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COMPARATIVE INVESTIGATIONS ON THE FILTRATION EFFICIENCY OF MEMBRANE FILTERS

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Preusser (1) conducted extensive electron-microscope studies with membrane filter surfaces. He found, among others, that neither the average pore diameter nor the diameter of the largest pores, by itself, is sufficient to define the filtration effectiveness. It therefore seemed a good idea to determine the quality of a larger number of different membrane filter types and products by investigating the filtrates of particle suspensions for comparison.

Material and Method

Filters Tested: Sartorius membrane filter MF 500, MF 250, MF 150, MF 125, MF 100, MF 50, MF 30, MF 15, MF 14, MF 12, MF 10 (diameter 50 mm); millipore filter SM, AA, HA, GS (diameter 47 mm); Gelman filter GA 4, GA 6 (diameter 47 mm). Altogether, at least 20 or 30, and in some cases considerably more filters of every grade and type were tested.

Test Particles: polystyrol latex, diameter 2.05 mmu, 1.90 mmu, 1.305 mmu, 1.171 mmu, 0.796 mmu, 0.557 mmu, 0.365 mmu (very kindly made available by the Dow Chemical Co, Midland, Michigan); solanyl green G from the Hoechst Dyestuff Factory; normal and inhibition forms of Serratia marcescens, strain 153 from the Gottingen University's Institute of Microbiology.

Preparation of Latex Suspensions: Each case, one drop of latex parent suspension was diluted with 50 ml of a 4% NaCl solution ([1] an extensively particle-free NaCl solution, filtered through Sartorius membrane filter MF 10, was used in all experiments). In a few cases, 2 or 3 drops of the parent suspension had to be used.

Preparation of Colanyl Green Suspension: A glass rod, at whose end a small quantity of Colanyl Green dough had been glued, was dipped in 50 ml of 4% NaCl solution until the developing suspension became nontransparent.

Preparation of Bacteria Suspensions:

(a) Normal form: after at least 8 days of cultivation at 26°-28° C in a peptone-glucose medium (peptone 0.5%, glucose 0.5%, NaCl 0.3%, $FeSO_4 \cdot 7 H_2O$ trace, pH 7.0-7.2; placed in 300-ml Erlenmeyer flasks in 100 ml increments) 1 ml of bacteria suspension (containing about $1-3 \cdot 10^8$ cells capable of multiplication) was diluted, in each experiment, with 49 ml of 4% NaCl solution.

(b) Inhibition [Inhibited] forms: Normal cells of the test bacterium were incubated at 26°-28° C in 50 ml of nutrient solution (100-ml Erlenmeyer flasks) with the following composition:

$Na(NH_4)HPO_4 \cdot 4 H_2O$ 0.15%, KH_2PO_4 0.05%, K_2HPO_4 0.05%, $MgSO_4 \cdot 7 H_2O$ 0.05%, Asparagin 0.5%, glucose 1%; pH about 7.2. After 5-8 days, the entire matured bacteria population was then used for inoculation of 450 ml of a nutrient solution which, in addition to the abovementioned components, contained 14.84 g LiCl. As a result of the rather moderate formation of precipitate, the LiCl concentration of the over-all solution was not quite 0.7 molar after inoculation. Incubation took place in upright 1-liter Roux flasks at 20°-21° C. After as little as 24 hours, it was possible to use 1 ml samples, diluted with 49 ml of a 4% NaCl solution, for the testing of the filters. After 10 days of cultivation, the LiCl-containing medium contained a large number of bacteria cells capable of multiplying but some of them by then had already lost their capability of forming dyestuff. According to optical and electron microscope investigations, the bacteria inhibition forms used for filter testing were on the average somewhat shorter and above all they were definitely slimmer than the "normal forms" cultivated in the peptone-glucose medium.

Filtration of Particle Suspensions: Increments of 50 ml of particle suspension were suctioned into extensively dust-free, previously carefully cleaned beakers, through the filters to be tested. The filters were clamped in "Coli 5" instruments from the Sartorius Membrane Filter Company, Inc, Gottingen. The metal fritts, which are provided as filter supports or bases in this apparatus, could be cleansed of particles only with great difficulty; this is why an easily cleaned perforated plate was used as filter support in the filtration of latex and Colanyl Green. For microbiological investigations, the filtration instruments were sterilized in autoclaves.

Particle Size Measurement: The magnitude spectra of the particles contained in the filtrates were determined with the help of a Coulter Counter, Model B (with recorder); (here we have the following dimensions: scale diameter 30 mm, 1/aperture current = 1/4 + fine motion = 100; 1/amplification = 1, volume per individual measurement = 0.05 ml, calibration of apparatus with latex particles having a diameter of 1.305 and 1.173 mm).

Cultivation Method for Establishment of Bacteria in Filtrate: The filtrates to be tested were filtered through sterile Sartorius membrane filter MF 30 and were incubated at 26°-28° C after placing on agar plates (peptone-glucose medium with addition of 1.5% agar). These experiments were always conducted parallel to the corresponding Coulter Counter measurements. There were no contradictory results in any case here.

Results and Discussion

The results of the investigations can essentially be seen in Tables 1 and 2.

Table 1 gives us a general picture of the filtration efficiency of the filters tested. Using the available test particles, it was possible to arrange almost all filter types according to their qualitative nature. Only the membrane filters MF 10 and MF 12 did not allow any type of particle to pass through in measurable quantities. On the basis of the differing null [zero] rates, registered in connection with the Coulter Counter measurements, it was however possible to determine that the MF 12 was definitely the coarser of the two.

Table 2 in particular illustrates the relationships between the form and the filterability of the test particles: assuming that the diameter is just about the same, the longitudinal bacteria cells turned out to be just as easily filterable as the latex particles whose volume on the basis of their spherical shape was many times smaller. The Colanyl Green particles, which reached the filtrates, likewise were disproportionately voluminous -- obviously as a result of the very high plastic deformability.

The volume of the latex particles used, which was 0.557 mmu, is already so small that the particles here, under the experimental conditions described, cannot be measured as individual particles. However, in watery suspensions, latex particles incline toward the formation of particle aggregates. The size of these aggregates, as Preusser determined also by means of the electron microscope (personal communication), is a function of the total particle concentration. Moreover, the Coulter Counter, at high particle concentrations, registers several or many individual particles found simultaneously in the nozzle area as a single, correspondingly larger particle. This so-called coincidence effect is likewise a function of the concentration. It was therefore possible also to establish the presence of latex particles with diameters of 0.557, 0.365, and 0.264 mmu; it was thus also possible to use the volumes of the largest "particles" registered in a filtrate in each case as a yardstick for the relative evaluation of the permeability of the filters tested.

The filters made by the Sartorius Membrane Filter Co, Inc, which were tested here proved to be uniform in terms of their quality. The volumes of the largest latex particle aggregates and bacteria cells, registered in the filtrates, differed from each other, assuming we work with filters of the same type, generally by no more than about 0.04 cubic mm.

([2] In the Colanyl Green filtrates, the fluctuations were somewhat greater although this is due obviously to the fact that the particle spectra of the unfiltrated pigment suspensions were not completely uniform.)

On the other hand, there was quite a bit of difference in the quality of the tested millipore filter of Type HA and the Gelman filter, Type GA 6. Particles of certain size were completely or almost completely retained by some of these filters while other filters (including those from the same charge [lot]) allowed these particles to pass in moderate or larger quantities. The volumes of the largest particle aggregates and bacteria cells registered in the filtrates of these two filter types differed quite considerably from one experiment to the next, partly by far more than 0.1 cubic mm.

I want to thank Mr G Konig for his extremely conscientious collaboration in these experiments. At the same time I want to express my appreciation to Dr D Thon and Dr R Gropl from the Sartorius Membrane Filter Co, Inc, Gottingen, and my colleague, Dr H.J. Preusser, for the discussions and ideas they offered; I am particularly indebted to the latter for the electron-microscope study of the bacterial inhibition forms.

Summary

The filtration effectiveness of a total of 17 Sartorius membrane, millipore, and Gelman filters was tested through experiments for test particles of varying form and size, suspended in a 4% watery NaCl solution (latex, Colanyl Green, and bacteria cells). This was done by means of electronic and microbiological filtrate analyses. The most important investigation results are compiled in the form of tables and are briefly discussed.

Bibliography

(1) Preusser, H.J., Kolloid Z u Z Polymere (in print)

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Table 1. Permeability of Sartorius Membrane, Millipore, and Gelman Filters for Particles of Different Shape and Size Suspended in a 4% NaCl Solution

Filter-type (a)	Latex ø 2,06 µm	Latex ø 1,90 µm	Latex ø 1,305 µm	Latex ø 1,171 µm	Latex ø 0,796 µm	Latex ø 0,557 µm	Latex ø 0,365 µm	Colanyl-grün (b)	Serratia marcescens/ Normal-formen (c)	Serratia marcescens Hemm-formen (d)
MF 500	+	+	+	+	+	+	+	+	+	+
Millipore SM	–	–	+	+	+	+	+	+	+	+
MF 250	–	–	+	+	+	+	+	+	+	+
MF 150	–	–	–	+	+	+	+	+	+	+
Gelman GA 4	–	–	–	–	+	+	+	+	+	+
MF 125	–	–	–	–	(+)	+	+	+	+	+
Millipore AA		–	–	–	–	+	+	+	+	+
MF 100	–	–	–	–	–	+	+	+	+	+
Millipore HA	–	–	–	–	–	–	(–) → +	+	+	+
Gelman GA 8	–	–	–	–	–	–	+	+	– → +	+
MF 50	–	–	–	–	–	–	+	+	(+)	+
Millipore GS	–	–	–	–	–	–	(+)	(+)	–	+
MF 30	–	–	–	–	–	–	(+)	–	–	+
MF 15	–	–	–	–	–	–	–	–	–	+
MF 14	–	–	–	–	–	–	–	–	–	(+)
MF 12	–	–	–	–	–	–	–	–	–	–
MF 10	–	–	–	–	–	–	–	–	–	–

Key.
a. filter type
b. Colanyl Green
c. Serratia marcescens/normal forms
d. Serratia marescens/inhibition forms

\+ = Particles definitely established in filtrate
– = No particles established in filtrate
(+) = Only very small particle quantities established in filtrate
(–) = Complete absence of small particle quantities established in filtrate
– → + = Filtration effectiveness differs from filter to filter.
For latex particles with diameter of 0.264 mm the same filters proved to be permeable as for latex particles with a diameter of 0.365 mm.

TABLE 2

MAXIMUM VOLUMES OF PARTICLES REGISTERED BY SARTORIUS MEMBRANE, MILLIPORE, AND GELMAN FILTERS

Filtertyp Bezeichnung und vom Hersteller angegebener mittlerer Porendurchmesser (a) (µm)		(b) Testpartikel: Polystyrol-Latex[1] Größe der Testpartikel (d) (Mittelwert) Durchmesser (f) (µm)	Volumen (g) (µm³)	(e) Volumen der größten im Filtrat registrierten Partikel und Partikelaggregate (µm³)	(c) Colanylgrün Volumen der größten im Filtrat registrierten Partikel (h) (µm³)	Serratia marcescens Volumen der größten im Filtrat registrierten Normalformen (i) (µm³)	Volumen der größten im Filtrat registrierten Hemmformen (j) (µm³)
MF 500	8 (5)	2,05	4,401	9,87	2,63[3])	3,44[3])	(k) nicht untersucht
Millipore SM	5	1,305	1,164	2,26	2,52	3,44[3])	" "
MF 250	3 (2)	1,305	1,164	1,96	2,41	3,37	" "
MF 150	1,2 (1,0)	1,171	0,841	1,15	2,33	2,33	" "
Gelman GA 4[4])	0,8	0,796	0,264	1,63	2,00	1,92	" "
MF 125	0,8 (0,9)	0,796	0,264	1,52	1,96	1,74	" "
Millipore AA	0,8	0,557	0,090	1,70	1,94	1,70	" "
MF 100	0,6 (0,8)	0,557	0,090	1,63	1,81	1,52	" "
Millipore HA[4])	0,45	0,365	0,025	1,74	1,74	1,55	" "
Gelman GA 6[4])	0,45	0,365	0,025	1,52	1,59	1,55	" "
MF 80	0,45 (0,6)	0,365	0,025	1,48	1,52[3])	1,22	" "
Millipore GS	0,22	0,365	0,025	1,41	1,42[3])	—	1,85
MF 50	0,2 (0,3)	0,365	0,025	1,37	(1,37)[3](k)	—	1,78
MF 15	0,15 (0,27)	—	—	—	nicht untersucht	—	1,67
MF 14	0,1 (0,2)	—	—	—	" "	—	1,52
MF 12	0,05 (0,15)	—	—	—	" "	—	—
MF 10	0,1 (0,01)	—	—	—	" "	—	—

Key. a. Filter type designation and average pore diameter given by manufacturer
b. Test particle
c. Colanyl Green
d. Size of test particles (average value)
e. Volume of largest particles and particle aggregates registered in the filtrate
f. Diameter
g. Volume
h. Volume of largest particles registered in the filtrate
i. Volume of largest normal forms registered in the filtrate
j. Volume of largest inhibition forms registered in filtrate
k. Not investigated

(1) For the Sartorius Membrane Filters, we have, in the first place here, the values determined with the help of the mercury intrusion method while the 2nd place [digit] in parentheses shows us the value determined by means of through-flow measurements.

(2) Only the values for the largest particle types passing through a filter here in each case are shown.

(3) It would seem to be possible that considerably larger bacteria could be filtrated through MF 500 and Millipore Filter SM, but in the bacteria populations investigated only very few cells had volumes of more than 3.44 cu mm (maximum volume about 3.55 cu mm). Accordingly, we must also take into consideration in any data on the maximum sizes of the Colanyl Green particles registered in the filtrates coming through the coarser filter types that the largest particles prior to filtration revealed volumes only about 3.11 cu mm.

(4) Considerable quality differences from one filter to the next, also within one and the same lot.

(5) Filtrate only very slightly stained [dyed].

(6) Filtrate colorless. Here we measured only the colorless spherical particles coming from the Celanyl Green preparation used.