–acetone at 436 mμ. The value obtained was 0.137, and the corresponding value of the light-scattering constant H was 5.29×10^{-6}. The calculated value of H for 546 mμ radiation was 2.14×10^{-6}. The value for dn/dc (and hence H) was assumed to be the same for the soluble and insoluble material.

Formulation of the Light-Scattering Equations

Both the length and the particle molecular weight determined for the gels considered in this report are *number-averages*.[5,6] This is in contrast to the usual weight-average molecular weight and Z-average length usually determined. This is because of the relatively large size of the gel particles as compared to quantities usually obtained by light-scattering techniques.

For small particles (less than $^1/_2$ the wavelength of the light), eq. (1) holds

$$H(c/\tau) = 1/\bar{M}_w + 2A_2c + \ldots \quad (1)$$

holds, $\bar{M}_w$ being the weight-average molecular weight and A_2 the second virial coefficient. However, for larger particles the number-average molecular weight $\bar{M}_n$ is determined by eq. (2):

$$H(c/\tau) = (1/2\bar{M}_n) + 2A_2c + \ldots \quad (2)$$

It should be noted that the particle molecular weight is expressed as if the gel were actually one molecule, i.e., the units of $\bar{M}_n$ are grams/mole.

In both cases the experimentally measured size is the radius of gyration. If a given shape is assumed, the characteristic dimension can be calculated. In this case it was thought that rods were the best approximation.

The equation used for the determination of the length of the rods is

$$L_n^2 = 6(\lambda')^2 \text{ (slope)}/8\pi^2 \text{ (intercept)} \quad (3)$$

where λ' is the wavelength of the light in the solution and the slope and intercept refer to the line obtained by plotting $H(c/\tau)$ against $\sin^2(\theta/2)$.

The equivalent sphere diameter can be obtained from the relation

$$D_n = 0.745L_n \quad (3a)$$

The scattering from spheres follows Debye's well-known relationship, where the intensity I varies with the angle θ and wavelength λ in the following manner:

$$I \propto [(3/x^3)(\sin x - x \cos x)]^2 \quad (4)$$

where

$$x = (4\pi n/\lambda) \sin(\theta/2) \quad (5)$$

n being the refractive index of the dispersing medium.

If the spheres are nearly monodisperse, and the difference in refractive index between the spheres and dispersing fluid is small, then $H(c/\tau)$ will have a maximum $I \propto (1/\tau)$ which depends on the size of the sphere.[7]

By setting $I = 0$ in eq. (4), Debye derived the following approximate equation governing the position of this minimum in I:

$$\lambda'/a = {}^8/_3 \sin(\theta/2) \quad (6)$$

where a is the radius of the sphere and $\lambda' = \lambda/n$.

Electron Microscopy

The table model RCA-EMT-3 was used in the first half of the work. Samples for this machine were prepared by adding a large excess of benzene to the acetone dispersion and placing a drop of the mixture on a collodion film. The sample was dried and the remaining purified gels were shadowed with metallic chromium. Later experiments involved the more precise RCA-EMU-2 model, direct drying of the acetone dispersion on a carbon background and shadowing with platinum being used.

EXPERIMENTAL RESULTS

A. Centrifugation of 12% Cellulose Acetate Solutions

The initial approach involved an examination of the filter-clogging portion of the gels which were separated from 12% solutions of cellulose acetate in acetone by centrifuging at 21,500 G for 30 min. at 15°C. in a Lourdes Model LRP centrifuge. The effectiveness of the centrifuging operation in removing clog-producing gels was measured by comparing the filterability of the dopes before and after centrifuging. Readings on a haze meter indicated that only a small amount of the haze was removed by the centrifugation. Filtration data, quantities of gels isolated, and carbohydrate content of the gels were compared for acetates from three pulps in Table II.

TABLE II
Tabulation of Centrifugation Results for 12% Solutions

Acetate	Amt. filtered, lb. Uncentrifuged	Amt. filtered, lb. Centrifuged	Insolubles gross, %	Xylose in insolubles, %
A	20.4	250	0.059	6.8
C	44.0	260	0.020	0
D	7.0	90	1.13	30.0

Only glucose and xylan were found by carbohydrate analysis. X-ray diffraction patterns show the xylan to be crystalline, and evidently not acetylated, and also indicated the presence of cellulose I, silica, and cellulose triacetate

B. Application of Light-Scattering Techniques to the Identification of Small Gels

Although centrifugation techniques separated most of the filter clogging gels, other techniques were required to isolate and purify the small gels responsible for the haze in acetate solutions.

Wood Pulps: Acetate A (Sulfate)

Preliminary results of light-scattering measurements of the small gels in the presence of the dissolved cellulose acetate (passed through a 15-micron filter) indicated a particle molecular weight of 6.25×10^8 and a length of 0.38 micron (see Fig. 2). However, when these measurements were repeated on highly purified gels, the size had increased to 0.52 micron, while the molecular weight remained the same. Electron micrographs of the highly purified gels showed irregular or

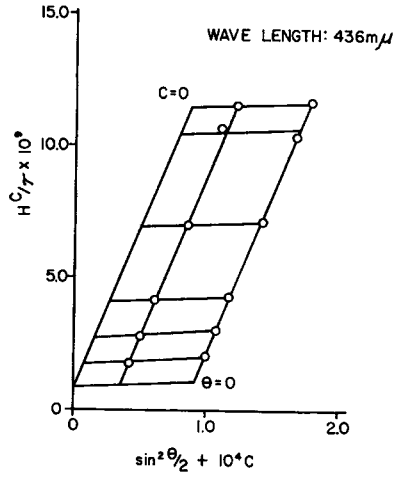

Fig. 2. Zimm plot of Acetate A gels in the presence of soluble acetate.

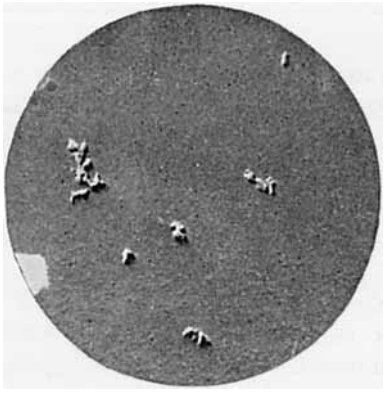

Fig. 3. Electron micrograph of Acetate A small gels (purified). 3750×.

potato-shaped particles of average size, 0.37 micron, with an extremely narrow size distribution, as shown in Figure 3.

However, when a microsample of these gels was prepared by passing the 1% solution through a 15-micron filter, then a 5-micron filter, and backwashing the 5-micron filter (a much gentler process), light-scattering measurements indicated a size or 0.41 micron, and electron micrographs showed elongated, monodisperse gels 0.25 micron in length. This suggested that the purification process mauled these gels somewhat, accounting for their apparent increased size. The differences observed between the light-scattering and electron micrograph results can be attributed to swelling in acetone.

X-ray analyses indicated that these gels contained cellulose I and xylan. The 53% of the sample that would hydrolyze contained 58.5% xylose, which accounted for about two-thirds of

the xylose in the acetate. The number of gels per gram of cellulose acetate was calculated to be 8.65×10^{12} gels. The small size of these gels makes the last result reasonable, and a volume computation shows that these gels would occupy only a fraction of 1% of the acetate solution, as expected.

The properties of these Acetate A (small) gels are summarized in Table III.

TABLE III
Properties of Acetate A Small Gels

Gels in acetate, %	0.90
Molecular weight, $\bar{M}_n$	6.25×10^8
Length, L_n, microns	0.38 micron
Composition	
Xylan, %	58.5
Cellulose I, %	41.5
Gels/g. acetate	8.65×10^{12}

Aggregation Properties. These gels were found to be aggregated in solution. The following investigations and calculations were made to explore this phenomenon.

(*1*) The intrinsic viscosity of the highly purified gels was determined and found to be 1.05. Even more interesting, however, is the negative slope of the plot of η_{sp}/c versus c (see Fig. 4). Usually this slope is positive or zero. A negative slope usually indicates a reversible type aggregation.

(*2*) When a sample of Acetate A was passed through successively a 15-micron filter and then a 5-micron filter, and the latter was backwashed, the observed gels were 0.41 micron in size. From the experiment 5–15 micron particles were expected. Kinetic experiments (by light-scattering techniques) showed that the larger expected particles

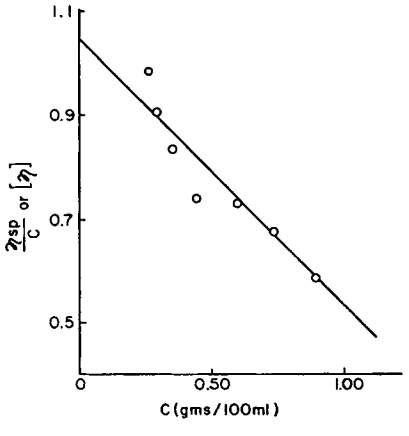

Fig. 4. Intrinsic viscosity of Acetate A gels.

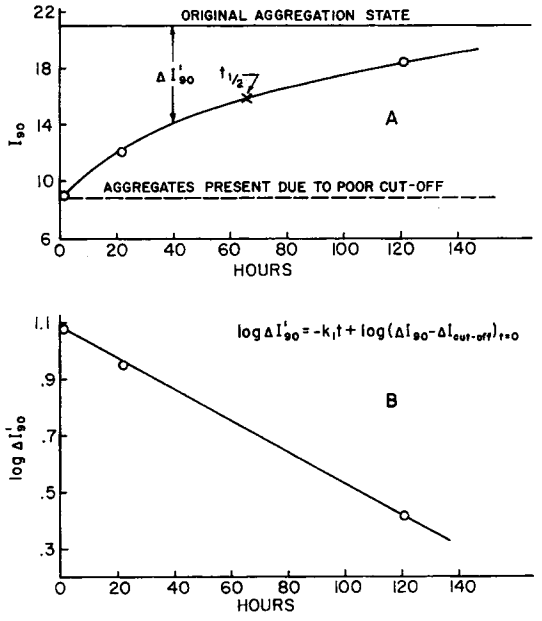

Fig. 5. Reaggregation of Acetate A gels: (*A*) increase in 90° scattering intensity with time; (*B*) application of the first-order rate equation.

were deaggregating completely in a few hours in this dilute solution.

(*3*) When the solutions in the preceding paragraph were allowed to stand for some time after being passed through the fine filter and the experiment repeated by again passing them through a fine filter, reaggregation was found to take place. In 1% cellulose acetate solutions, first-order kinetics were shown to hold, and the half-time of the reaggregation reaction was calculated to be 65 hr. This is illustrated in Figure 5, where the intensity at 90° (I_{90}) is plotted against time. Also shown in Figure 5 is the first-order reaction equation and its application to this problem.

(*4*) Numerical calculations according to the method of Flory[8] (see Appendix I) suggested that phase separation, as indicated by an infinite-sized aggregate, would take place at 41% cellulose acetate concentration. The highly purified gels exhibited phase separation at what would correspond to a 100% concentration of cellulose acetate. The concentrated phase when observed under a light microscope was found to have a stringlike structure.

(*5*) The aggregation theory also suggested that the rate of aggregation should be dependent on the rate of diffusion, which would depend on the concentration of cellulose acetate. This was confirmed by a filtration test (to be discussed

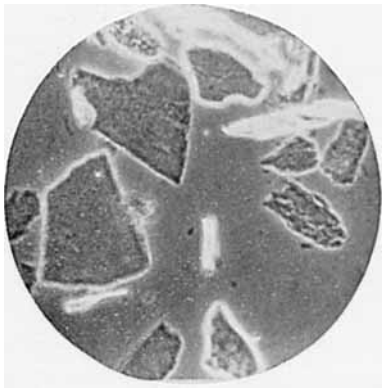

Fig. 6. Gels larger than 15 microns isolated from Acetate A. 156×.

below) where the amount filtered for a centrifuged 12% solution decreased from 1200 lb. to 269 lb. in two weeks.

Examination of the Large Gels. The gels stopped by the 15-micron filter were also investigated. X-rays showed the presence of cellulose I and cellulose triacetate. Light microscope studies showed rods and plates (see Fig. 6).

The plates were isolated and found by x-rays and infrared analyses to be cellulose I with a trace of triacetate. The rodlike structures are believed to be fragments of cellulose I fiber. (This is supported by results of an experiment with Acetate B in which the isolated fiberlike material was cellulose I.)

TABLE IV
Filtration Experiments on Acetate A

	Amt. filtered, lb.
Normal filtration value	16
Centrifuged filtration value	1200
Centrifuged, + small gels	144
Centrifuged, + large gels	33.5
Centrifuged, after standing 2 weeks	269

Filtration Properties. Filtration characteristics of both the small and the large gels in Acetate A were surveyed: 12% acetate solutions were prepared and centrifuged at 21,500 *g* for 30 min. To these centrifuged solutions were then added various quantities of gels. The results are shown in Table IV.

Reaggregation of the small gels may be responsible for the decrease in filterability in the last case. These aggregates are seen to be responsible for about one-third of the filtration difficulty. These data indicate that the preliminary experiment removed both the large gels and the existing aggregates of small gels.

Acetate B (Sulfite)

The molecular weight of the small gels was found to be 3.84×10^8, both in the presence of cellulose

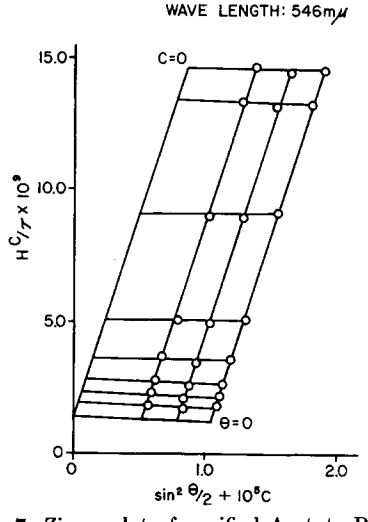

Fig. 7. Zimm plot of purified Acetate B gels.

TABLE V
Gels Purified by Centrifuging (Under 15 Microns)

Acetate	Composition by x-ray	Composition: Glucose, %	Composition: Xylose, %	L_n, microns	$\bar{M}_n$	Acetate, wt.-%	Appearance
A	Cellulose I + xylan	41.5	58.5	0.52 0.38[a]	6.25×10^5	0.90	Monodisperse, elongated
B	Cellulose I + xylan	70	30	0.38 0.30[a]	3.84×10^8	0.52	Monodisperse, slightly elongated
D	Cellulose I + cellulose triacetate	—		0.92	2.00×10^9	0.19	Spheres and spheres interconnected by fibrous material
E	Cellulose I + cellulose triacetate	90.1	9.9	1.60	3.34×10^9	0.10	Spheres and spheres interconnected by fibrous material

[a]Best value for original gels.

acetate and in the purified state. In the former case, the size was estimated at 0.30 micron, while in the purified state their size was found to be 0.38 micron. A Zimm plot of the latter data is given in Figure 7.

Electron micrographs were also prepared of the highly purified gels. They were nearly monodisperse, 0.22 micron in diameter, and irregular in shape. However, calculations from light-scattering data, assuming the particles to be cylindrical in shape, indicated that they have a somewhat elongated structure in the original state. These results are summarized in Table V.

Both Acetate A and Acetate B contain the same number of gels, the most significant differences being in size and xylan content; the latter value was 30% for Acetate B gels and for the Acetate A gels about twice as large.

The composition of the gels over 15 microns in size was the same as for the Acetate A gels (see Table VI).

TABLE VI
Gels Prepared by Filtration (Larger Than 15 Microns)

Acetate	Appearance	Composition by x-ray	Acetate, wt.-%
A	Fibers and plates	Cellulose I + cellulose triacetate	0.019
	(Plates isolated)	Cellulose triacetate, trace of Cellulose I	
B	Fibers and plates	Cellulose I + cellulose triacetate	0.036
	(Plates isolated)	Cellulose triacetate	—
	(Fibers isolated)	Cellulose I + cellulose triacetate	—
D	Fibers	Amorphous	0.043
E	Fibers	Cellulose I	0.01

The filtration value for Acetate B was found to be 42.6 lb. This is especially interesting when compared to the quantity of xylan isolated from the two wood acetates.

Acetate D and E (Linters)

Gels from both linters acetates were found to give unreliable light-scattering results in the presence of the dissolved cellulose acetate, probably due to the very small amounts of gel (haze) present. Upon purification, however, reliable light-scattering and electron micrograph studies could be made on the gels.

Light-scattering measurements on purified Acetate D small gels resulted in particle molecular

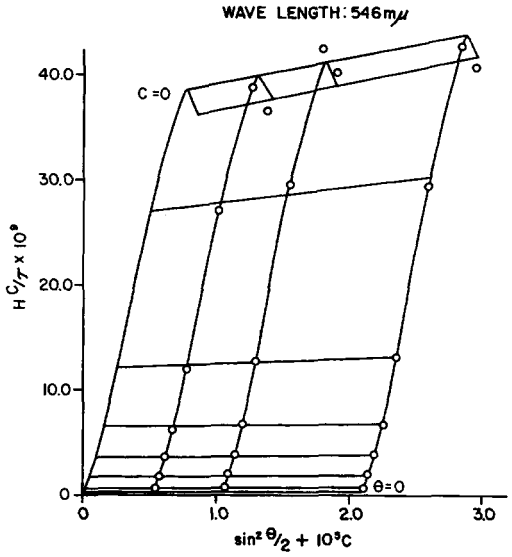

Fig. 8. Zimm plot of Acetate E small gels.

weights of 2.00×10^9 and lengths of 0.916 micron, as shown in Table V.

The intercept values obtained from the Zimm plot indicated that Acetate E small gels were larger than Acetate D small gels, the former having a molecular weight of 3.34×10^9 (see Fig. 8 and Table V).

In Figure 8 a maximum is observed at about 120° which represents an equivalent sphere diameter of about 0.34 micron, as calculated from eq. (6). The length of the particles as determined by eq. (3) was found to be 1.60 microns, which does not agree with the result obtained from the maximum. The intercept sphere diameter, as calculated by eq. (3a), is 1.2 microns.

This apparent discrepancy was cleared up by the electron micrographs of the dialyzed gels shown in Figure 9. Apparently, the intercept values measure an average of both the spheres

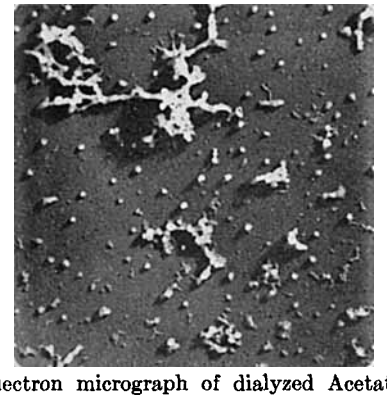

Fig. 9. Electron micrograph of dialyzed Acetate E gels. 9600×.

and the interconnected particles, while the spheres alone scatter enough of the light to cause the observed maximum. This phenomenon was also observed for the Acetate D gels, the maximum occurring at 135°, which corresponds to a sphere diameter of 0.33 micron. The number of these gels per gram of acetate was found to be 1.89×10^{11}.

Large Gels in Linters Acetates. The chemical analysis of the gels over 15 microns in size is shown in Table VI. These were fiberlike in appearance; however, Acetate D gels proved to be amorphous to x-rays, while acetate E gels gave an x-ray diagram indicating cellulose-I. In both cases these gels comprised only a small fraction of 1%.

C. Dialysis Purification of Small Gels (Acetates, A, B, D, and E)

The electron micrograph obtained from Acetate E is shown in Figure 9.

The predominant feature in the electron micrographs is uniform, monodisperse, spherical or potato-shaped gels about 0.1 micron in diameter. The smaller size noted in this experiment is probably the result of gentler handling in purification by dialysis.

Some differences among the gels were observable however; the wood acetate gels were slightly elongated, while those from the linters (especially Acetate E) were formed into almost perfect spheres. On the other hand, the spheres in the linters gels were sometimes connected into highly elongated structures by a semifibrous-appearing material, as illustrated in Figure 9.

The gels appear to be swollen to three times their dry volume in acetone. A calculation of the molecular weight of a sphere of 0.1 micron in diameter having a density of 1.3 g./cm.^{3} bears out this assumption. The resulting particle molecular weight, 4.1×10^8 g./mole, is very close to those obtained for wood acetate gels, which lack the interconnecting structures almost entirely.

Polydispersity calculations on the gels appearing in the electron micrographs, assuming a Gaussian distribution function, indicate that the ratio of the weight-average diameter to the number-average diameter (D_w/D_n) is less than 1.1.

Light-scattering measurements performed on the dialyzed gels gave good agreement with the wood acetate gels in the presence of dissolved cellulose acetate, and fair agreement with the purified linters small gels. However, loss of intensity indicates that some of the gels were lost in the dialysis and, therefore, molecular weight determinations, which depend upon accurate knowledge of the concentration, were unreliable.

A summary of all the results obtained on the gels appears in Tables V and VI.

Carbohydrate analyses were also carried out on the parent acetates. The results, shown in Table VII, made possible the calculation of the fraction of the total xylan present in the small gels.

TABLE VII
Carbohydrate Analyses of the Parent Acetates

Acetate	Glucose, %	Mannose, %	Xylose, %	Arabinose, % (?)
A	98.22	0.49	0.78	0.51
B	98.59	0.38	0.62	0.41
D	100.00	—	—	—
E	100.00	—	—	—

In the case of Acetate A, 67% of the xylan is in the small gels, as compared to 24% for Acetate B. From Table V we may compute that acetate E actually has about 0.01% xylan, which failed to show up in the carbohydrate analysis of the parent acetate.

CONCLUSIONS

A. Gels Under 15 Microns

(*1*) The small gels from all acetates appear to be uniform, nearly spherical, and about 0.1 micron in diameter They are sometimes interconnected to give larger structures. Maxima observed in light-scattering studies indicate spherical structures of about 0.33 micron. This difference may be due to swelling. The average length of the linters acetate gels was about 0.9 micron, and wood acetate gels were about 0.35 micron in length.

(*2*) The main difference between the Acetate A and the Acetate B gels seems to be in size and xylan content, as shown in Table VIII.

(*3*) Polydispersity calculations on both of these gels (from electron micrographs) indicate D_w/D_n is less than 1.1. There are only two likely mechanisms of formation which would result in gels of such a narrow distribution either (*a*) the gels are some well-defined part of the cellulose structure and hence would naturally all be the same size, or (*b*) they are all formed at the same time in the acetylation process and grow in size only by the addition of xylan and cellulose molecules precipitating from solution.

(*4*) Acetate A gels were found to be subject to aggregation in acetone solution. Preliminary re-

TABLE VIII
Comparison of Gels in Acetates A and B

Acetate	L_n, microns	$\bar{M}_n$	Acetate, wt.-%	Xylan, %	Gels/g. acetate
A	0.38	6.25×10^8	0.90	58.5	8.65×10^{12}
B	0.30	3.84×10^8	0.52	30	8.14×10^{12}

sults indicate that these aggregates definitely affect filtration adversely.

B. Gels Over 15 Microns

(*1*) For the wood acetates, fibers and plates were identified: the fibers are cellulose I, and the plates are cellulose triacetate.

(*2*) In the case of the linters acetates, fibers were found in both cases. Acetate E fibers showed cellulose I, but Acetate D fibers are amorphous.

(*3*) The large gels were found to be the predominant cause of filter plugging; however, it was shown that the small gels in their aggregated form contribute to this problem.

APPENDIX. CRITICAL CONCENTRATION OF ACETATE A SMALL GELS

The system under consideration has n_1 spherical solvent molecules and n_2 rigid, rodlike solute particles which are x times as long as the solvent molecules and have molecular weight M; v_1 and v_2 are the volume fractions of these particles, respectively, and xv_s is the molar volume of a solute molecule. The principle equations[8] are:

$$A_2 = [H(c/\tau) - (1/M)]/2c \qquad (2a)$$

$$A_2 = x(xv_s)/M^2 \qquad (7)$$

$$v_2 \text{ (critical)} = 8/x \qquad (8)$$

The value for A_2 as determined by eq. (2) is 3.38×10^{-6}. Here, $M = 6.25 \times 10^8$, and xv_s 6.15×10^8 ml./mole (a density of 1.3 g./ml. for the gels being assumed). The results in $x = 2.14 \times 10^3$ and v_2 (critical) = 0.37% gels. If the cellulose acetate contains 0.90% gels, the critical concentration of corresponding cellulose acetate is 41%.

The experimental value for the highly purified gels is near 100%, cellulose acetate basis. The difference can be attributed to the fact that the gels are not rods, but potato-shaped.

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Synopsis

An examination of the chemical and physical properties of gels in wood and linters cellulose acetates was carried out with special emphasis on light-scattering and electron microscopy. Gels were fractionated by filtration, centrifugation, and dialysis techniques to produce gels over and under 15 microns in size. The predominant phenomena observed in the small gels are nearly monodisperse spherical or potato-shaped gels of about 0.3 micron in dispersion (light-scattering) and 0.1 micron dry (electron microscopy). Chemical analysis shows these gels much enriched in xylan. The large gels were examined also and were found to consist of fibers (cellulose I) and plates (cellulose triacetate).

Résumé

On a fait une étude des propriétés chimiques et physiques des gels des acétates de cellulose de bois et de linters portant principalement sur la diffusion lumineuse et la microscopie électronique. On a fractionné les gels par filtration, centrifugation et dialyse afin de produire des gels de taille supérieure et inférieure à 15 μ. Le phénomène prédominant observé dans les petits gels est l'apparition de gels de forme sphérique on en forme de pomme de terre, étroitement monodispersés, de taille égale environ à 0,3 μ en dispersion (diffusion lumineuse) et 0,1 μ à sec (microscopie électronique). Une analyse chimique montre que ces gels sont plus riches en xylane. On a aussi examiné les grands gels et on a trouvé c'étaient des fibres (cellulose I) et des plaques (triacétate de cellulose).

Zusammenfassung

Eine Untersuchung der chemischen und physikalischen Eigenschaften des Gelanteils in Celluloseacetaten aus Holz und Linters wurde mit besonderer Heranziehung der Lichtstreuung und Elektronenmikroskopie durchgeführt. Die

Gelteilchen wurden durch Filtrations-, Zentrifugierungs- und Dialyseverfahren in solche mit einer Grösse über und unter 15 μ fraktioniert. Bei den kleinen Gelteilchen wurden überwiegend nahezu monodisperse, kugel- oder kartoffelförmige Gele von 0,3 μ im dispergierten (Lichtstreuung) und 0,1 μ im trockenen (Elektronenmikroskopie) Zustand beobachtet. Die chemische Analyse ergab eine starke Anreicherung dieser Gele an Xylan. Die grossen Gele wurden ebenfalls untersucht und erwiesen sich als Fasern (Cellulose-I) und Plättchen (Cellulosetriacetat).

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