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THE ECONOMIC OPTIMIZATION OF BAFFLES TO DISPERSE
 TiO_2 IN OPEN IMPELLER AGITATORS

BY

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A Thesis submitted
in partial fulfillment of
the course requirements for
The Bachelor of Engineering Degree

Western Michigan University

Kalamazoo, Michigan

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INTRODUCTION

This project began with an observation of a coating make-down tank at a nearby paper mill. The make down tank, a cowles dissolver, was being used to disperse and mix pigment and other common coating ingrediants. Because the tank was unbaffled, the circulating suspension climbed the walls of the vessel, and could only be half filled to prevent overflowing.

There is a view held by some people that good dispersion cannot be obtained when baffles are used in these tanks. This paper mill obviously held this view.

The basic motivation for this thesis was the fact that the paper industry is currently using various numbers of baffles in the agitated vessels used for coating makedown. This must stem from a difference in opinion as to optimum number of baffles to use.

The purpose of this thesis was to investigate and make conclusions on how the number of baffles affects the efficiency of open impeller mixers to disperse pigments.

THEORETICAL DISCUSSION

WHY DISPERSION IS REQUIRED

In order to achieve the maximum benefits from pigments in the coating layer of coated paper it is necessary to disperse the particles to some ultimate small size. First, this small size permits the particles to fit between surface fibers and thus to level or smooth the geometric contour. Second, breaking the agglomerates down into discrete particles increases the surface area for light scattering. Third, the rheology of the pigment will be enhanced when the pigment acts as smaller discrete particles. Fourth, the uniformity of ink penetration and most other paper properties will be enhanced by applying a larger number of smaller particles rather than a smaller number of large particles (1). Pigment particle size easily manifests itself in paper tests such as gloss and smoothness (2).

Most dry pigments are composed of agglomerates (clusters of aggregates) of the individual particles. The small amount of energy which holds the agglomerates together must be overcome to produce the smaller aggregates. Finally the aggregates can be broken down further with larger amounts of energy to the optimum particle size (The individual particle size).

The dispersion of pigment particles can be described qualitatively as a two step process. The first is a mechanical separation of particles to their ultimate size. The second is a chemical stabilization of the particles to prevent recombination and/or settling of the particles from the suspension state (1).

CHEMICAL STABILIZATION

Chemical stabilization has been well documented and is not the purpose of this discussion. However it should be noted that chemical stabilization is an important parameter in any pigment dispersion process.

PIGMENT PROPERTIES

The aggregative properties of various dry pigments vary considerably. Thus they will vary widely in their resistance to dispersion. These variations may be due to differences in chemical composition, particle size distribution, crystalline structure, drying methods, nature of the surface, etc. The agglomerate forces are strong in such pigments as TiO_2 and ZnO , but are quite weak in Talc and clays. Calcium Carbonate falls between these two classes. For this reason, TiO_2 was utilized for this project (3).

MIXING INTENSITIES

There are many types of mixers/agitators used to disperse pigment. Each unit may use a different mechanism to disperse the particles.

Since the open impeller agitator is widely used throughout the industry for this purpose, it was chosen for this project.

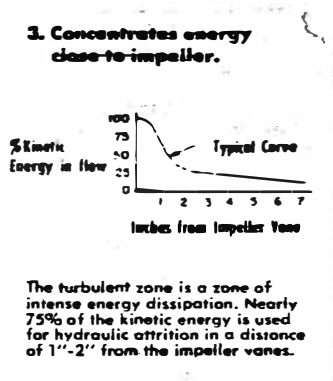


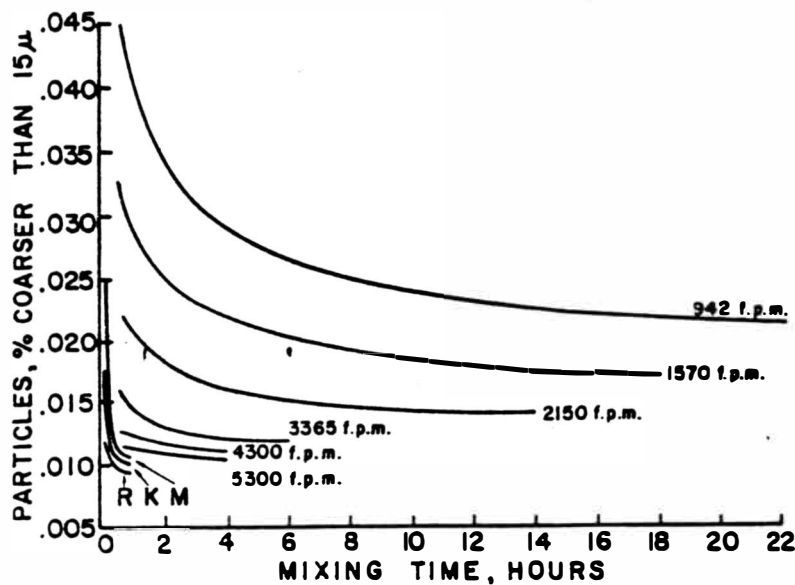
Figure 1 (4)

These types of agitators work on the principle of creating a high intensity shear in the impeller proximity while creating enough pumping action to keep the entire batch moving around the vessel and through this high intensity zone. It has been documented that the zone of highest energy intensity for these agitators lies in the immediate vicinity of the rotating member (3). Figure 1 shows how a cowles dissolver concentrates energy close to the impeller (4).

Thus it is important that circulation patterns in an agitated vessel allow each particle to pass through this high intensity zone to break down the agglomerates. An important parameter in the dispersion process is the time required to break apart the agglomerates. Time becomes a factor because enough time must be allowed for each particle to pass through this high intensity zone (3).

Millman & Whitley (3) demonstrated the effects of time of dispersion on particle size. Figure 2 demonstrates how particle size reduction took place over a period of hours. This of course was attributed to the fact that as the time of dispersion increased, each agglomerate had more chances of passing through the high intensity zone. The diagram also shows how particle size reduction is a function of impeller velocity.

FIGURE 2(3)



A higher tip velocity drastically reduced the time required to obtain the same particle size.

In short it was found that time cannot compensate for a lack of intensity. The concept that agglomerate breaking occurs when some threshold force is exceeded does not involve the element of time. Time plays a role because time is required to bring all particles through the zone of highest energy density.

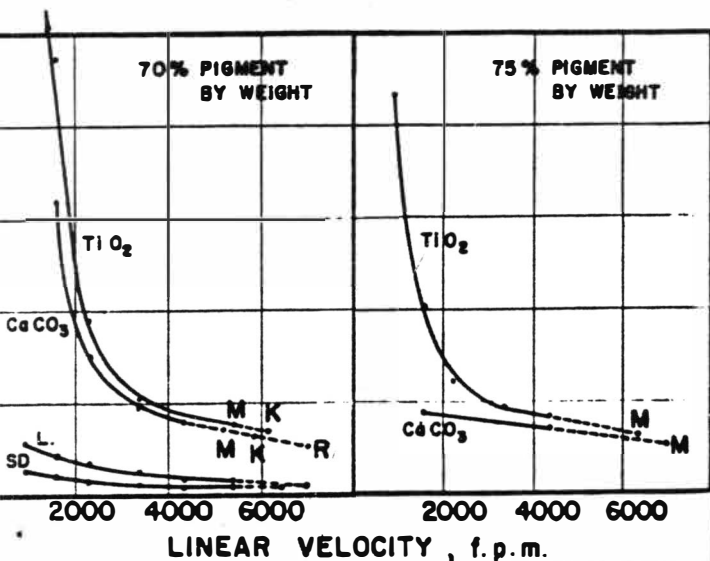


Figure 3 (3)

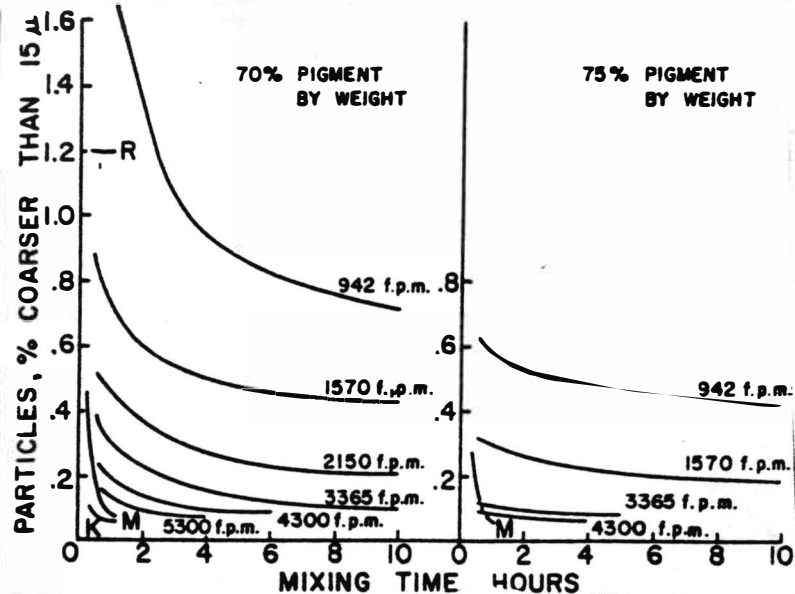


Figure 4 (3)

Higher speeds raise the energy input and pass material through points of maximum intensity more frequently, therefore the time of processing is reduced. After a period of time, the particle size is not affected by time, thus a smallest ultimate particle size that can be obtained with a given linear tip velocity can be plotted. Figure 3 shows this relationship for TiO_2 and CaCO_3 .

Studies have been performed on the solids/degree of dispersion relationship also. Figure 4 shows that dispersion improves with increasing pigment concentration because of the greater shearing forces produced by the higher viscosity and increased particle interference (less water to lubricate).

For TiO_2 no marked change in dispersion was observed until the concentration reached 70%. This can be correlated to TiO_2 's higher specific gravity. If two pigments are compared on a weight concentration basis, the volume of TiO_2 present will be less and therefore less interparticle shearing forces are present (3). For this reason, the TiO_2 used in this project was dispersed at 72 % solids.

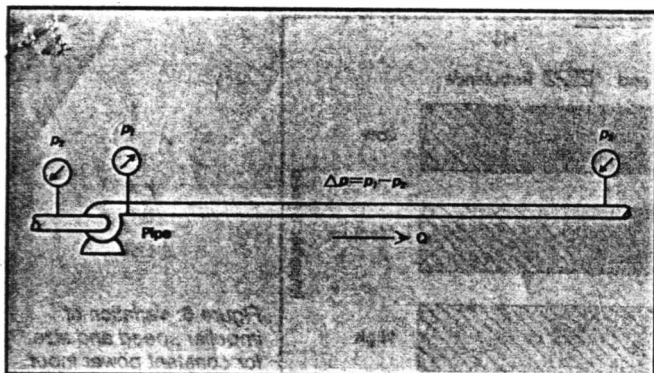
POWER CONSIDERATIONS

The amount of power used during disagglomeration may not be proportional to the results achieved. Neither the amount of power nor total work input determines whether the agglomerates are

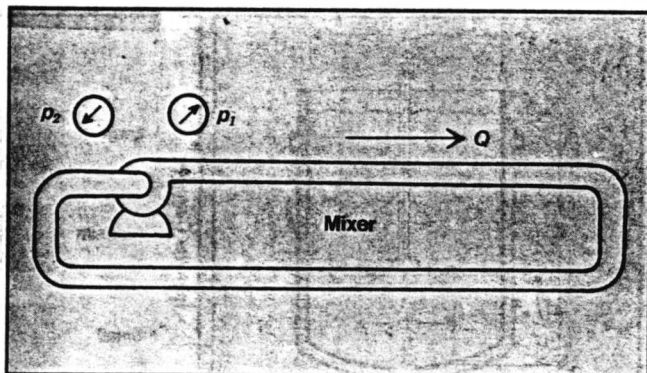
broken. This is determined rather by the force reaching each agglomerate. While it is frequently true that the greater the power input the greater force which each aggregate will experience, in the case of dilute suspensions however, it is possible to supply high power input without subjecting individual aggregates to forces large enough to disrupt them.

The power input is used to pump the fluid around the vessel, and is dissipated as heat resulting from viscous work, not interparticle friction required for disagglomeration. The overall determining factor is the power input per unit volume of material in the effective zone of the machine, with the qualification that the power is dissipated in interparticle friction, not viscous friction (5).

Uhl (6) describes agitating vessels as they relate to a simple pump-pipe relationship. He states that mechanical mixers are basically pumps because pumps transfer fluid in conduits at a specified flow rate against an opposing pressure of head, similar to agitated vessels which can be described as a recirculating pump-pipe relationship (Fig 5&6). The circulation in an agitated vessel differs from this simple pump recirculation loop because the



A conventional pumping system



A pumping loop, which is analogous to a mixer

FIGURE 5 (6)

FIGURE 6 (6)

circulation in the agitator is not controlled like it is in the conduit. The velocity and direction of fluid flow in an agitated vessel is determined by the geometry of the system.

The power input into the fluid stream can be given by the equation:

$$P = Q (p_1 - p_2)$$

where P = power input (ft lb/min)

Q = flow (ft³/min)

p₁ = pump inlet pressure

p₂ = pump outlet pressure

Since the pressure difference can be represented by a height of fluid of known density, we have:

$$P = Q \rho \hat{H} \quad (6)$$

where P = power input

Q = flow

ρ = fluid density (lb/ft³)

H = Head (static, pressure, velocity, turbulent)

Depending on the geometry of the impeller and speed of rotation, the flow component or the head component of the input can be emphasized.

Thus the same amount of power can be applied to produce a large flow-small head relationship or a small flow-large head relationship.

Operations that require large flows will use a large impeller at low speed, while operations requiring high head will use small impellers at high speeds.

In the case of agitated vessels, the flow capacity component of the impeller controls the circulation rate in the tank. The fluid head component is proportional to the turbulence or shear, and defines the intensity of agitation in the immediate vicinity of the impeller (7).

When the flow in the tank is turbulent, the power requirement to drive the impeller can be estimated as the product of the flow (q) produced by the impeller, and the kinetic energy per unit volume of the fluid : (8)

$$P = n^3 N_q \rho / 2 g_c (\alpha \times \pi n D)^2$$

where $\alpha = \frac{\text{total liquid velocity}}{\text{velocity of blade tips}}$

velocity of blade tips

Recombining we get:

$$\frac{P g_c}{n^3 D^5 \rho} = \frac{\alpha^2 \pi^2 N_q}{2}$$

and Np (power number) is defined as: $\frac{P g_c}{n^3 D^5 \rho}$

To estimate the power required (P) empirical correlations are needed to relate the power number with the other variables of the system. The other variables such as fluid viscosity and density can be combined to form the Reynolds number (N_{re}):

$$N_{re} = \frac{n D^2 \rho}{\mu}$$

where n = rev/sec
 D = impeller diameter
 p = density
 u = viscosity

By plotting N_p versus N_{re} curves such as figure 8, one can eventually find P for a given reynolds number. Note that at a reynolds number above 5000-10000 the power number remains constant in baffled tanks.

The shape factors of a tank such as impeller diameter to tank base width, fluid height to fluid depth, and distance between impeller and bottom of tank to impeller diameter will shift the curve up or down slightly (8).

BAFFLE CONSIDERATIONS

Based on the preceding summary of power consumption, the relationship between baffles and power consumption can be considered.

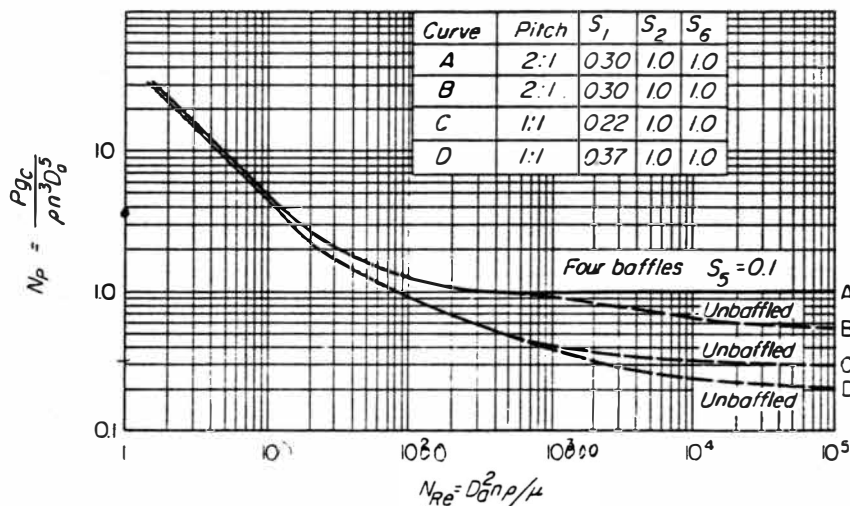


Figure 7 (8)

At very low reynolds numbers (1-100) the flow is laminar and there is no need for baffles because vortex formations do not occur. Based on the reynolds equation, low reynolds numbers usually occur when the viscosity of the fluid is high. At the same time, addition of baffles will not change the power consumption by an appreciable quantity. If the reynolds number is high enough to create fully turbulent conditions, a difference in power consumption occurs between baffled and unbaffled tanks.

The power consumed and/or the power reaching the aggregates will be higher for baffled tanks than for unbaffled tanks (See figure 7) (8). This is because the baffles create resistance to flow, similar to pipe friction in a pump system. In order to maintain a constant speed, the motor torque increases, which

increases the current, and therefore the power consumed.

The power reaching each particle is increased because some of the horizontal velocity components (which do not allow the fluid to come into contact with the impeller) are changed into vertical velocity components when the suspension strikes a baffle. This allows the particles to come into contact with the impeller more often.

Thus for viscous systems, the vertical flow needed to move the particles to the high intensity energy zone can usually be obtained without the use of baffles. The viscous drag of the fluid on the sides of the tank will act as a "dynamic baffle". In these situations baffles should actually be avoided because they may interfere with the vertical flows needed (7).

Baffles are therefore required when high mixing intensities are required and the viscosity of the fluid is low. This scenario occurs when dispersing pigments. The baffles produce localized turbulence that develops high shear stresses, as well as to redirect the horizontal component of the fluid velocity to a vertical component so that all particles will reach the high intensity zone (7).

It was determined that what was needed were plots of particle size with time using various numbers of baffles. From these, the efficiency of the operation with respect to energy costs to disperse the pigment could be obtained by selecting a particle size, comparing how long it took for each configuration to obtain the same size, then multiplying this time by the power consumed. These results would show the configuration that used the lowest amount of energy to obtain the same particle size.

EXPERIMENTAL PROCEDURE

MATERIALS

The TiO_2 used was dry Dupont TI-PURE R-900. It is approximately 94% titanium dioxide and 4.5 % aluminum oxide. This grade was suggested by Dupont because of its popularity in coatings.

The TiO_2 was dispersed using Dispex N-40 (40 % sol) as a chemical dispersant.

Distilled water was used as a dispersing medium.

EQUIPMENT

A laboratory cowles dissolver was used with a 2" impeller. The operating speed was 5400 rpm. The shape factors used were as follows:

$$\begin{aligned} D_i/D_t &= .31 \\ D_i/E &= 1 \\ H/D_t &= 1 \\ J/D_t &= .11 \end{aligned}$$

where:

$$\begin{aligned} D_i &= \text{impeller diameter} \\ D_t &= \text{tank bottom diameter} \\ E &= \text{distance from tank bottom to impeller} \\ H &= \text{height of fluid suspension} \\ J &= \text{baffle width} \end{aligned}$$

A direct reading Simpson wattmeter was used to measure the power consumed.

The particle size was measured using a Micromeritics Sedigraph 5000D. This device uses x-rays to measure the settling velocity of the TiO_2 particles. The settling velocity is then correlated to the particle size using Stoke's law. A detailed description and procedure is available in the Appendix.

PROCEDURE

Adding Ingredients

The 72 % suspension was created by adding the proper amounts of water, dispersant, and TiO_2 . The water and dispersant were added, the cowles dissolver was turned on, and the TiO_2 was added. Time zero was selected when all the pigment was in. Samples were then drawn at 5, 15, and 35 minute intervals. This procedure was carried out with 0, 2, and 4 rectangular baffles in the tank.

Power Measurement

The power consumption was initially measured after 5 minutes for each baffle configuration. Later this method was changed because the power consumed during each dispersion changed as the dispersion continued. Therefore a later trial was set up just to measure the power consumed at 2 minute intervals for each baffle configuration. The power versus time curve was then integrated by hand to obtain the true overall power consumed during the time considered.

Sample Preparation

The 72 % samples had to be diluted to some unknown concentration for the sedigraph analyzer. This concentration varies from pigment to pigment and is related to how well the pigment absorbs x-rays. Therefore a trial method is used to obtain the proper concentration. This concentration was found to be 2.53% solids.

Because a relatively large amount of water had to be added to obtain this concentration, there was a concern that the chemical aspects of the dispersion may be upset and that flocculation of the particles may occur when the water was added. For this reason, a preliminary trial was run with various levels of dispersant being added to the water before dilution. This was to prevent flocculation of the pigment particles.

The initial level added to the water was half of the original ratio between the dispersant and the water (in the 72% concentration). The level was then cut in half twice for a total of three levels. This preliminary trial showed that when the level of dispersant added to the dilution water was decreased, the particle size measured decreased tremendously. It was concluded that the instrument was measuring the particle size of the dispersant and not the TiO_2 . Therefore, no dispersant was added to the dilution water for the final trials presented here.

Particle Size Measurement

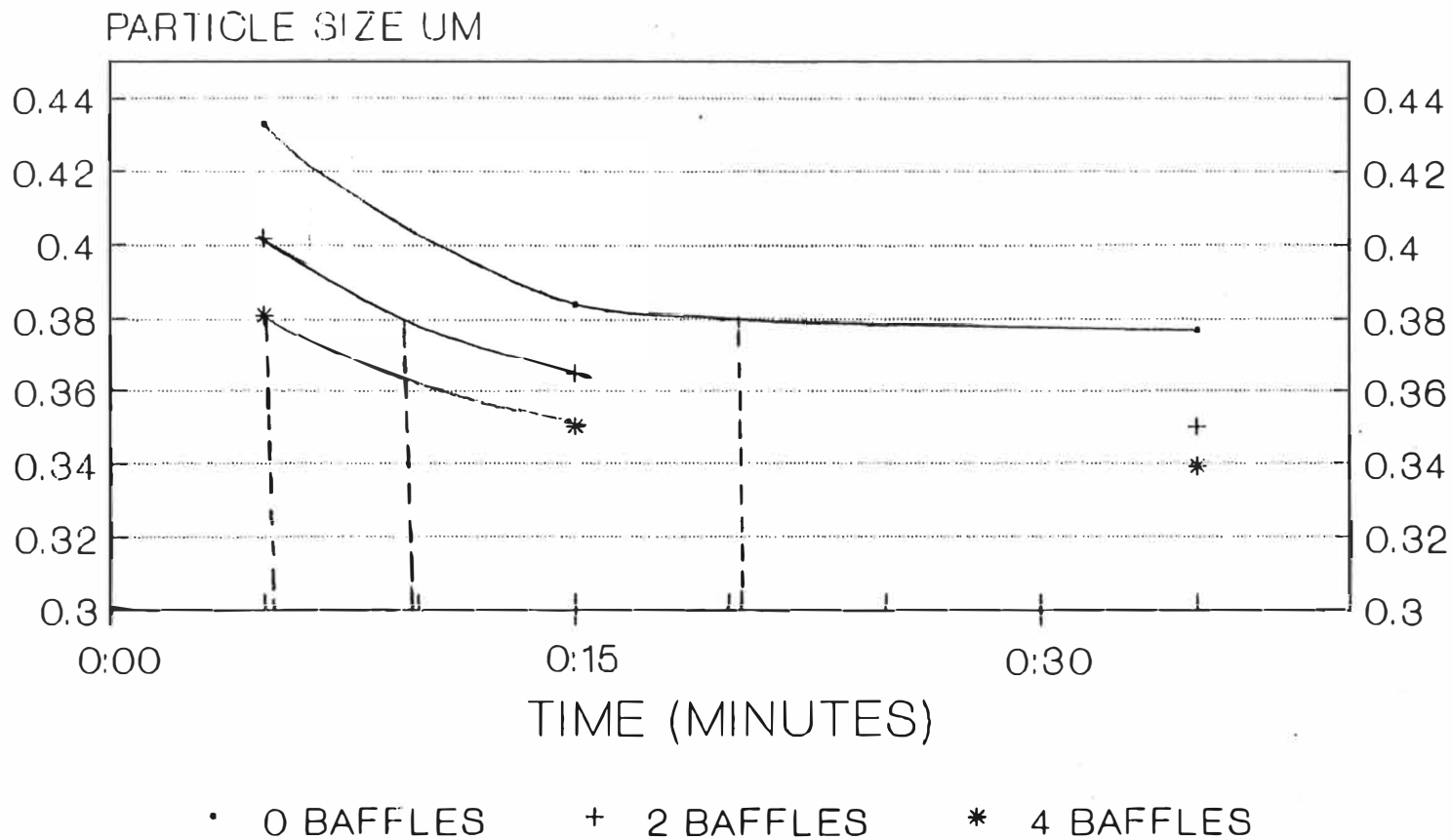
Once the samples were diluted, the particle size was measured according to the procedures outlined in the appendix. Since the sedigraph graphs the particle size by cumulative mass percent less than a particle size, the average particle size was found by integrating the graphs by hand. (see appendix for integration data)

Statistical Analysis

The testing error was measured by running three tests on one sample. This would give the standard deviation of the procedure. A t-test was run with two degrees of freedom to determine the level of confidence between baffle configurations at the same time intervals. The number of samples taken was hampered by the long time required for particle size analysis.

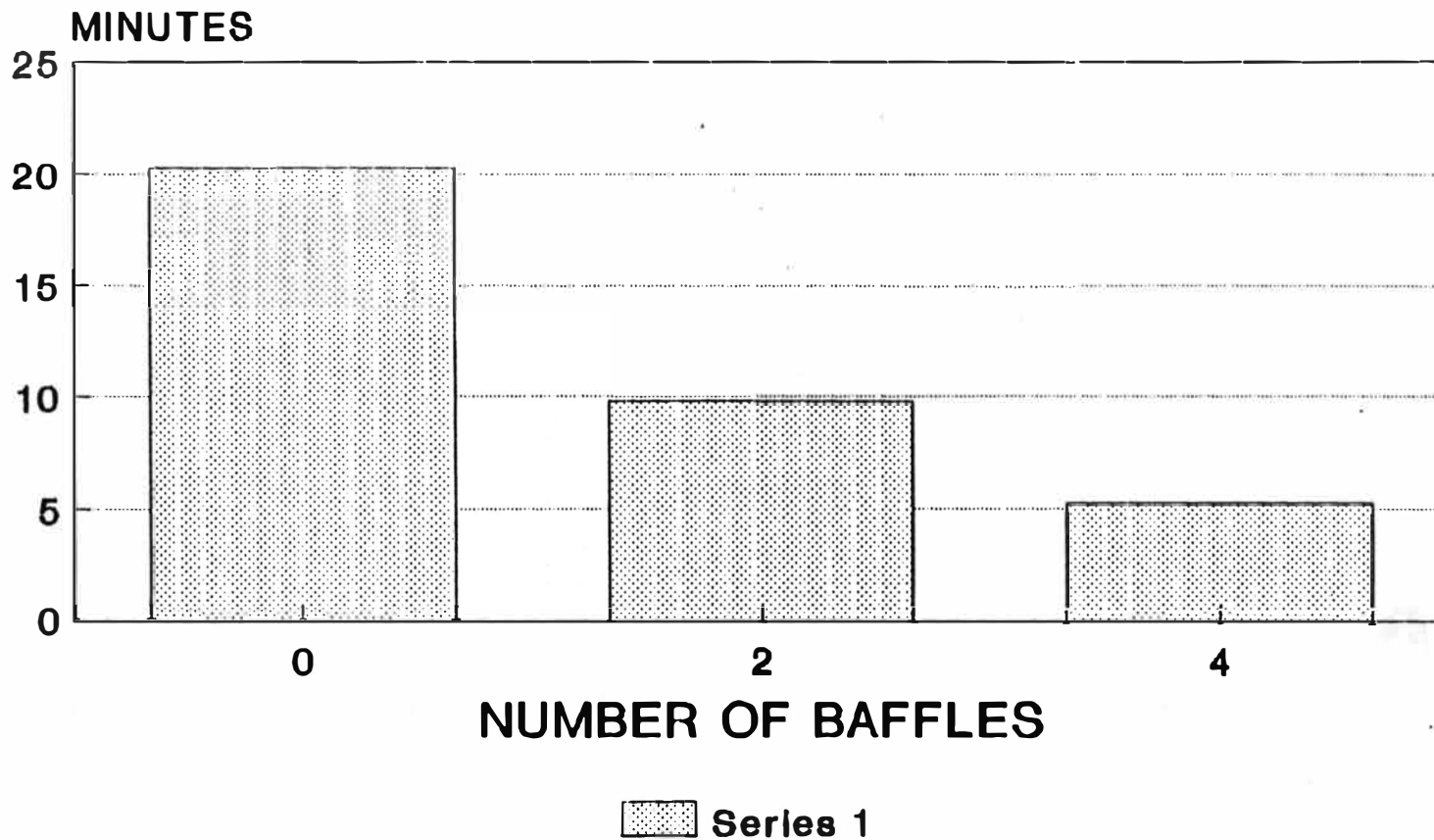
SIZE VS TIME

At 0,2,4 Baffles



RESULTS
GRAPH #1

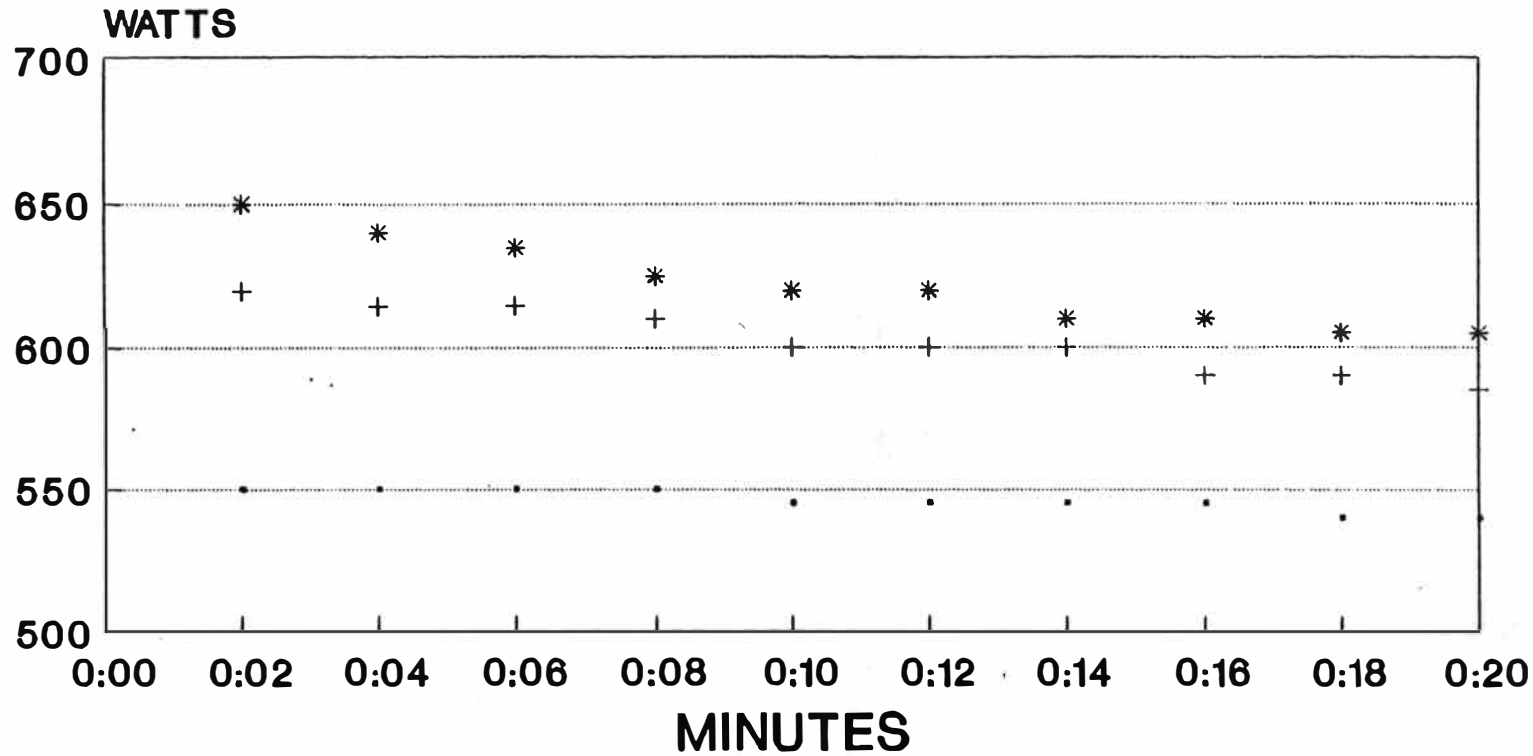
TIME TO REACH 38 μM AT 0,2,4 BAFFLES



RESULTS
GRAPH 2

POWER VS TIME

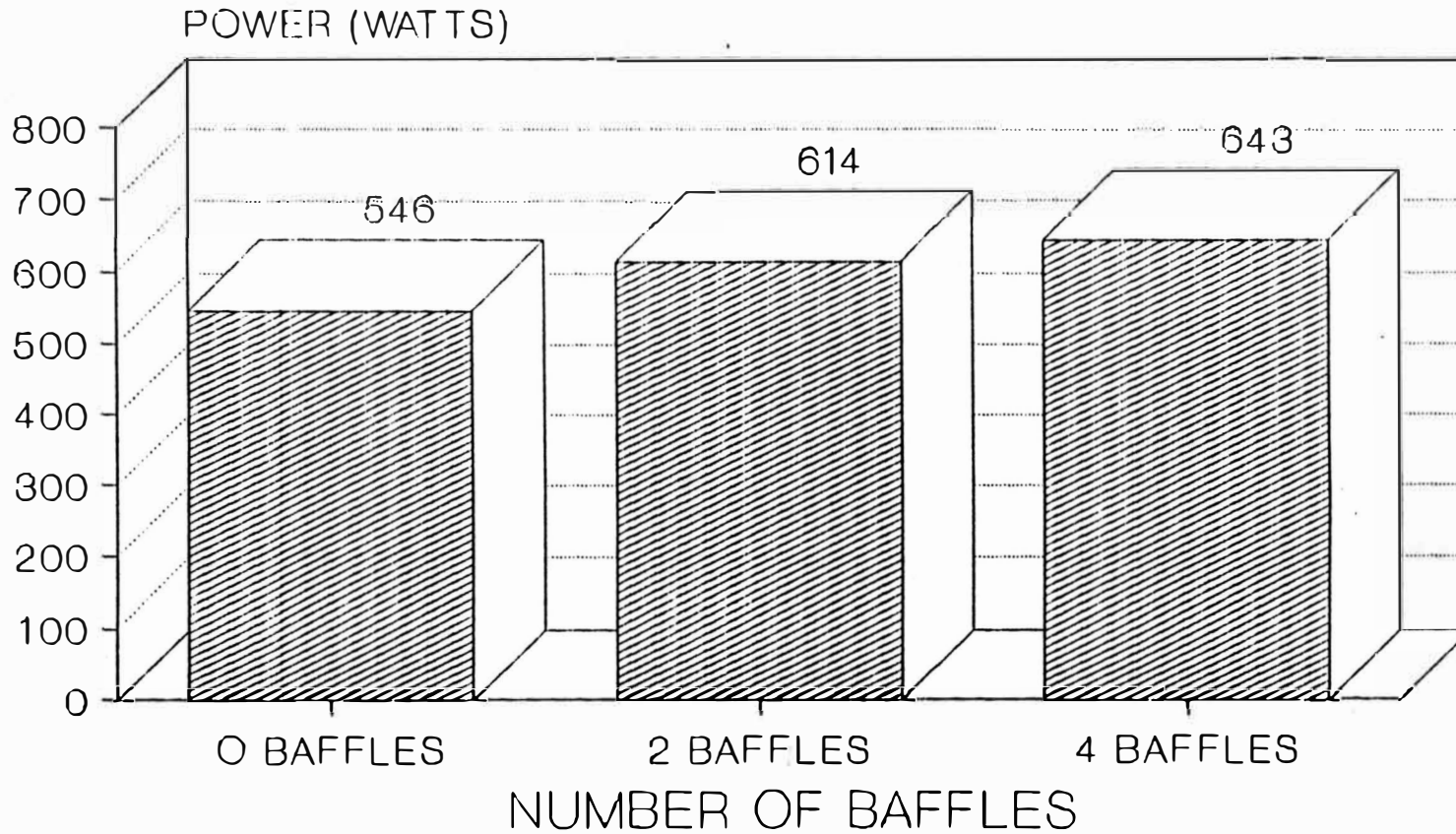
0,2,4, BAFFLES



· 0 BAFFLES + 2 BAFFLES * 4 BAFFLES

RESULTS
GRAPH 3

POWER CONSUMED

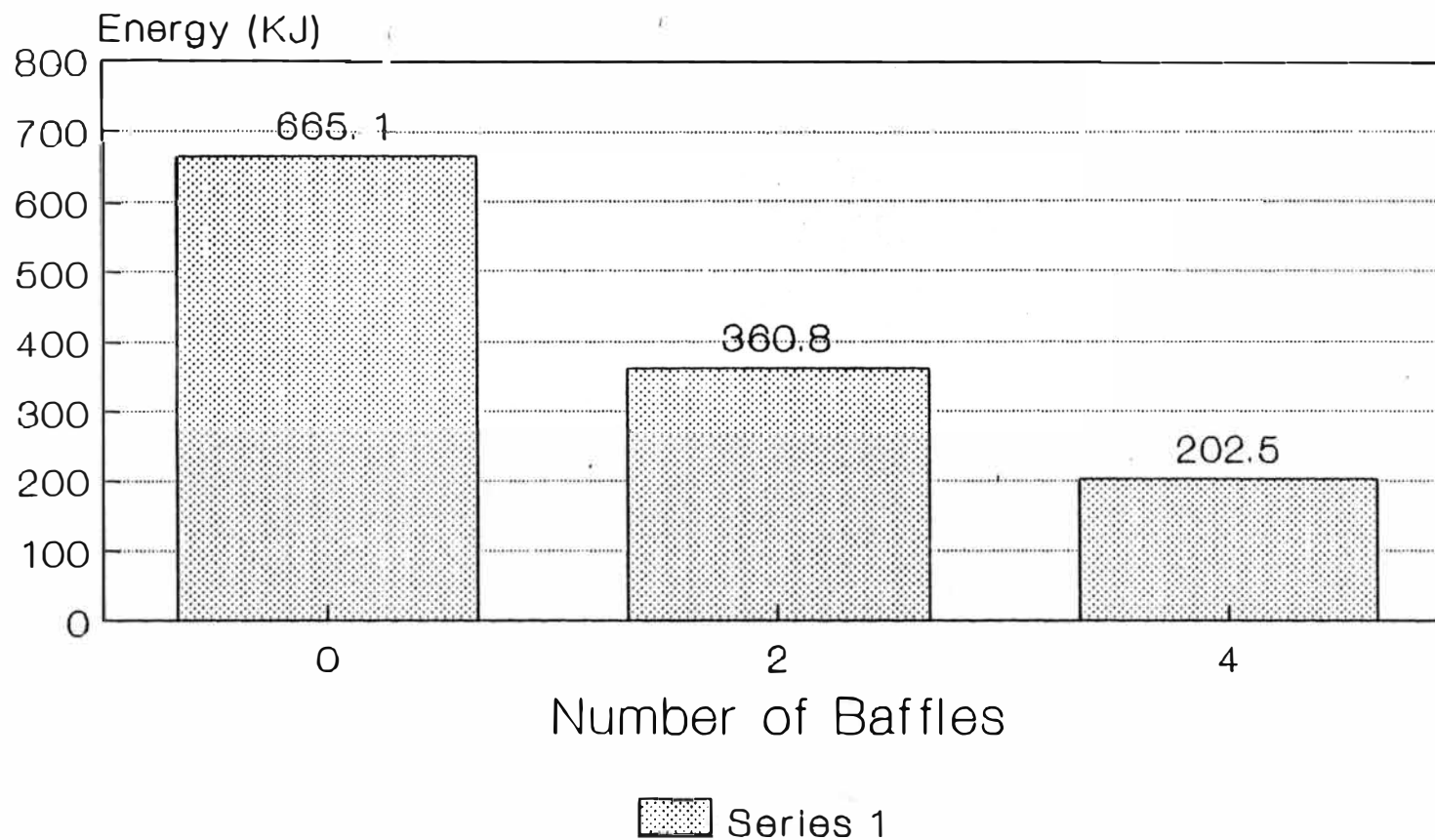


RESULTS
GRAPH 4

▨ SERIES 1

ENERGY TO OBTAIN 38uM

$$\text{Energy} = \text{Power} \times \text{Time}$$



RESULTS
GRAPH 5

TABLE #1
 STATISTICAL ANALYSIS

		<u>5 MIN</u>	<u>15MIN</u>	<u>35 MIN</u>
0 BAFFLES (uM)		.4328	.3843	.3773
CONFIDENCE BETWEEN 0 AND 2 (%)	(99.5)		(97.7)	(99.3)
2 BAFFLES	AVG	.4037	.3651	.3500
	(4023)	.4043		
		.3990		
CONFIDENCE BETWEEN 2 AND 4 (%)	(99.0)		(97.6)	(96.0)
4 BAFFLES		.3810	.3501	.3390

DISCUSSION OF RESULTS

Graph #1 shows the particle size versus time for the 0, 2, and 4 baffle configuration. Adding baffles clearly reduced the time needed to reach the same particle size. The curves were not drawn in completely because it was not needed for the analysis. By selecting a particle size of 38 μM (this was almost the only size which fell on all three curves) the time required to reach 38 μM was determined from the graph.

The times taken off of the curves are shown in graph #2. The time required to obtain 38 μM was reduced from 20.3 to 9.8 to 5.3 minutes by using 0, 2, and 4 baffles respectively. This indicates that the agitator could be run only one fourth the time with four baffles to obtain the same size with 0 baffles. This obviously would reduce the necessary energy costs associated with dispersing the pigment.

The exact reduction in energy is calculated by multiplying the times required for each by the power required for each. The power versus time curves are shown in graph #3. This graph shows that the power consumed for the 0 baffles was relatively constant compared with the others. This value in fact was the same power consumed as when the mixer was rotating by itself, not submerged in the suspension. Once the suspension began to rotate with no baffles, the fluid had very little contact with the impeller offering very little resistance. This may explain why there was no difference between the values.

With 2 and 4 baffles the power consumed continued to drop as the dispersion continued, but the curves were shifted upward slightly, indicating more power was being consumed by adding baffles. With both of these dispersions the fluid came in intimate contact with the impeller.

By integrating the power curves from two minutes to the time needed to reach 38 μM , the total power needed was obtained. This value is represented in graph #4. As expected the power consumed increased as baffles were added. This shows proof that adding baffles changes the resistance on the impeller which in turn increases the current flow to the motor.

The real cost difference is determined by multiplying the power required times the time required. This is displayed in graph #5. This graph shows that the total energy required to reduce the average particle size to 38 μM is reduced by more than a third by adding four baffles.

Table #1 shows the statistical confidence between the same times for the various number of baffles. These results show that there was from 96 to 99.5% confidence that the differences between the number of baffles was due to the baffles alone and not in the procedures or tests used. A t-test was used with two degrees of freedom. The testing error was determined by running three tests on the two baffle, five minute sample. From these three tests the standard deviation was calculated. Then the t-test value was calculated and the confidence level was determined. (See appendix for sample calculations)

While the operational costs of this particular configuration have been shown to be one-third lower when using four baffles, a discussion concerning the limits of this experiment are warranted.

First of all, the model used was a small scale version of an industrial application. There should be no doubt that the results may vary when the tank, impeller, and motor are scaled up to industrial sizes. This arises mainly because of the formation of eddies and the amount of turbulence present may change significantly.

There have been many studies on this subject which have all come to the same conclusion; a linear scale up is certainly not a correct assumption.

Secondly, another aspect of the cost is the capital cost. Because the time required for reaching 38uM was different for the three configurations, on practical terms the size of the tank would have to be changed to maintain the same throughput. If the time required for 4 baffles is half the time required for 2 baffles, then there will have to be twice as many tanks with 2 baffles to obtain the same throughput as with 4 baffles. Although this difference is not so neatly calculated, it is important to keep in mind.

Finally the last discussion point. Frequently the same tank used to disperse the pigment will be used to mix the pigment with all of the other coating ingrediants. When the other ingrediants such as starch, alginates, etc. are added, the coating may become 2 to 10 times its original viscosity. If the tank has four baffles, the resistance to flow may be to great for the impeller to adiquatly mix the suspension. Because of this, a lesser amount of baffles or a smaller batch size may have to be used.

CONCLUSIONS

It has been shown in this project that on a small scale version of an industrial open impeller agitator the operational cost of dispersing TiO_2 can be reduced by using baffles in the tank. The capital costs would be expected to be less for the four baffle system also.

RECOMMENDATIONS/FURTHER WORK

The nature of this project required a narrow set of conditions to test under. This includes the type of pigment, the shape factors, speed of impeller, and baffle shape. All of these parameters could be changed to see how these parameters contribute to the results.

One interesting idea arises when considering the problem of using baffles to disperse the pigment and using the same tank to mix the other ingredients. If the baffles did not extend the full height of the vessel then they would serve their purpose for the pigment dispersion. When the thickening ingredients were added the extra volume (and height) would rise above the baffles so that their effect would be lessened.

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APPENDIX A: PARTICLE SIZE AND POWER INTEGRATIONS

0 BAFFLES 5 MINUTE INTEGRATION:

28	TO	4	0	0	0
4	TO	2	100	TO 99	1 X 3
2	TO	1	99	TO 95	4 X 1.5
1	TO	.8	95	TO 92	3 X .9
.8	TO	.6	92	TO 86	6 X .7
.6	TO	.4	86	TO 68	18 X .5
.4	TO	.3	68	TO 45	23 X .35
.3	TO	.25	45	TO 29	16 X .275
.25	TO	.2	29	TO 12.5	16.5 X .225
.2	TO	.155	12.5	TO 0	12.5 X .1775
TOTAL				100	43.28
AVG SIZE					.4328

0 BAFFLES 15 MINUTE INTEGRATION:

28	TO	2	0	0	0
2	TO	1	100	TO 96.5	3.5 X 1.5
1	TO	.8	96.5	TO 94.5	2 X .9
.8	TO	.6	94.5	TO 90	4.5 X .7
.6	TO	.4	90	TO 73	17 X .5
.4	TO	.3	73	TO 48	25 X .35
.3	TO	.25	48	TO 31.5	16.5 X .275
.25	TO	.2	31.5	TO 13.5	18 X .225
.2	TO	.155	13.5	TO 0	13.5 X .1775
TOTAL				100	38.43
AVG SIZE					.3843

0 BAFFLES 35 MINUTE INTEGRATION

28	TO	2	0	0	0
2	TO	1	100	TO 97	3 X 1.5
1	TO	.8	97	TO 94.75	2.25 X .9
.8	TO	.6	94.75	TO 89	5.75 X .7
.6	TO	.4	89	TO 73	16 X .5
.4	TO	.3	73	TO 51	22 X .35
.3	TO	.25	51	TO 35	16 X .275
.25	TO	.2	35	TO 16	19 X .225
.2	TO	.15	16	TO 0	16 X .175
TOTAL				100	37.73
AVG SIZE					.3773

APPENDIX A: PARTICLE SIZE AND POWER INTEGRATIONS

2 BAFFLES 5 MINUTE 1RST TEST

28	TO	2	0	0	0
2	TO	1	100 TO 96.5	3.5 X 1.5	5.25
1	TO	.8	96.5 TO 94	2.5 X .9	2.25
.8	TO	.6	94 TO 87	7 X .7	4.9
.6	TO	.4	87 TO 67	20 X .5	10
.4	TO	.3	67 TO 45	22 X .35	7.7
.3	TO	.25	45 TO 29.5	15.5 X .275	4.2625
.25	TO	.2	29.5 TO 12	17.5 X .225	3.9375
.2	TO	.145	12 TO 0	12 X .1725	2.07
TOTAL				100	40.37
AVG SIZE					.4037

2 BAFFLES 5 MINUTE 2ND TEST

28	TO	2	0	0	0
2	TO	1	100 TO 96.5	3.5 X 1.5	5.25
1	TO	.8	96.5 TO 94	2.5 X .9	2.25
.8	TO	.6	94 TO 87	7 X .7	4.9
.6	TO	.4	87 TO 67	20 X .5	10
.4	TO	.3	67 TO 44.5	22.5 X .35	7.875
.3	TO	.25	44.5 TO 29	15.5 X .275	4.2625
.25	TO	.2	29 TO 12	17 X .225	3.825
.2	TO	.145	12 TO 0	12 X .1725	2.07
TOTAL				100	40.43
AVG SIZE					.4043

2 BAFFLES 5 MINUTE 3RD TEST

28	TO	2	0	0	0
2	TO	1	100 TO 97	3 X 1.5	4.5
1	TO	.8	97 TO 94.5	2.5 X .9	2.25
.8	TO	.6	94.5 TO 87.5	7 X .7	4.9
.6	TO	.4	87.5 TO 67	20.5 X .5	10.25
.4	TO	.3	67 TO 45	22 X .35	7.7
.3	TO	.25	45 TO 29	16 X .275	4.4
.25	TO	.2	29 TO 12	17 X .225	3.825
.2	TO	.145	12 TO 0	12 X .1725	2.07
TOTAL				100	39.895
AVG SIZE					.39895

APPENDIX A: PARTICLE SIZE AND POWER INTEGRATIONS

2 BAFFLES 15 MINUTE INTEGRATION

28	TO	2	0	0	0
2	TO	1	100	TO 97	3 X 1.5
1	TO	.8	97	TO 94.5	2.5 X .9
.8	TO	.6	94.5	TO 90	4.5 X .7
.6	TO	.4	90	TO 76	14 X .5
.4	TO	.3	76	TO 56	20 X .35
.3	TO	.25	56	TO 40	16 X .275
.25	TO	.2	40	TO 17.5	22.5 X .225
.2	TO	.16	17.5	TO 0	17.5 X .18
TOTAL				100	36.51
AVG SIZE					.3651

2 BAFFLES 35 MINUTE INTEGRATION

28	TO	2	0	0	0
2	TO	1	100	TO 97	3 X 1.5
1	TO	.8	97	TO 95	2 X .9
.8	TO	.6	95	TO 90.5	4.5 X .7
.6	TO	.4	90.5	TO 78.5	12 X .5
.4	TO	.3	78.5	TO 61	17.5 X .35
.3	TO	.25	61	TO 44	17 X .275
.25	TO	.2	44	TO 22	22 X .225
.2	TO	.145	22	TO 0	22 X .1725
TOTAL				100	34.995
AVG SIZE					.3500

APPENDIX A: PARTICLE SIZE AND POWER INTEGRATIONS

4 BAFFLES 5 MINUTE INTEGRATION

28	TO	2		0		0		0
2	TO	1	100	TO	96.5	3.5	X	1.5
1	TO	.8	96.5	TO	94.5	2	X	.9
.8	TO	.6	94.5	TO	90	4.5	X	.7
.6	TO	.4	90	TO	73.5	16.5	X	.5
.4	TO	.3	73.5	TO	49.5	24	X	.35
.3	TO	.25	49.5	TO	32	17.5	X	.275
.25	TO	.2	32	TO	13.5	18.5	X	.225
.2	TO	.14	13.5	TO	0	13.5	X	.170
TOTAL					100	38.121		
AVG SIZE						.3812		

4 BAFFLE 15 MINUTE INTEGRATION

28	TO	2		0		0		0
2	TO	1	100	TO	97	3	X	1.5
1	TO	.8	97	TO	95	2	X	.9
.8	TO	.6	95	TO	90.5	4.5	X	.7
.6	TO	.4	90.5	TO	78.5	12	X	.5
.4	TO	.3	78.5	TO	61	17.5	X	.35
.3	TO	.25	61	TO	44	17	X	.275
.25	TO	.2	44	TO	21.5	22.5	X	.225
.2	TO	.145	21.5	TO	0	21.5	X	.1725
TOTAL					100	35.022		
AVG SIZE						.3502		

4 BAFFLE 35 MINUTE INTEGRATION

28	TO	2		0		0		0
2	TO	1	100	TO	98	2	X	1.5
1	TO	.8	98	TO	96	2	X	.9
.8	TO	.6	96	TO	91.5	4.5	X	.7
.6	TO	.4	91.5	TO	79	12.5	X	.5
.4	TO	.3	79	TO	61	18	X	.35
.3	TO	.25	61	TO	44	17	X	.275
.25	TO	.2	44	TO	21.5	22.5	X	.225
.2	TO	.14	21.5	TO	0	21.5	X	.17
TOTAL					100	33.893		
AVG SIZE						.3389		

APPENDIX A: PARTICLE SIZE AND POWER INTEGRATIONS

POWER DATA:

MIN	0 BAFFLES	2 BAFFLES	4 BAFFLES
2	550	620	650
4	550	615	640
6	550	615	635
8	550	610	625
10	545	600	620
12	545	600	620
14	545	600	610
16	545	590	610
18	540	590	605
20	540	585	605

INTEGRATIONS:

0 BAFFLES:

2 TO 8	550 TO 550	6 X 550	= 3300
8 TO 10	550 TO 545	2 X 547.5	= 1095
10 TO 16	545 TO 545	6 X 545	= 3270
16 TO 18	545 TO 540	2 X 542.5	= 1085
18 TO 20	540 TO 540	2 X 540	= 1080
		9830/18	= 546.1 WATTS

2 BAFFLES:

2 TO 4	620 TO 615	2 X 617.5	= 1235
4 TO 6	615 TO 615	2 X 615	= 1230
6 TO 9	615 TO 605	3 X 610	= 1830
		4295/7	= 613.6 WATTS

4 BAFFLES:

2 TO 4	650 TO 640	2 X 645	= 1290
4 TO 5	640 TO 637.5	1 X 638.8	= 638.8
		1928.8/3	= 642.9 WATTS

APPENDIX B
SAMPLE STATISTICAL ANALYSIS

A T-test was used to determine the statistical significance of the results. This involves comparing the particle sizes of no baffles with two baffles, and two baffles with four baffles (at the same sample times). From the three samples of the two baffle, 5 minute sample the standard deviation was determined.

.4037
.4043
.3990 sigma = .0029

This sigma was then assumed to be constant and was used to determine the t values as follows:

0 baffle 5 min	.4328uM
2 baffle 5 min	.4023uM

$$t = \frac{.4328 - .4023}{.0029} = 10.5$$

This t value was compared to a t-test table and the confidence level of each comparison was determined.

APPENDIX C
OTHER CALCULATIONS

Rate for sedigraph: $\frac{211.8 (p_s - p_l)}{\text{viscosity}}$ x multiplier

$$\text{Rate} = \frac{211.8 (4.0 - .9951) \times .314}{.7679} = 260$$

Machine RPM : 5400



measure particle size by sedimentation

features

The SediGraph was developed to quickly and accurately perform size analysis of particles dispersed in a liquid. The particle diameter range of 100 to 0.1 μm is applicable to most finely powdered materials. This rugged, easy-to-operate instrument automatically plots the size analysis as an easy-to-use graph of cumulative mass percent versus particle diameter.

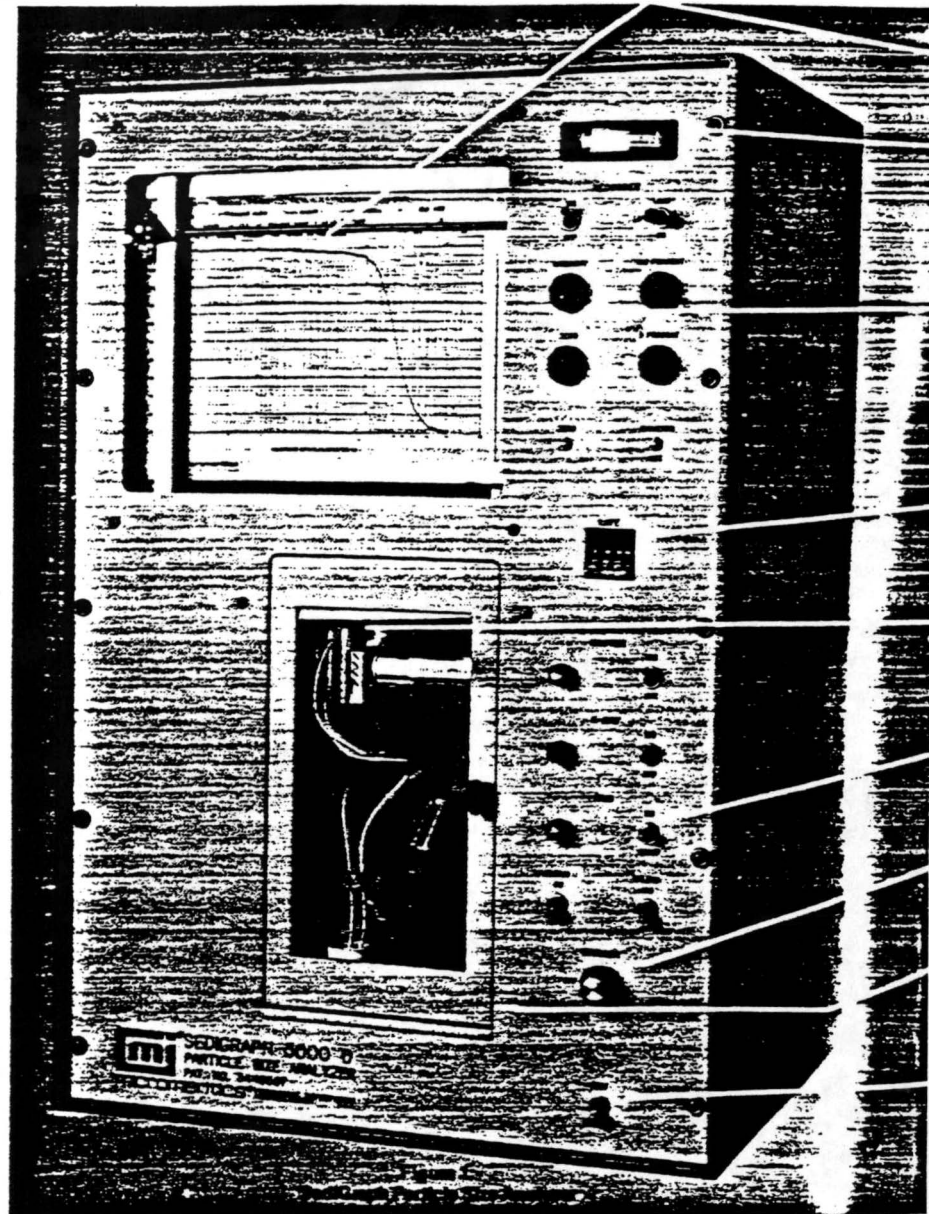
Versatile — For R & D and QC: The SediGraph measures size distribution of particles dispersed in a liquid. Sample concentration need not be exact, i.e., precisely weighed or measured. The instrument is easily programmed for any sample density as well as for different liquid densities and viscosities. A variety of sedimentation liquids, both aqueous and organic, can be used.

Automatic, Fast and Accurate: The compact, bench-top SediGraph operates unattended and resets automatically for the next analysis. A typical analysis is completed in minutes. All factors contributing errors to the analysis are minimized by careful design. Accuracy is better than $\pm 1\%$.

World-Wide Acceptance: The SediGraph Particle Size Analyzer is industry proved and used throughout the world. Many industries have accepted the SediGraph as a standard analytical instrument for particle size analysis.

Direct Data Plot — No Calculations or Calibration: A solid-state digital sequencer controls all instrument functions to continuously solve the fundamental relationships of Stokes' law, and data are automatically plotted as cumulative mass percent versus equivalent spherical diameter.

Useful Size Range: 100 to 0.1 μm equivalent spherical diameter. SediGraph data agrees with other sedimentation measurements. Highly skilled operators are not required.



High Resolution: The sedimenting sample is scanned by a narrow beam of soft x-rays which gives high resolution. Collimating slits — not the cell walls — determine the beam width. The digital sequencer moves the cell through the beam in 13,391 increments of 1.88×10^{-4} cm.

Optimal Sample Cell Design: The sample cell is completely filled

and has no liquid-air or liquid-liquid interfaces and no fragile, easily-clogged aperture. The cell gives uniform particle dispersion and allows easy flushing and clean-up.

Built-In Self-Check Features: The operator can quickly scan the sample cell to check the particle concentration base line. A rapid program check allows the instru-

Automatically, quickly & accurately

DIRECT DATA PLOT
No Data Reduction Needed

BEAM INTENSITY HEADOUT
Permits Rapid Set-Up When Changing Sedimentation Liquid and/or Sample

VERSATILE
100 to 0.1 μm Diameter Range, Precise Sample Concentration Not Needed, and Uses Aqueous and Organic Liquids

SIMPLE PROGRAMMING
FOR FAST ANALYSIS
Uses Solid-State Digital Sequencer

UNIQUE SAMPLE CELL
Gives High Resolution and Easy-to-Clean

AUTOMATIC ANALYSIS
Highly Skilled Operators Not Needed

EASY SAMPLE LOADING
Built-In Magnetic Stirrer and Sample Loading Pump

THERMALLY STABLE
Operating Compartment Temperature is Proportionally Controlled to Permit Use in Plant or Lab

QUICK PROGRAM VERIFICATION
Requires No Calibration

ment to verify proper operation in minutes.

Thermal Stability: The dispersed sample, circulating pump, sample cell and associated tubing are all contained in a temperature-controlled compartment. The analyzer is suitable for both in-plant and lab operation. A large acrylic door in the front panel provides easy access and visibility.

the technique

The SediGraph Particle Size Analyzer measures the sedimentation rates, according to Stokes' law, of particles dispersed in a liquid and automatically plots these data as cumulative mass percent versus equivalent spherical diameter, i.e., the diameter of a sphere of the same material which falls under gravity with the same velocity as the particle. The instrument uses a finely collimated beam of low energy x-rays to measure the concentration of particles. To minimize analysis time, the position of the sedimentation cell is continuously changed so that the effective sedimentation depth is decreased with time. The cell movement is coordinated with the pen movement along the X-axis of the built-in X-Y recorder such that the equivalent spherical diameter indicated at any instant corresponds to the maximum equivalent spherical diameter at the depth where the beam is making the concentration measurement. The solid-state digital sequencer in the SediGraph controls all functions of the instrument so that the fundamental relationships of Stokes' law are continuously solved, and the time required for particle size analysis is dramatically reduced.



Figure 2:
SediGraph Operating Compartment



Figure 3:
Loading a Sample into the SediGraph

the sample

The SediGraph can analyze most powders for particle size distribution in the range of 100 to 0.1 μm equivalent spherical diameter. Three general criteria must be satisfied in order to analyze a powder with the SediGraph: 1) the particles must be more dense than the liquid in which they are dispersed so they will sediment, 2) the particles must be more absorptive of x-rays than the liquid in which they are dispersed so that there is enough contrast to detect them, and 3) the particles should disperse in the sedimentation liquid.

Sample preparation involves dispersion of the powder to be analyzed in a liquid. The SediGraph can use a wide range of liquids, both aqueous and organic. Usually, a surfactant is added to the particle-liquid system to aid in dispersion of the particles and some form of shear energy is used to disperse the particles completely in the liquid. This energy can be generated by a blender, an ultrasonic bath, or just extensive stirring with a simple, mechanical stirrer.

Micromeritics offers a series of *Sedisperse Liquids* which greatly

facilitate the preparation of a dispersed sample for analysis. These Sediperse liquids are available in both aqueous and organic formulations and are designed to disperse particles at low solids concentrations. They have a built-in surfactant system and are useful in a laboratory running a variety of materials, particularly unfamiliar materials.

A beaker of dispersed sample is placed in the sample compartment and the magnetic stirrer is used to keep the sample dispersed during the loading operation. A built-in peristaltic pump is used to circulate the sample into the sample cell. After the sample is loaded into the sample cell and all the starting conditions are set into the instrument, the automatic analysis is started simply by actuating the RUN switch.

The sample compartment is maintained at constant temperature by built-in heaters and a proportional temperature control circuit. The constant temperature control, coupled with the speed of the analysis, allows the SediGraph to work reliably in the sub- μm particle diameter range.

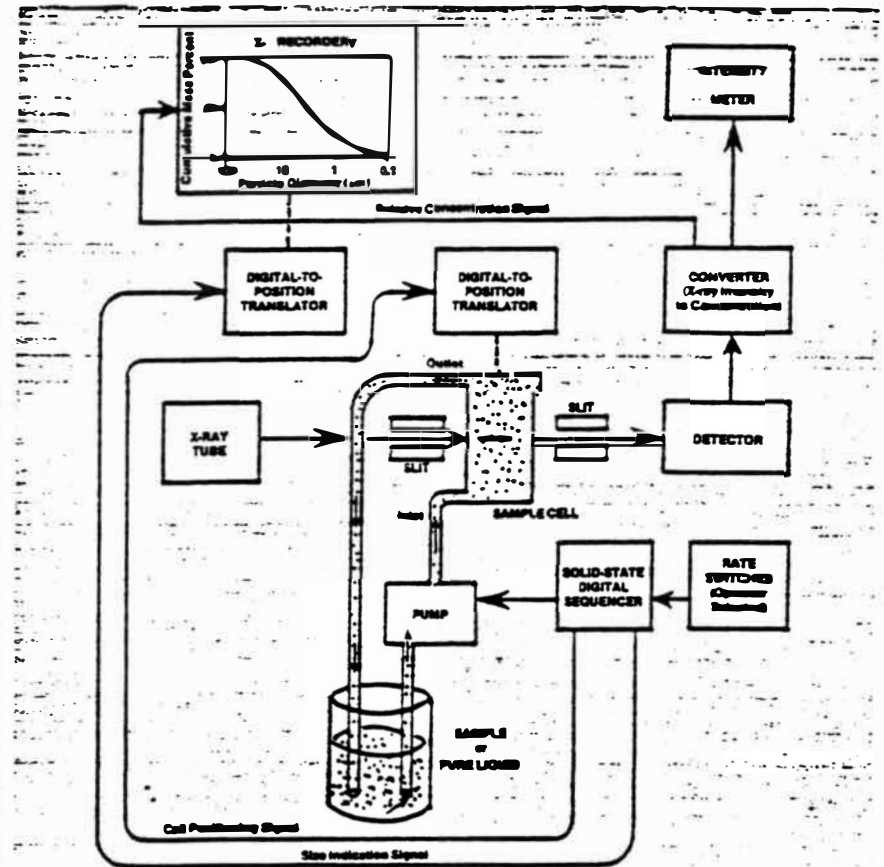
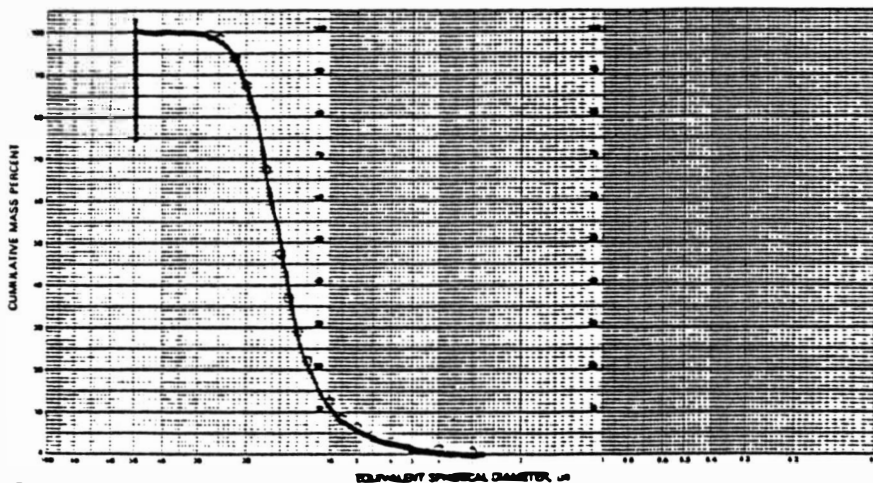


Figure 4:
SediGraph Operational Schematic

Figure 5: Calibrated glass spheres, N.B.S. Standard Reference Material 1003, are quickly analyzed. The circled points are data supplied by the National Bureau of Standards.

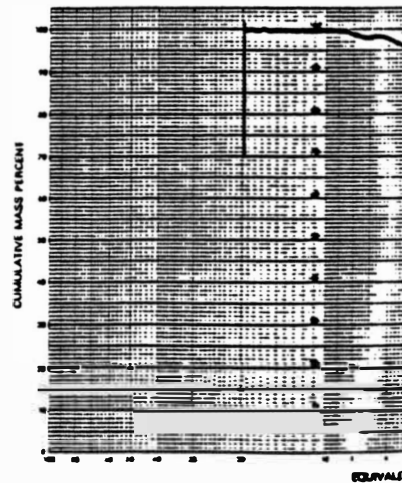


Particle Size Distribution

SAMPLE IDENTIFICATION: Calibrated Glass Spheres (N.B.S. S.R.M. 1003)
 DENSITY: 2.54 g/cc LIQUID: Deionized Water DENSITY: 0.9947 g/cc VISCOSITY: 0.7523 cp
 PREPARATION: 3-g glass spheres in 30-ml 0.05% aqueous Calgon solution—dispersed ultrasonic bath for 2 minutes

BY: SediGraph TEMPERATURE: 33 °C RATE: 111 START DIA. 50 μm

Figure 6: Metal powders are easily analyzed and larger than 10 μm diameter.



Particle Size Distribution

SAMPLE IDENTIFICATION: Titanium
 DENSITY 19.3 g/cc LIQUID: deionized water
 PREPARATION: 0.5-g W + 1 drop Romex 648 in 35-cc ϕ

BY: SediGraph TEMPERATURE: 33 °C

fast, simple operation

Micromeritics' SediGraph Particle Size Analyzer automates particle size analysis based on sedimentation and consolidates all aspects of the analysis and data reduction into one compact instrument. The rugged, reliable SediGraph provides accuracy, precision and flexibility for R&D applications — and a fast, simplified operation for quality and process control applications.

The basic design of the SediGraph is illustrated schematically in Figure 4. To perform an analysis, the operator first calculates a rate. The rate is a function of the particle density, the viscosity and the density of the liquid, and the starting particle diameter. The rate is a dimensionless number which programs the SediGraph to solve the Stokes' law relationships for the particular particle-liquid system being analyzed. The sample dispersion is prepared and placed in the cell compartment to equilibrate with the compartment temperature.

While thermal equilibrium is being

established, pure (particle-free) sedimentation liquid is pumped through the sample cell and the recorder pen is adjusted to 0% concentration on the Y-axis. Then the dispersed sample is circulated through the cell and the recorder pen is adjusted to 100% concentration. Next, the rate is switched into the instrument and the recorder pen is set to the starting diameter on the recorder X-axis.

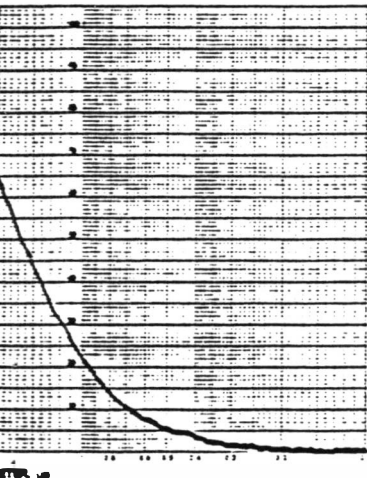
Actuation of the RUN switch stops the pump and starts an automatic programmed analysis. An initial, rate-dependent pause allows particles larger than the starting diameter to settle below the X-ray beam, and then the sample cell begins moving down through the x-ray beam such that the effective settling depth is decreased with time. The solid-state digital sequencer controls the programmed movement of the sample cell and concurrently controls movement of the recorder pen along the X-axis so that the particle size being indicated at any instant will be the largest size present at the depth

where the concentration is being measured.

A beam of low energy x-rays is collimated by the slits and passes through the sample cell to the detector. The cell is constructed so that its inlet and outlet do not interfere with the x-ray transmission. To eliminate meniscus effects, the cell is closed at the top and, in use, is completely filled with the sample dispersion.

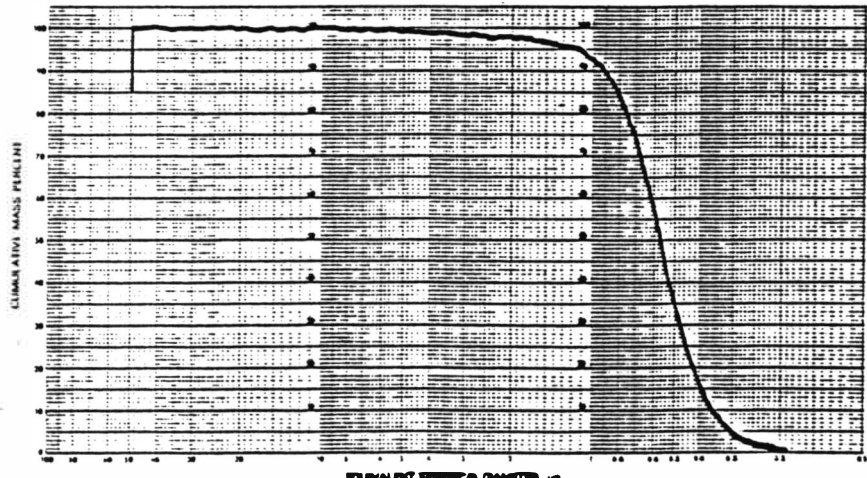
The intensity meter gives a continuous indication of x-ray transmission. This enables the operator to conveniently change the intensity level for different liquids and to adjust the sample concentration for optimum analysis conditions.

The tungsten powder has no particles



DENSITY: 0.9847 g/cc VISCOSITY: 0.7523 cp
 PREPARATION: 1 mg at high speed in blender
 RATE: 211 START DIA.: 20 μ m

Figure 7: Powders having an appreciable sub- μ m fraction are accurately analyzed. Here a titanium dioxide pigment has an average diameter of 0.58 μ m.



Particle Size Distribution
 SAMPLE IDENTIFICATION: Titanium Dioxide
 DENSITY: 4.12 g/cc LIQUID: Deionized water DENSITY: 0.9847 g/cc VISCOSITY: 0.7523 cp
 PREPARATION: 0.8-g sample in 25-ml deionized water + 0.1 wt. % TEPP. Blended in a Waring Blender for 2 min on low speed.
 BY: SediGraph TEMPERATURE: 33°C RATE: 225 START DIA.: 50 μ m

the results

The scintillation detector monitors the beam intensity transmitted through the sample cell and produces a current proportional to the pulse rate. The converter changes this current to a voltage proportional to the logarithm of the x-ray intensity. When the cell is filled with the sample, a net output signal is generated that is directly proportional to the particle concentration at the depth of the x-ray beam. This signal is fed to the X-Y recorder and moves the pen along the Y-axis to indicate percent concentration. Zero percent corresponds to pure (particle-free) liquid. The signal produced when the cell is filled with a circulating, dispersed sample is scaled to 100% concentration by adjustment of the recorder sensitivity control.

Because of the broad size range covered, the recording format is 3-cycle semi-logarithmic coordinate paper. The cell position is established as a function of time from the start of sedimentation by the solid-state digital sequencer that simultaneously positions the recorder pen along the X-axis to indicate the particle size corresponding, per Stokes' law, to the instantaneous values of settling distance and time. The digital sequencer has one output mechanically coupled, through a digital-to-position translator, to the cell positioning mechanism and another output, also mechanically coupled through a digital-to-position translator, producing the required displacement on the X-axis of the recorder as a function of time. Both outputs can be simultaneously adjusted to account for different particle and liquid densities and liquid viscosity by switching in a calculated rate appropriate for the particular particle-liquid system being analyzed.

options & accessories

Matched cells are available to permit easy switching between immiscible liquids without requiring disassembly, cleaning and reassembly of the sample cell. This is particularly desirable when doing many analyses on a variety of materials.

Auxiliary Output signals are available for interfacing to an external computer or data processor. Three BNC connectors are provided on the rear panel of the SediGraph. The INITIALIZE output provides a positive 2.5 volt signal into a 100 ohm load. It is positive as long as the operator holds the Run switch in the ON position. This pulse is used as a signal to reset the X-axis counter and clear the Y-axis data buffer to receive new data. The CHART PULSE output provides 6,000 .1-ms pulses of 2 volts amplitude into 100 ohms. If the pulses are counted by an external counter (or interrupt driven software), then the particle diameter at any instant can be determined by a simple formula. The ANALOG Y-AXIS output provides a 0 to 2.5 volt, high impedance, positive output signal to indicate the percent of particles finer than the present diameter.

The three-cycle option permits a choice of 1, 2 or 3 cycles on the X-

axis (particle size). The operator can choose either 1-cycle (100 to 10 μm E.S.D.), 2-cycles (100 to 1 μm E.S.D.) or 3-cycles (100 to 0.1 μm E.S.D.) data presentation. A selection switch is located on the front panel.

Glove Box Accessories are available for operation with materials exhibiting both low-level and high-level radiation. In addition, the low-level glove box accessory can be used with toxic materials and materials requiring an inert atmosphere. All the operating controls, the digital sequencer and the X-Y recorder are used outside the glove box; only the X-ray source, the detector, the sample cell and its operating mechanism, and the high voltage power supply are within the glove box.

Most magnetically susceptible materials may be analyzed with the SediGraph 5000D by slightly modifying the procedure. The inlet and outlet tubes leading to the pump and from the cell must be routed away from the built-in magnetic stirrer to a sample container placed some distance from the stirrer magnet. The Ultrasonic Dispersion Accessory, MIC P/N: 500/58901/01, is available for this purpose. Following dispersion, such materials are analyzed normally, except for use of the remote sample system. The ultrasonic bath also is very useful for routine sample dispersion and cell clean-up.

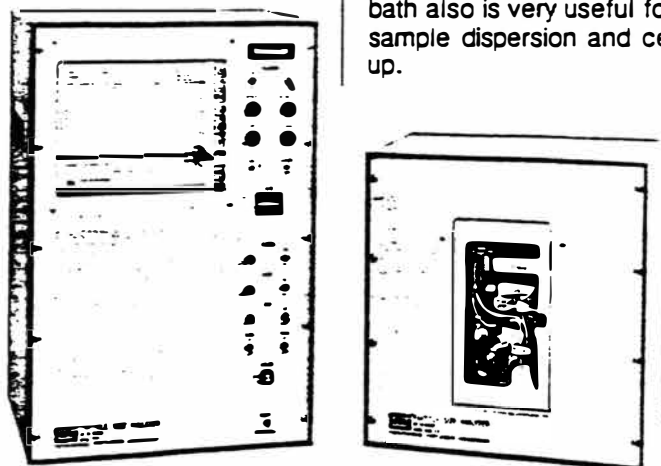


Figure 8: SediGraph with Glove Box Accessory

applications and service support

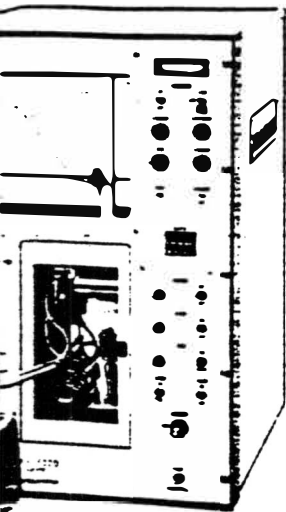


Figure 9:
SediGraph with
Ultrasonic Dispersion Accessory

Micromeritics has a talented group of professional instrument people for responding quickly to your particle analysis needs. Our years in the industry have allowed us to build a well-trained service force to give you the quickest possible service and warranty coverage with a minimum of downtime. Their efforts are reinforced by our research and development people who are constantly striving to improve the performance and operational characteristics of our particle technology instruments.

In addition to our research, service and production personnel, Micromeritics supports a large marketing force with a well staffed

service laboratory designed to support your needs and assist you with instrument operation and optimization of your particular analysis problem.

Micromeritics is a high-technology company which has emerged as the leader in the design of professional, performance-oriented particle technology instrumentation. When your analytical needs demand investigation of the physical properties of materials, particularly particle size, be sure and call your Micromeritics representative. He will be glad to work closely with you in meeting your requirements with the aid of our trained factory people.

Particle Size Range:
100 to 0.1 μm Equivalent Spherical Diameter.

Span:
Four intermediate spans
(be used)

Starting Diameter (μm)	End of Program - Diameter (μm)
100	0.36
80	0.29
70	0.25
50	0.18
35	0.13
28	0.10

Representation:
Continuous graphical plot of "Cumulative Mass Percent"
versus "Equivalent Spherical Diameter."

Operation:
The sedimenting sample is scanned in a narrow beam —
less than 0.2% of the total distance scanned — permitting
high resolution.

Wetted Materials:
Stainless steel, Hamalite and Viton, vinyl or rubber tubing.

Sample Size:
25 to 50 ml of dispersed sample — precise concentration is
not required.

Sedimenting Liquids:
Any liquid compatible with sample cell materials and not
highly absorptive of X-rays. Typical liquids are water, glycols,
kerosene, mineral oils, alcohols, hexane, mineral spirits, etc.

Power Requirements:
115V, 60 Hz, 600W (230V, 50 Hz available)

Cabinet Size:
74H x 50W x 52D cm (29.5H x 19.5W x 20.5D inches)

Color:
Light gray with slate blue trim. Solvent resistant acrylic paint.
Engraved panel lettering filled with contrasting paint.

Weight:
68 Kg (150 lb)

SAMPLE OPERATING PROCEDURE
SEDIGRAPH 5000

1. Make sure cell is clean.
2. Set in sample of base solution (.05% Dispex N-40 in distilled H₂O), position tubes, and move flow switch to ON, and *turn X-RAY Source ON.*
3. Remove cell and rid cell compartment of all air bubbles (gentle shaking).
4. Replace cell firmly in clip holder and close compartment door.
5. Adjust the 100% PERCENT dial fully clockwise.
6. Make the following adjustments with recorder Load-Lift-On Switch in Lift position.
7. Press and hold the diameter set button until the recorder pen aligns with the selected diameter value on the graph paper. (If the desired diameter value is overshoot, move RUN switch to RESET and then back to its center OFF position. After pen has reset itself; again push Diameter Set button and hold until pen aligns with the selected diameter value on the graph paper.
8. Press and hold the Zero button while adjusting the Zero dial to align recorder pen with the zero horizontal ruling of the graph paper.
- ~~9. Turn Flow Off.~~
10. Release the Zero push button and realign the recorder pen with the zero horizontal ruling of the graph paper using the 0 percent dial.
11. Drain cell of pure liquid.
12. Set in sample to be tested; position tubes, and move Flow switch to ON.
13. Remove cell and evacuate it of all present air bubbles.
14. Replace cell and close compartment door.
15. Adjust recorder pen to align with the 100 horizontal ruling of the graph paper. (DO NOT decrease the 100 Percent dial below 500. If you have to do this, then the sample solids is too high. If the pen drops below 100 ruling of the graph paper and cannot be adjusted up enough to reach the 100 ruling, then sample solids are too low. Adjust accordingly).
16. Set in the appropriate rate using the thumb wheel RATE switch.
17. Check zero by momentarily pushing the zero button.
18. Move recorder Load-Lift-On Switch to ON.
19. Move Run switch to ON momentarily, then move back to center position (OFF).
20. Machine is now testing sample.

SAMPLE IDENTIFICATION 0 BUFFLES 5 minute

1-57

2-19

DATE

Density g/cc LIQUID

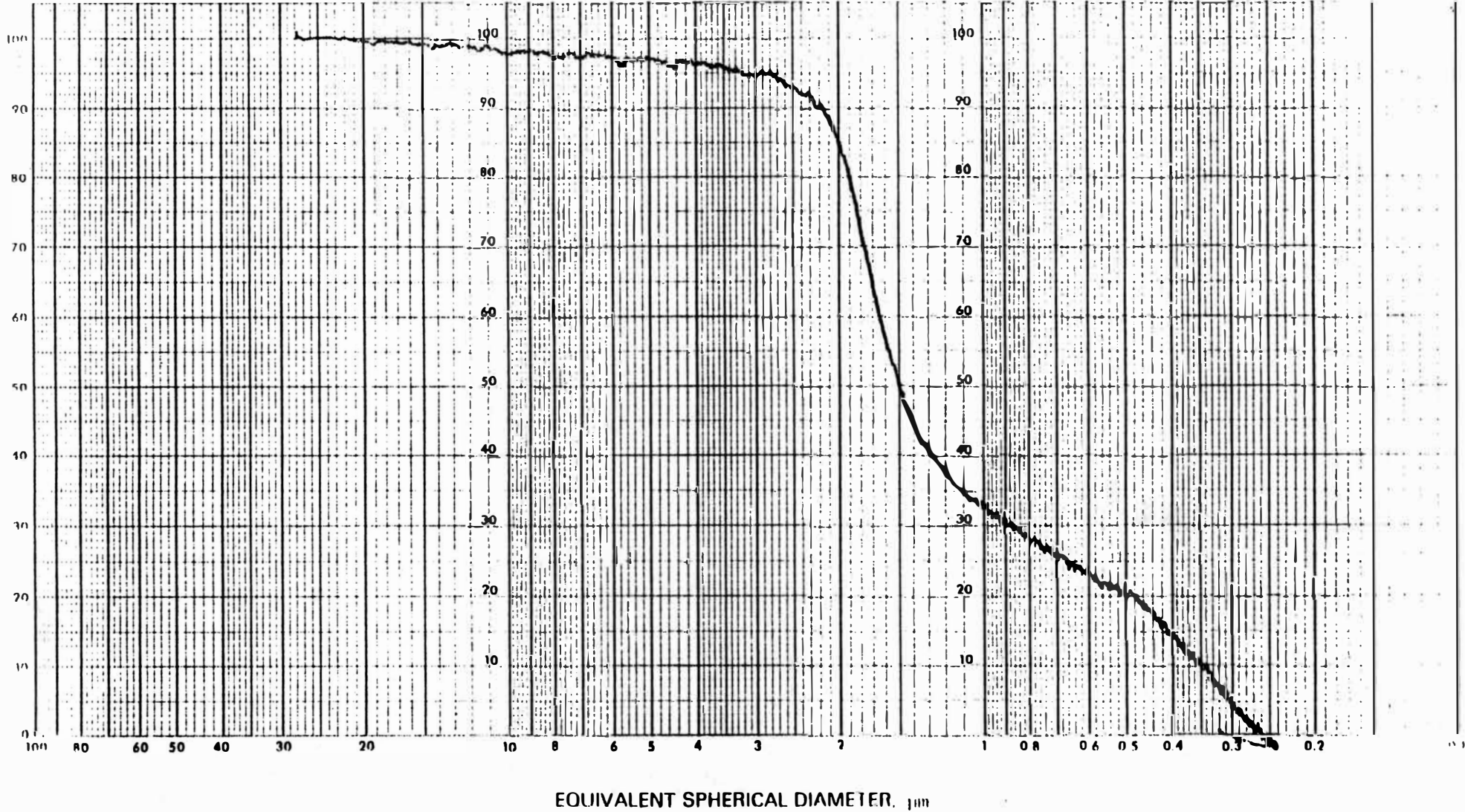
Density g/cc Viscosity cp

BY

Preparation

TEMPERATURE

RATE START DIA



RECORDING CHART

GRAPHIC CONTROLS CORPORATION
BUFFALO, NEW YORK
PRINTED IN U.S.A.