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Number and Size Distribution of Particles in Cellulosic Solutions*

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INTRODUCTION

The efficiency of dissolving procedures for cellulose derivatives is a very important economic factor, since well over 2 million tons of dissolving grade cellulose are processed by industry each year. In order to make best use of the raw material it is necessary that the dissolving procedure be as efficient as possible; however, cellulose is not an easy material to dissolve. It is a highly crystalline polymer and, like most natural products, is nonhomogeneous. One must overcome both chemical and physical problems to approach true solutions of commercial cellulose pulps. The two most important soluble derivatives of cellulose are cellulose xanthate and cellulose acetate. Cellulose xanthate is dissolved in dilute sodium hydroxide to form viscose, which is extruded and regenerated as fiber (rayon) and film (cellophane), and the cellulose acetate (secondary) is usually dissolved in aqueous acetone for extrusion as fiber or film. Expensive filtration equipment and procedures are used to remove particles several microns in diameter from the solution to improve their processability and the product obtained. In the work reported here, a commercially available counter (Coulter counter, Coulter Industrial Sales, Elmhurst, Ill.) which operates on electronic principles, has been applied with considerable success to determining the size and size distribution of particles in cellulose solutions.

A large amount of work has been reported in the literature on the influence of insoluble materials on the viscose processes. The detrimental effect of insolubles on filtration has been the subject of the majority of the research in the past,[1-3] but some efforts have included studies of the effect of insolubles on yarn strength.[4] A full discussion of the literature in this field would require a prohibitive amount of space, but leading references can be found in the recent paper by Treiber.[5] A wide range of physicochemical techniques has been applied to the problem with varying degrees of success. Counting of insoluble particles has been mainly done by microscopic methods[6] or equipment utilizing the breaking of a light beam and photoelectric detection.[5]

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EXPERIMENTAL

Experimental Counting Method

Berg[7] has published a rather complete description of the instrument used in this work. The description here will be limited to its applicability to cellulosic materials. The instrument can be used to determine the number and size of particles in an electrically conductive liquid by forcing the suspension to flow through a small aperture having an immersion electrode on each side. As each particle passes through the aperture it replaces its own volume of electrolyte within the aperture, momentarily changing the resistance value between the electrodes. This produces a voltage pulse of short duration having a magnitude proportional to particle volume. The voltage pulses are amplified and fed to a threshold circuit having an adjustable screen-out level, and if the level is reached or exceeded by a pulse, the pulse is counted. By taking a series of counts at various threshold settings, data are directly obtained for plotting cumulative particle frequency vs. particle volume. The instrument is calibrated against ragweed pollen, which has a fairly uniform size of 20 μ, and other pollens.

A schematic diagram of the instrument is shown in Figure 1. When the stopcock is opened, a controlled external vacuum initiates flow from the beaker through the aperture and unbalances the mercury manometer. Closing the stopcock then isolates the system from the external vacuum, and the siphoning action of the rebalancing mercury column continues the sample flow. The advancing mercury column activates the counter via start and stop probes, thus providing a constant suspen-

sion volume for all counts. A volume of 2 cc. per count was satisfactory for cellulose work with a concentration of 1% cellulose acetate or viscose.

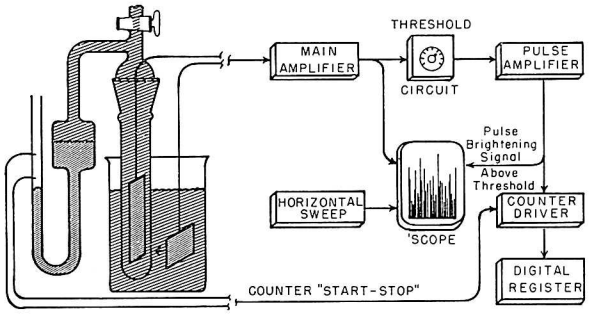

Fig. 1. Schematic diagram of the Coulter counter.

A 100-μ aperture tube, used successfully for both viscose and cellulose acetate, permits counting of particles 4–40 μ in diameter.

Solvent Systems

Particles in viscose can be counted readily in a solution prepared by adding an additional amount of dissolving caustic. The aqueous sodium hydroxide (approximately 6%) serves as an excellent electrolyte system. Normal viscose viscosity is too high for direct counting. Dilutions of 1:3, 1:5, and 1:10 gave the same counts when the dilution factors were considered. The 1:10 dilution has been used for this work since its low viscosity permits fairly rapid counting. Approximately 30 min. is required per sample.

Experimental work with organic soluble material, such as cellulose acetate, has proven more difficult. It is necessary to add a considerable amount of water and strong electrolyte to an organic solvent to reduce solvent resistance to a workable level. Such solutions do not easily dissolve organic soluble materials such as cellulose acetate. Most of the work on cellulose acetate has been done in 80:20 acetone:water with 6% sulfuric acid. There was some success with 90:10 acetone:water containing 4 wt.-% potassium thiocyanate. At high aperture current settings some electrolysis occurs, a white precipitate being formed in this solution. Unsuccessful attempts were made (*1*) to add dimethylformamide in place of the acetone; (*2*) substitute acetic acid for acetone; (*3*) substitute sodium iodide for potassium thiocyanate. HCl in acetone:water was somewhat successful, but the mixture corroded parts of the instrument badly.

RESULTS

Counting Particles in Viscose

The number of particles in viscose has been found to be a function of many variables, as would be expected. Some of these are pulp type, wood species, presence of surfactant, and the viscose preparation process. Day-to-day variations in viscose preparations are often reflected strongly in the number of counts. Typical data showing the effect of raw material species and pulping method are shown in Table I. Filtered and unfiltered counts were made on the same sample. It can be seen from these data that sulfite pulps from pine have fewer particles than sulfate pulps from the same wood, particularly in the larger size ranges; however, a lower percentage of the particles in the

TABLE I
Effect of Pulping Procedure and Species on Particle Counts in Viscose

Particle size range, μ	No. of particles in various pulps[a]									
	A Unfiltered	A Filtered	B Unfiltered	B Filtered	C Unfiltered	C Filtered	D Unfiltered	D Filtered	E Unfiltered	E Filtered
4.5–6.1	508	554	906	296	408	394	597	539	556	571
6.1–6.8	340	325	368	458	207	223	323	287	376	386
6.8–8.6	196	214	287	318	121	97	227	164	335	288
8.6–10.5	144	97	192	225	65	46	111	108	127	115
10.5–13.0	64	81	131	101	16	19	79	65	88	33
13.0–16.4	46	34	56	46	13	2	18	11	30	5
16.4–20.5	35	19	34	9	6	4	16	9	7	0
20.5–25.5	15	9	32	5	3	0	2	0	14	0
25.5–32.2	17	1	4	3	0	0	2	2	3	0
32.2–40.6	4	1	2	1	0	0	2	0	1	0
40.6–51.1	1	0	0	0	0	0	0	0	0	0

[a] Pulp A: prehydrolyzed sulfate southern pine; pulp B: prehydrolyzed sulfate southern pine; pulp C: sulfite Japanese red pine; pulp D: sulfite southern pine; pulp E: prehydrolyzed sulfate gum.

TABLE II
Particles in Viscose from Cellophane Wood Pulps and Cotton Linters

Particle size range, μ	No. of particles in various pulps[a]					
	A Unfiltered	A Filtered	B Unfiltered	B Filtered	C Unfiltered	C Filtered
4.5–6.1	17,441	16,904			2,258	2,441
6.1–6.8	6,065	5,728	58,552	51,781	1,207	1,888
6.8–8.6	1,935	1,583	21,645	19,264	1,063	1,343
8.6–10.5	535	554	5,496	4,105	678	802
10.5–13.0	220	187	1,299	895	504	586
13.0–16.4	88	52	330	198	432	369
16.4–20.5	14	16	103	64	328	130
20.5–25.5	20	2	47	23	140	12
25.5–32.2	8	2	25	6	27	3
32.2–40.6	2	0	11	2	4	0
40.6–51.1	0	0	7	0	1	0

[a] Pulp A: sulfite hemlock; pulp B: sulfite birch and maple; pulp C: cotton linters.

larger size ranges is removed by filtering the sulfite viscoses.

Examination of the data in Table I also shows a marked reduction of particles greater than 8 μ by the filter used (a cotton batting supported by unbleached domestic). The filter cutoff is not sharp, but no reduction in particles occurs to any extent in size ranges below 8 μ. The filtration process definitely does not remove all particles larger than 8 μ, but the efficiency of filtration improves with increased particle size. An increase in particles in the smaller size range, 4–6 μ, is often seen, as is shown by the sulfite textile viscose in Table I. This may be the result of washing out the filter, breaking up large particles, or both. The effect of wood species on particle size and size distribution is also illustrated by data in Table I. The sulfate hardwood pulp is seen to have particle sizes and size distributions similar to the sulfite viscoses shown in Table I, and the effect of filtration is similar.

An increase in number of large particles is generally correlated with a lowering of filtration, but not in every case. Table II contains data from rather low purity cellophane pulps which produce viscoses with particle counts several times as high as the purer pulps in Table I, but which filter in a very similar manner. Cotton linters viscose is also included in Table II. The count levels of the cotton linters viscose are approximately two times those of most wood pulp viscoses.

The presence of surfactant in the xanthating step[3] reduces the counts in viscose from sulfate softwood pulp to a level similar to that obtained with sulfite softwood and sulfate hardwood pulps. Surfactants used in the viscose process are usually anionics (for example, Naccenol) or nonionics (for example, the Plurionics). These data for unfiltered viscoses are found in Table III.

TABLE III
Reduction of Number of Particles in Viscose by Addition of A Surface-Active Agent to Pulp

Particle size range, μ	No. of particles in various pulps[a]			
	A Without surfactant	A With surfactant	B Without surfactant	B With surfactant
4.5–6.1	784	299	630	631
6.1–6.8	476	197	601	222
6.8–8.6	236	115	446	172
8.6–10.5	185	67	267	99
10.5–13.0	101	17	287	87
13.0–16.4	82	24	141	29
16.4–20.5	51	10	109	19
20.5–25.5	16	1	58	11
25.5–32.2	1	1	20	4
32.2–40.1	3	2	7	2
40.1–50.0	3	0	1	0

[a] Pulp A: prehydrolyzed sulfate southern pine (tire cord grade); pulp B: prehydrolyzed sulfate southern pine (textile yarn grade).

Evaluation of Filter Media for Viscose

The Coulter counter is an ideal tool for evaluating filter media. Results are shown in Table IV of filtering separate quantities of one standard viscose through Buckeye filter papers of different porosities, with the counts from the filtration for the regular cotton batting shown for comparison. The complete cutoff for the filter is at progressively smaller size ranges as the porosity decreases.

TABLE IV
Evaluation of Filter Media with Coulter Particle Counter

Particle size range, μ	No. of particles in filtered and unfiltered viscose[a]					
	Unfiltered standard	Regular filter pad + screen	Filter 1	Filter 2	Filter 3	Filter 4
4.1–6.1	1566	785	464	413	254	210
6.1–6.8	572	449	255	156	97	88
6.8–8.6	349	170	120	93	44	15
8.6–10.5	213	106	30	24	13	0
10.5–13.0	124	68	11	11	0	0
13.0–16.4	81	23	2	0	0	0
16.4–20.5	48	11	1	0	0	0
20.5–25.5	20	3	0	0	0	0
25.5–32.2	17	2	0	0	0	0
32.2–40.1	6	0	0	0	0	0
40.1–52.0	2	0	0	0	0	0

[a] Frazier air porosity of the filters: Filter 1 = 22.0 cu. ft./min./ft.²; Filter 2 = 20.0 cu. ft./min./ft.²; Filter 3 = 5.6 cu. ft./min./ft.²; Filter 4 = 3.0 cu. ft./min./ft.² at 0.5-in. differential across sheet.

having certain dimensions in a plane perpendicular to the direction of view. The Coulter counter counts the radii of particles expressed as equivalent cylinders. The data from which comparisons of the two methods can be made are also listed in Table V.

Cellulose Acetate Solutions

As mentioned in the section on solvents, a considerable quantity of water and electrolyte (sulfuric acid) had to be added to the acetone to supply a solution with low enough resistivity for counting. Typical results on filtered and unfiltered acetate solutions are shown in Table VI. The particle size cutoff by the filter is not quite so definite for acetate as for viscose particles and smaller size ranges are removed from the acetate solution. The filter medium is a cotton linters paper sheet with a Frazier air porosity of 14. Figure 2 illustrates another method of portraying Coulter counter data. The logarithm of number greater than the stated diameter is plotted against diameter, i.e., a cumulative number versus particle diameter plot.

TABLE V
Comparison of Coulter Counts and Microscopic Counts on Pulp[a]

	Particle size range, μ		No. of particles, 34% CS_2				No. of particles, 30% CS_2			
			Unfiltered		Filtered		Unfiltered		Filtered	
	Microscopic	Coulter	Microscopic	Coulter	Microscopic	Coulter	Microscopic	Coulter	Microscopic	Coulter
Small particles	6.5–16.0	4.5–10.5	1,730	3,059	3,030	4,305	13,200	12,389	16,950	13,864
Large particles	16.0–32	10.5–50	650	850	1,080	483	2,810	3,587	3,900	2,780

[a] Pulp: 50:50, cotton linters and prehydrolyzed sulfate southern pine.

Effect of Carbon Disulfide Levels on Particle Counts

In Table V are data showing the effect of better viscose preparation conditions. The better method provides 34% carbon disulfide as compared with 30% for the less efficient procedure. The reduction in counts by addition of more carbon disulfide is evident. This observation is in agreement with Samuelson[2] and supports his contention that the particles are not intrinsic properties of the pulp, but can be reduced by further reaction.

Comparison of Coulter Counts and Microscopic Counts

Agreement between the two methods of counting is as good as could be expected, considering the fundamental differences in the two methods. The light microscopic technique counts the particles

TABLE VI
Filtered and Unfiltered Linters Acetates, Coulter Counts

Particle size range, μ	No. of particles			
	Cotton linters A		Cotton linters B	
	Unfiltered	Filtered	Unfiltered	Filtered
4–6	31,796	31,812	28,292	17,756
6–8	6,248	5,600	6,972	2,276
8–10	1,552	772	1,696	412
10–15	1,500	900	2,168	108
15–20	252	32	692	0
20–25	74	8	373	0
25–30	36	0	175	0
30–40	4	0	8	0
>40	4	4	12	0

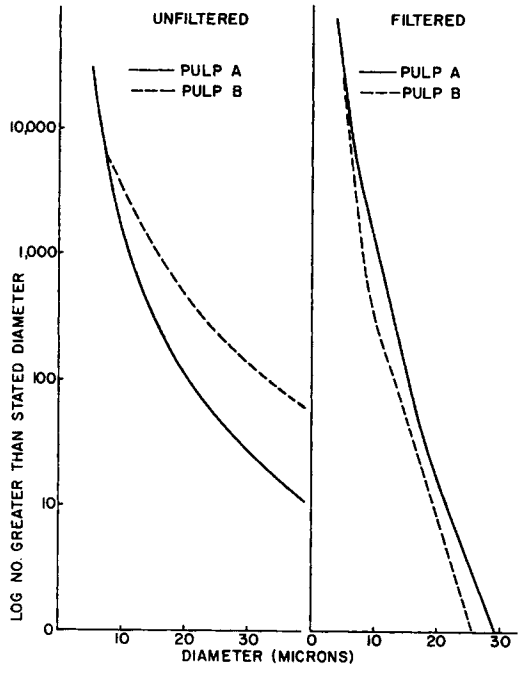

Fig. 2. Coulter counter data from cellulose acetate.

DISCUSSION

The Coulter particle counter can be used to count many different types of cellulosic particles ranging from cellulose derivatives to unreacted fibers. Particles found in cellulose acetate and viscose solutions in the micron range (greater than 4 μ) are mostly swollen pieces of fiber debris which have a much higher electrical resistance than the solutions in which they are found. Early fears that the particles might be extremely porous and very conductive were unfounded. The reasonable agreement of electronic counting with microscopic counting supports this observation. Removal of most of the material which counts in the Coulter counter by centrifuging and subsequent microscopic examination of the undissolved substances gives the strong impression that most of the material (greater than 4 μ) is solid and not a partially dissolved gel substance. The counting method used in this work would certainly be more responsive to solid materials, and the possibility that very low density gels of large diameter are present is not ruled out. These could be seen by microscopic viewing of flow through a capillary such as found in the Treiber equipment. Manley[9] reported a dispersion of particles separated from viscose in 1*N* NaOH gave a low value for intrinsic viscosity, indicating the particles are fairly compact. Marschall[3] reported the incompletely dissolved cellulose particles are the principal cause of poor filtration.

The Coulter counter technique can be used to study particles in the size range between the upper limit of light-scattering (ca. 2 μ) and the convenient lower limit of light microscopy (ca. 15 μ). Light microscopy is difficult on particles in cellulose solutions which are less than 15 μ in size because of the extremely large number of particles and the small difference in refractive index between the particles and the viscose solution. The 2–15 μ range is of real importance to viscose and acetate technology since: (1) large numbers of particles in this size range are caught by the filters; (2) coincident passage of particles of this size range can plug spinnerets; and (3) those particles which pass through into the yarn could provide potential weak spots and wedgelike areas (slugs) which may break adjacent fibers when the yarns are twisted. A further study of the correlation of yarn strength with particle size and size distribution in the viscose or in solutions of the yarns with the Coulter counter would be a logical extension of this work that should be of real interest to yarn manufacturers. The correlation of particle counts in the micron size range with other properties, such as spinnability, color, or clarity, of cellulose end products also remains to be done.

The Coulter counter is an excellent tool and the data serve to complement those obtained in filtration experiments. Both procedures suffer as a result of variations in viscose preparation, but the instrument has fewer sources of error than a conventional filter material (holes, etc.). Changes in particle counts at high levels can often be detected by the counter before any significant amount of filtration will occur.

The number of micron size particles in cellulose solution depends on many variables. The three basic ones studied in this work were: (1) pulp raw material, (2) type of pulp, and (3) the viscose preparation process. Cotton linters pulps and prehydrolyzed sulfate pine pulps contain more particles than prehydrolyzed sulfate hardwoods or sulfite pulps, but fewer particles are removed by the filter from the two latter types. For a given series of pine sulfate pulps, filtration behavior correlates well with the number of particles. Variation in particle counts from day to day make direct correlation of counts and filtration behavior difficult, but relative levels between pulps are usually maintained. It is not known if the micron size particles studied in this work are the direct

cause of filtration difficulties or are just indicators of the presence of materials which may plug the pores of the filters, such as gel substances or very small fiber particles. Centrifugation or prefiltration to remove a small fraction of the cellulose (less than 0.1%) containing much of this micron size fraction does greatly increase filtration.

The number of 4–50 μ particles in a cubic centimeter of viscose prepared from well purified pulp is usually less than 20,000. Sperling[10] and others[9] have shown by light-scattering that the particles a fraction of a micron in size are far more numerous than the larger particles in acetate and viscose solutions. The range covered by the procedure described here must comprise the extreme upper range of the distribution of particle sizes.

The importance of the types of particles in cellulosic solutions has been very apparent in these studies. The kinds of particles present must be decidedly different when the numbers in the size ranges removed by the filter in a viscose can vary as much as 20-fold, but have the same general effect on a filter. This study of number and size distribution of particles is the initial phase of the efforts in these laboratories to better characterize cellulose solution insolubles. Further work, some of which is in progress, will include an investigation of the intrinsic nature of the insolubles.

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Synopsis

A large amount of work has been reported in the literature on the influence of insoluble materials on the viscose process. Detrimental effects of insolubles on filtration has been the subject of the majority of the research in the past, but some efforts have included studies of the effect of insolubles on yarn strength. A wide range of physicochemical techniques have been applied to the problem with varying degrees of success. Counting of the insoluble particles has been mainly done by microscopic methods or equipment utilizing the breaking of a light beam and photoelectric detection. In the work reported herein, a commercially available particle counter (Coulter counter) which operates on electronic principles has been applied with considerable success to cellulosic solutions. The instrument permits determination of cellulosic particle sizes and size distributions in the size range of real interest to cellulose workers (4–50 μ) and over an even wider range with other accessories. Viscose solutions, diluted 1:10 with dissolving caustic, have been counted before and after filtration. The number of counts varies considerably with species, pulp type, processing conditions, and the presence or absence of surfactant. Filtration through a medium similar to that used in commercial viscose preparation causes a fairly sharp drop in the number of particles greater than 8 μ. The counts below 8 μ are not changed appreciably by filtration. The filter cutoff of cellulose acetate solutions (diluted to similar concentrations) is much less sharply defined. Acetate counting is done in aqueous acetone solution with sulfuric acid as electrolyte. Coulter counts parallel microscopic values, but are far less laborious, and more information about the size distributions is obtained. The instrument has also been used to evaluate filter media for both viscose and cellulose acetate filtration.

Résumé

Dans la littérature on consacre une grande quantité de travaux à l'influence de matière sinsolubles sur la préparation de la viscose. Les effects défavorables des matières insolubles sur la filtration ont constitué le sujet de la majorité des recherches dans le passé, mais quelques efforts ont porté sur l'étude de l'effet des matières insolubles sur la force de la fibre. Une grande quantité de techniques physicochimiques ont été appliquées à ce problème avec plus ou moins de succès. Le recensement des particules insolubles a été effectuée principalement par une méthode microscopique ou par un équipement utilisant la réfraction d'un rayon lumineux et sa détection photoélectrique. Dans ce travail, un compteur commercial de particules (compteur Coulter), basé sur des principles électroniques, a été appliqué avec grand succès à des solutions cellulosiques. Cet instrument permet la détermination des dimensions des particules cellulosiques, leur distribution dans le domaine de dimension présentant un intérêt réel pour les travailleurs de la cellulose, c'est à dire de 4 à 50 μ et dans un domaine plus large avec d'autres accessoires. Des solutions de viscose diluées au 1/10 avec de la lessive caustique ont été comptées avant et après filtration. Le nombre de particules comptées varie considérablement avec l'espèce, le type de pulpe, les conditions opératoires et la présence ou l'absence d'un réactif de surface. La filtration à travers un milieu semblable à celui qu'on utilise dans la préparation de la viscose occasionne une chute très prononcée du nombre des particules supérieures à 8 μ. Le nombre des particules inférieures à 8 μ n'est pas changé d'une façon appréciable par filtration. La chute du nombre de particules par filtration de solutions d'acétate de cellulose (diluées à des concentrations analogues) est beaucoup moins prononcée. Pour compter les particules dans l'acétate de cellulose on opère avec des solutions

dans l'acétone aqueuse avec H_2SO_4 comme électrolyte. Les résultats du compteur Coulter s'accordent avec les résultats trouvés par microscopie mais sont moins laborieux et on recueille plus d'information sur la distribution des tailles des particules. L'appareil a aussi servi à évaluer les milieux de filtration pour la viscose et l'acétate de cellulose.

Zusammenfassung

In der Literatur finden sich eine grosse Zahl von Arbeiten über den Einfluss von unlöslichem Material auf den Viskoseprozess. Nachteilige Einflüsse des Unlöslichen auf die Filtration waren in der Vergangenheit der Hauptgegenstand der Untersuchungen, aber in einigen Fällen wurde auch der Einfluss des Unlöslichen auf die Festigkeit des Garns untersucht. Ein weiter Bereich physiko-chemischer Methoden wurde mit wechselndem Erfolg auf das Problem angewendet. Die Zählung der unlöslichen Teilchen erfolgte hauptsächlich mit mikroskopischen Methoden oder mit einer Apparatur, die die Brechung eines Lichtstrahls und seine photoelektrische Registrierung benützt. In der vorliegenden Arbeit wurde ein handelsüblicher Teilchenzähler (Coulter Counter), der auf elektronischer Grundlage arbeitet, mit gutem Erfolg auf Celluloselösungen angewendet. Das Instrument erlaubt im Grössenbereich, der für Celluloseuntersuchungen wirklich interessant ist, nämlich 4–50 Mikron, eine Bestimmung der Grösse und Grössenverteilung von Celluloseteilchen und mit anderen Zusatzgeräten über einen noch grösseren Bereich. An Viskoselösungen, die mit kaustischem Lösungsmittel 1:10 verdünnt waren, wurden vor und nach der Filtration Zählungen durchgeführt. Die Zahl der Stösse hängt in beträchtlichem Ausmass von der Spezies, dem Pulptyp, den Verarbeitungsbedingungen und der An- oder Abwesenheit von oberflächenaktiven Substanzen ab. Filtration durch ein Medium, das dem bei der technischen Viskosedarstellung verwendeten ähnlich ist, führt zu einem ziemlich scharfen Rückgang der Zahl der Partikel, die grösser als 8 Mikron sind. Die Stösse unter 8 Mikron werden durch Filtration nicht sehr verändert. Der Filtereffekt ist bei Celluloseacetatlösungen (auf ähnliche Konzentrationen verdünnt) viel weniger scharf definiert. Die Zählung wird an Acetat in wässriger Acetonlösung mit Schwefelsäure als Elektrolyt durchgeführt. Die Zählung mit dem Coulter-Gerät stimmt mit den mikroskopischen Werten überein, ist jedoch bei weitem weniger mühsam und man erhält mehr Angaben über die Grössenverteilung. Das Instrument wurde auch zur Auswertung von Filtermedien sowohl für die Viskose- als auch Celluloseacetatfiltration verwendet.

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