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# Effects of Liquid Properties on Sedimentation Volume of Powder

(Received July 20, 1959)

Hiroshi KUNO\*

#### Abstract

It is experimentally confirmed that the sedimentation volume of the powder in the liquid increases with the increase of the density of the liquid, but is not affected by the change of the viscosity of the normal liquid.

When the liquid, however, is thixotropic, the sedimentation volume of the powder in the liquid of high effective viscosity is remarkably large.

Sedimentation volumes of calcium carbonates in solutions of several surface active agents were measured, and the fact that the sedimentation volume of the powder has no simple relation with the degree of dispersion of the powder was confirmed.

# I. Introduction

Sedimentation volumes of powders are usually used as a measure for the determination of the degree of dispersion of the powders in liquids. Though this method of estimation is practically important, factors determining the sedimentation volume of the powder are not yet fully understood and frequently overlooked. It is the purpose of this paper to elucidate the effect of the physical properties, viscosity and density, of liquids on the sedimentation volume of the powder in liquids and to study the real relation between the sedimentation volume of the powder and the degree of dispersion of that powder in the liquids.

Powders dispersed in liquids are customarily judged well dispersed when the sedimentation volumes of the powders are small. Such a simple pallarel relation between the sedimentation volume and the degree of dispersion of the powder is not acceptable when we think over the complex properties of the mechanism of the sedimentation of the powder. This idea of simple relationship, however, is very familiar in practical field, and it is necessary to determine the validity of this idea.

#### II. Experimental

The sedimentation volumes of powders were measured with 30 ml. graduated glass

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tube containers, one centimeter in diameter. About two grams of the powder was weighed in the container, 25 ml. of the liquid was added, the whole was shaken vigorously with hand and stood vertically. The settling volume of the powder after a night was measured, and the volume of the powder of unit weight was given as the sedimentation volume of the powder.

In the cases of organic liquids, a stopcock was attached to the container by a ground joint and the powder was evacuated an hour at 150 °C before the introduction of the liquid in order to avoid the effect of the moisture of the powder.

Every series of the experiments was performed at the same time under the same conditions at room temperature.

# **III.** Results and Discussion

## Effects of Viscosity of Liquid

Effects of the viscosities of liquids were studied on the sedimentation volume of



Fig. 1. Relations between viscosity of liquid and sedimentation volume of crushed glass powder (av. dia. 18. 3  $\mu$ ).

studied on the sedimentation volume of crushed glass powders. Several concentrations of aqueous solution of glycerin, acetone-methanol solution of nitrocellulose and aqueous solution of sodium carboxymethyl cellulose (C. M. C.) were used as liquids of different viscosities.

The viscosity of the liquid was measured with an Ostwald viscometer. The results are given in Fig. 1 and Table 1.

As given in Fig. 1, the sedimentation volume of the powder does not change with the viscosity of the liquid in the cases of aqueous solution of glycerin and acetone-methanol solution of nitrocellulose.

On the contrary, as given in Table. 1, in the case of the aqueous solution of

Table. 1. Sedimentation volume of crushed glass powder in aqueous solution of C. M. C. av. particle dia. 29. 7  $\mu$ 

conc. of C. M. C. soln. wt $\mathscr{H}$	viscosity of soln. c. p.	sedimentation volume cm <sup>3</sup> /g
0	1. 39	0.833
0.003	1.65	0.890
0.009	2.03	0.889
0.015	2,26	0,905
0.030	3, 53	0.930
0.065	4.92	0.950
0.15	8.50	1.218
0.50	43.76	2.287

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C. M. C., the sedimentation volume of the powder increases with the increase of the viscosity of the liquid.

The aqueous solution of C. M. C. is known to have thixotropic property. Therefore, the effective viscosity of the solution will change with the shear stress applied. Since the shear stress produced by the powder particle is much smaller than that applied in the measurement of viscosity by the Ostwald viscometer, the effective viscosity against the particle may be much greater than the viscosity obtained in the viscosity measurement. Though the effective viscosity against the particle remains unknown, it can be concluded that very high viscosity will increase the sedimentation volume of the powder. But, as given in Fig. 1, in the case of ordinary liquids we need not consider the effect of the viscosities of liquids on the sedimentation volume of the powder.

#### Effects of Density of Liquid

Several concentrations of solutions of polar liquids, aqueous solution of sulfric acid, and non-polar liquids, mixture of benzene and carbon tetrachloride, were prepared as liquids of different density, and the sedimentation volume of crushed glass powder in these liquids were measured. The density of the liquid was measured with an Ostwald-Sprengel pycnometer. The density of the glass powder was 2.24 g/cm<sup>3</sup>.

The results are given in Fig. 2 on the sulfric acid solution, and in Fig. 3 on the benzene-carbon tetrachloride mixture.

In both cases the effect of the density of the liquid on the sedimentation volume of the powder is obvious. The sedimentation volume of the powder increases almost linearly with the increase of the density of the liquid used. The effect of the density



Fig. 2. Sedimentation volume of crushed glass powders in aqueous solutions of sulfric acid. Numerals near the lines indicate the av. particle diameter.



Fig. 3. Sedimentation volume of crushed glass powder in the benzene-carbon tetrachloride mixture (av. particle dia. 29.7  $\mu$ ).

of the liquid is more remarkable as the particle size of the powder becomes smaller.

At the ends of the curves, where the liquids change from pure liquids to mixtures of two liquids, the sedimentation volumes of the powder change sharply and deviate from the straight lines. These sharp changes of the sedimentation volume are due to the changes of the chemical properties of liquids. This effect of the chemical properties of liquids, as given by Wolf<sup>1)</sup>, occurs sharply when the concentration of the solution is very dilute and does not increase with the increase of the concentration. Therefore, we can conclude that the linear change of sedimentation volumes with the concentrations of solutions, given in Figs. 2 and 3, is not the effect of the chemical properties of liquids but mainly due to the effect of the density of liquids.

When we compare the results given in Figs. 2 and 3, we find that the sedimentation volumes do not show the same value in the solutions of the same density. This diffrence is due to the difference of the chemical properties of the liquids. The affinity of the glass powder particle to the liquid changes as the polarity of the liquid changes. The powder flocculates in the non-polar liquid, where the affinity between the particle and the liquid is weak, and shows a large sedimentation volume. This effect is clear in Table. 2, where the sedimentation volume of spherical glass powders are larger in benzene than in alcohol.

Table. 2.	Sedimentation	volume o	of	spherical	glass	powders.	$cm^{3}/g$ .
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av.	particle	diameter	, μ.
37.1	24.7	17.2	11.4
0.743	0.749	0.749	0.749
0.676	0.675	0.678	0.668
	av. 37.1 0.743 0.676	av. particle   37.1 24.7   0.743 0.749   0.676 0.675	av. particle diameter,   37.1 24.7 17.2   0.743 0.749 0.749   0.676 0.675 0.678

1) K. L. Wolf and R. Kurtz, Z. angew. Chem. 66, 739 (1954)

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# Dispersion of Powder and Sedimentation Volume.

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It is usually believed that the sedimentation volume of the powder is small when the powder is well dispersed. This relation between the sedimentation volume and the degree of dispersion of the powder was examined on a calcium carbonate powder in solutions of surface active agents. The results are given in Tables 3 and 4.

The size distributions of the floccules in liquids were determined by the sedimentation methods. The results in Table 3 were measured by the photo-extinction method of Rose<sup>2)</sup>, and those in Table 4 were measured by the automatic recording sedimentation balance of Shimazu Seisakusho, Ltd.

As it is inconvenient to show the whole size distributions, the degree of dispersion were given by the diameter of the floccule where the weight percentage undersize of the cumultive size distribution curve is 50 %,  $d_{50}$ , or 70 %,  $d_{70}$ . The degree of dispersion is greater where the value of  $d_{50}$  or  $d_{70}$  is smaller.

surface active agent	conc. wt. <i>%</i>	$d_{70} \ \mu$	sedimentation volume cm <sup>3</sup> /g
sodium-	0.1	44.5	2.63
pyrophosphate	0.3	xx	2.10
•••••	0.6	52.7	3.01
	0.01	57.8	3.27
Texaphol	0.05	44.7	3.47
-	0.5	ХX	3.03
pure water	-	67.8	3.08

Teble. 3. Sedimentation volume of calcium carbonate in aqueous solution of surface active agents. cm<sup>3</sup>/g.

xx means perfect dispersion.

Table. 4. Sedimentation volume of calcium carbonate in solutions of surface active agents.  $cm^3/g$ 

solvent	surface active agent	concentration	$d_{50} \atop \mu$	sedimentation volume cm <sup>3</sup> /g
			10.5	3.21
water	Aerosol OT sodium dodecyl-	1 mmol/1	17.0	4.00
	benzene sulfonate	1 mmol/l	17.0	4.46
	Tween 80	1  mmol/l	7.0	3.01
	<u> </u>		22.0	7.75
xylene	Arlacel 83	0.1 wt%	<1.0	2.29
-	Aerosol OT	1 mmol/l	10.0	6.30
carbon-			26.0	10.54
tetra-	Arlacel 83	0.1 wt%	<1.0	2.04
chloride	Arlacel 83	10.0 wt%	<1.0	2.20

2) H. E. Rose, "The Measurement of Particle Size in Very Fine Powders." 1953, London.

We can see from Tables 3 and 4, that the sedimentation volume generally becomes smaller as the degree of dispersion becomes greater, except in the case of Texaphol solutions. But the sedimentation volumes are slightly different for the same degree of dispersion when different surface active agents are used, as in the cases of aqueous solutions of Aerosol OT and sodium dodecylbenzene sulfonate in Table 4. This tendency is more remarkable when the solvents are different as in the case of Aerosol OT solutions, where the relation between the sedimentation volume and the degree of dispersion is reversed in water and in xylene as given in Table 4.

In the case of aqueous solutions of Texaphol, the degree of dispersion increases with the increase of the concentration of the solutions, but the sedimentation volume of the powder reaches a maximum and decreases as the concentration increases. Though I can not present the reason for the exceptional behavior of Texaphol solutions, it is interesting that this case is a clear example of the exception in the relation between the sedimentation volume and the degree of dispersion which is usually believed.

#### IV. Conclusion

It is well known that the sedimentation velocity of the powder particle is proportional to the difference of the densities of the particle and the liquid, and inversely proportional to the viscosity of the liquid. It is experimentally proved in this paper that the sedimentation volume of the powder is not directly connected with the sedimentation velocity of the powder particles. The sedimentation volume of the powder in the liquids increases almost proportionally with the increase of the density of the liquids, but the viscosity of usually used normal liquid has no effect upon the sedimentation volume.

When the liquid is thixotropic and the effective viscosity against the powder particle is very high, the sedimentation volume in this liquid becomes remarkably large.

That the sedimentation volume of the powder becomes small as the degree of dispersion increases seems to hold roughly true in many cases. But, there are many exceptions such as given in this paper, and we have to use this relation with great care.