

# Ultrasonic Dispersion of Pigment In Water Based Paints

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The best known chemical effect of ultrasonic energy is doubtlessly its ability to break up solid particles present in liquids, as in an ultrasonic cleaning process. The effect is achieved by the cavitation produced by the sound energy.

This phenomenon is recognized and used to prepare a fine paint suspension by dispersing the pigment particles in the pre-mix solutions. Judicious selection of external conditions, frequency, intensity, duration of exposure, viscosity, additive, etc., permit a sharp increase in the efficiency of the ultrasonic dispersion process. A series of latex paint formulations have been prepared in a beaker comparing the properties of the finished paints with those made by the conventional mixing process.

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## INTRODUCTION

Pigment dispersion is the most important step in the production of paints. It is an expensive part of paint manufacture and, under present conditions, a vital cost factor.

Dispersion efficiency is related to the total amount of energy which can be transmitted to the system being dispersed. In high-speed disk dispersion, only 5-10% of the power input is expended in useful dispersion action.<sup>1</sup> Most of the energy is diverted into creating turbulence and/or heat. Ultrasonic dispersion is an energy efficient technique which depends essentially on cavitation.

Cavitation is the process of expansion and subsequent violent collapse of bubbles. The collapse of these cavitation bubbles leads to the generation of strong pressure pulses (shock waves), which are the main cause of dispersion of pigments in liquids. Therefore, the dispersion

efficiency must be assessed in terms of cavitation intensity, since dispersion is a direct result of cavitation.

The purpose of this paper is, first, to investigate and to discuss the effect of various parameters on cavitation. Thus, the criteria for choice of the pre-mix solution and acoustic field parameter for maximum cavitation activity can be determined. Second, a comparison of the properties and the energy consumption of these ultrasonically made paints with their high speed mixer counterparts will be presented.

## THEORY

Ultrasonic is the science of putting sound waves to work. The term "ultrasonic" is applied to high frequency vibrations higher than 20 kHz, above the threshold of audibility of the human ear. Ultrasonic action falls into two main categories—echo ranging and cavitation.

Echo ranging involves sending sound waves through air, a liquid, or a solid and using the echoes to determine distance and/or direction. Sonar is a good example.

Cavitation is an important part of acoustics, especially in regard to the physical, chemical, and biological effects of ultrasound. It has been the subject of many theoretical and experimental studies and of several reviews.<sup>2-6</sup> Predicting the incidence of cavitation is somewhat of a statistical problem which depends on the properties of the medium as well as the characteristics of the ultrasonic field.

When an ac voltage is applied to a crystal or transducer, the crystal changes shape in turn with the electric field. This is the piezoelectric effect. These continuous changes in shape or length are the pulsations which travel through the liquid. Ultrasonic wave traveling through a liquid consists of alternate compressions and rarefactions. If this wave is high enough in pressure amplitude, a phenomenon is produced, known as cavitation, which is

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the formation and collapse of bubbles. It is known to set in only when the sound pressure amplitude exceeds a certain threshold value. However, it results in a complex set of effects acting in various ways on the medium in which it takes place. The forcing function for the generation and collapse of the bubbles is the alternation time—varying pressure in the liquid. The bubble originates from some of gas “nuclei” growing primarily during the negative pressure portion of the acoustic cycle. These bubbles take few cycles to grow to what is known as the resonant size, at which point they collapse instantly and violently in part of the positive pressure portion. The collapse of large cavitation bubbles leads to the generation of strong pressure pulses which are the main cause of metal erosion, ultrasonic cleaning, and in this present work, the dispersion of pigments in liquid.

Noltingk and Neppiras<sup>7,8</sup> computed this behavior, developing an equation for the acoustically induced motion of gas-filled bubbles. They solved the equations numerically and were able to predict the growth and collapse patterns for the bubbles under various acoustic conditions.

### Types of Cavitation

There are two types of cavitation, stable and transient.<sup>3</sup> Stable cavities are simple bubbles that oscillate, often nonlinearly, around some equilibrium size. On the other hand, transient cavities generally exist for less than one acoustic cycle. They start as small bubbles and expand to at least double, and often to many times their original size, then collapse violently. Either type may contain permanent gas, generally air, or liquid vapor.

Transient cavities are responsible for most of the well-known disruptive effects, such as erosion and emulsification. Also, several important industrial applications of power ultrasonics make use of transient cavitation. These include cleaning, chemical processing, and dispersion of solids in suspension (as in paint). Stable cavitation generates important side effects. These include the initiation of surface oscillations and microstreaming in the liquid.

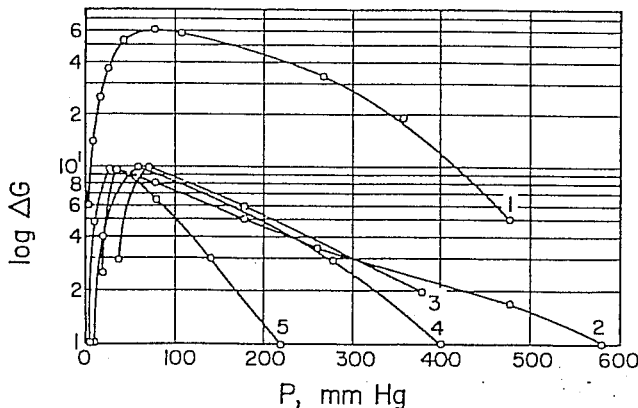


Figure 1—Dependence of the magnitude of the cavitation damage on the saturated vapor pressure of the liquid. 1—Water, 2—alcohol, 3—acetone, 4—trichloroethylene, and 5—carbon tetrachloride

### Collapse of Transient Cavities

It has been established that the cavitation obstruction of solid surfaces in contact with a liquid, as the pigment particles in the pre-mix solution, is caused by the collapse of bubbles adhering to the interfacing surface. No known solid material is able to resist the effects of cavitation destruction, although some are more resistant, others are less so. This disruptive effect is the result of the high concentrations of energy and consequent high liquid pressures and velocities.

Rayleigh,<sup>9</sup> in his paper, “On the Pressure Developed in a Liquid During the Collapse of Spherical Cavity,” first calculated the velocity  $U$  with which the bubble radius diminishes in a liquid. He assumed there was a vacuum inside the bubble and that the liquid was incompressible. Assuming that the work done in the liquid when the bubble is compressed from the maximum radius  $R_m$  to  $R$  (the residual radius) appears as kinetic energy, Rayleigh found the rate of decrease of a spherical hollow cavity:

$$U = \sqrt{\frac{2P_o}{3\rho} \left( \frac{R_m^3}{R^3} - 1 \right)} \quad (1)$$

where:

$P_o$  = external pressure and  
 $\rho$  = density of the liquid.

$U$  is considered a measure of hardness of the cavitation. From this, by a few simple manipulations, it is possible to obtain an expression for the time  $\tau$  during which collapsing occurs:

$$\tau = \left( \frac{3P}{2P_o} \right) \int_R^{R_m} \frac{R_m \cdot R^{3/2} dR}{(R_m^3 - R^3)^{1/2}} \quad (2)$$

Rayleigh calculated the time for the case when a hollow cavity completely collapses, as  $R \rightarrow 0$ :

$$\tau = 0.915 R_m \sqrt{\frac{\rho}{P_o}} \quad (3)$$

When the empty cavity collapses, a spherical shock wave is formed. The maximum pressure in this wave at a distance  $r = 1.587 R$  from the center of collapse is:

$$P_{max} = \frac{P_o}{4^{4/3}} \left( \frac{R_m}{R} \right)^3 \quad (4)$$

Actually, however, the cavity often contains gas. As the gas bubble collapses, the gas inside it is compressed more and more adiabatically. For this case, assuming that the liquid is incompressible, Noltingk and Neppiras<sup>7,8</sup> used an equation characterizing the energy of a collapsing gas bubble to find the pressure in the bubble at minimum radius:

$$Q \left[ \frac{P(y-1)}{Q} \right]^{y/y-1}$$

where  $Q$  is the pressure of the gas in the bubble at maximum radius and  $y$  is the specific heat ratio ( $C_p/C_v$ ) of the gas in the bubble. Here the surface tension forces have not been accounted for, since they are small in comparison with the forces that arise at the instant the shock wave is formed.

The maximum pressure at the collapse of a gas bubble is:

$$P_{max} = Q(R_m/R)^{3y} \quad (5)$$

The variation in pressure amplitude of the shock wave near the collapsing gas bubble is  $Z = (R_m/R)^3$ . It can be shown that the largest value is obtained when the ratio  $R_m/R$  is largest. Therefore, the pressure in the shock wave increases with increasing  $R_m$ . From calculations, the pressure  $P_{max}$  in the shock wave at collapse of a cavitation bubble can attain a large value of  $10^6$  atm.

## PARAMETERS

### Effect of Frequency

In the ultrasonic field, the nuclei expands and collapses in the phase of negative and positive pressure, respectively. The size to which a nucleus will expand, under a certain hydrostatic pressure, depends on the time available for expansion and, thus, on the frequency of the sound field. At low frequencies, there is a greater time interval between the compression and the rarefaction waves which gives the bubble more time to grow before pulsing or imploding. Therefore, lower frequencies produce a larger cavitation bubble than do high frequencies. It is evident<sup>8</sup> that as the frequency is increased,  $R_m$  decreases. The size of the bubble is directly proportional to the force of the implosion [ $P_{max} = Q(R_m/R)$ ]; therefore, as the frequency increases the pressure amplitude  $P_{max}$  at the collapse of the cavitation bubble must decrease. Figure 1 expresses the dependence of the maximum pressure on the frequency ( $P_{am} = 4 \times 10^6$ ,  $R_0 = 3.2 \times 10^{-4}$ ).<sup>7,8</sup>

### Effect of Surface Tension

Surface tension is related to the work required to expand the surface of a liquid by unit area. Its forces always work in one direction, that is, to close a cavity that is opened in a liquid.

In general, the primary effect of surface tension on cavitation occurs during the process of inception from nuclei and in the final collapse stages of the bubbles that contain very little gas. An increase in surface tension tends to reduce the maximum size of cavities. For otherwise equal conditions, surface-tension force will increase the rate of collapse of the cavities.

The greater the surface tension of the cavitation bubble, the greater will be the amount of energy released when it collapses. Liquids with high surface tension therefore will exhibit a greater cavitation action. In this respect, water medium as cavitation agents enjoys a marked advantage over the organic solvents.

### Effect of Vapor Pressure

The vapor pressure of the liquid refers to the presence of vapor in equilibrium with a pure liquid at any given temperature. It is an inherent property of a liquid and does not depend on the amount of liquid or vapor present, nor on the area of the interface between two phases. It depends only on temperature. The vapor pressure of a liquid always increases with increasing temperature.

The maximum cavitation damage depends on the vapor pressure. Babchuk<sup>10</sup> investigated the dependence of the

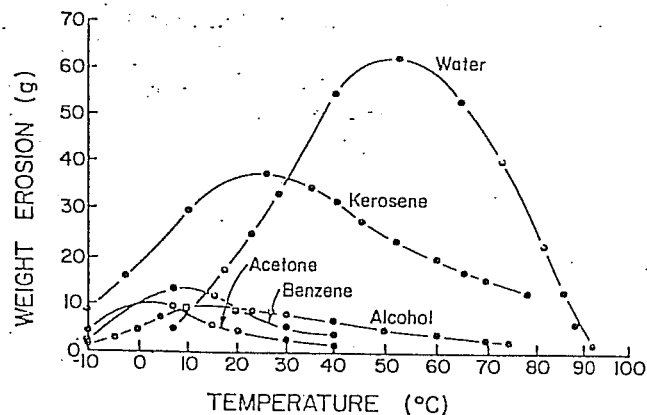


Figure 2—Cavitation erosion as a function of temperature for aluminum in various liquids

magnitude of the cavitation damage on the vapor pressure of a number of liquids, including water, ethyl alcohol, and carbon tetrachloride. He found the maximum cavitation damage for each liquid occurs at a temperature for which the vapor pressure of the liquid falls in a range of 35 to 80 mm Hg. As shown in Figure 2, his results indicate that cavitation damage is much greater in water than in any organic liquid. The maximum erosion in water was approximately six times that in ethyl alcohol and carbon tetrachloride. This factor, again, puts water in first place as a medium for sonic dispersion.

### Effect of Air and Gas Content

The violence of collapse of cavitation bubbles appears to be strongly influenced by the quantity of gas in liquids. Therefore, air content should have a substantial effect. It is established<sup>11</sup> that higher air and gas contents within individual bubbles reduce cavitation damage through the "cushioning" effect on individual bubble collapse. This fact can be explained as follows: the maximum pressure at the collapse of a gas bubble [ $P_{max} = Q(R_m/R)^3$ ] increases with increasing  $R_m$  and diminishes with increasing  $Q$ , where  $Q$  is the pressure of the gas in the bubble at maximum radius. Often the bubbles contain a larger amount of gas than one would expect on the basis of ordinary diffusion, because at the instant of expansion, when the pressure in the bubble is reduced, additional diffusion of the gas inside the bubble takes place. This is also promoted by the increase in surface of the bubble during such time.

From this expression, it is evident that the greater the solubility of the gas in the liquid, the greater the gas pressure inside the bubble. While the bubble radius is diminishing during the process of collapse, this pressure will increase, and consequently, intensity of the shock wave formed at collapse will diminish.

In organic liquids, the solubility of some gases is higher than in water. Inside the cavitation bubble, therefore, in organic liquids, a larger quantity of gas will be diffused, and the shock wave formed upon collapse of the bubble will not be as intense.

Table 3—Formula C—High Speed Dispenser; Flat Interior

Material	Formula		Total Solids	
	Lb	Gal	Lb	Gal
Water	300	36.0	—	—
AMP 95	5.0	0.64	—	—
Propylene glycol	30.0	3.47	—	—
SCT 200	25.0	2.89	5.0	0.58
Water	20.0	2.40	—	—
Triton X-100	2.0	0.22	2.0	0.22
Nopco NDW	2.0	0.28	2.0	0.28
Troysan 192	4.0	0.51	4.0	0.51
<b>Mix, then add:</b>				
Ti-Pure R960	200.0	6.4	200	6.4
Atomite	375.0	16.58	375.0	21.23
<b>Grind:</b>				
Nopco NDW	2.0	0.28	2.0	0.28
E.B. solvent	30.0	3.99	—	—
<b>Mix:</b>				
Amsco 3077	231.0	25.67	127	12.96
Water	47.0	5.67	—	—
	<u>1273</u>	<u>105</u>	<u>717</u>	<u>37.81</u>

Grind = 6.  
Grind time = 6 min.  
Watts = 6.9 AMP × 122 V.

$$\text{Energy requirement} = (6.9 \times 122) \frac{W}{\text{gal}} \times 1.0 \times 6 \text{ min} \times \frac{1}{60,000}$$

$$= .08418 \text{ (kW-hr)/gal.}$$

kept constant at the following values: N=250 W, duration t=90 sec, and content = 200 g water and 117 g TiO<sub>2</sub> in 400 mL beaker. The temperature of the ultrasonically excited fluid was of decisive importance in dispersion action. Its effect can be seen from Figure 4. It took half the time by increasing the water temperature to 45°C. This can be explained by the fact that between 45°-47°C, the vapor pressure of water is 72-80 mm Hg, where the

Table 4—Formula A—Ultrasonic; Semi-Gloss Interior

Material	Formula		Total Solids	
	Lb	Gal	Lb	Gal
Water	323.41	38.81	—	—
Propylene glycol	30.0	3.47	—	—
E. B. solvent	30.0	3.99	—	—
AMP 95	4.0	0.51	—	—
<b>Mix, then add:</b>				
Ti-Pure R-900	250.0	7.3	250	7.3
<b>Disperse, then add:</b>				
SCT 200	25.0	2.89	5.0	0.58
Water	20.0	2.40	—	—
Triton X-100	2.0	0.22	2.0	0.22
Troysan 192	4.0	0.51	4.0	0.45
Nopco NDW	4.0	0.56	4.0	0.56
Amsco 3077	400	44.44	220	22.45
	<u>1092.41</u>	<u>105.1</u>	<u>485.0</u>	<u>31.6</u>

Grind = 8.  
Grind paste = × 1/2.  
Watts = 2.2 AMP × 122 V.  
Grinding time = 3/4 min for 400 mL beaker batch.

$$\text{Energy requirement} = (2.2 \times 122) \frac{W}{\text{gal}} \times 2 \times 3/4 \text{ min} \times \frac{1.0 \text{ K-hr}}{60,000}$$

$$= 0.00671 \text{ (kW-hr)/gal.}$$

Table 5—Formula B—Ultrasonic; Flat Exterior

Material	Formula		Total Solids	
	Lb	Gal	Lb	Gal
Water	324	38.88	—	—
Propylene glycol	30.0	3.47	—	—
E. B. solvent	30.0	3.99	—	—
AMP 95	5.0	0.64	—	—
<b>Mix, then add:</b>				
Ti-Pure R-900	250.0	7.3	250	7.3
Atomite	205.0	9.11	205	9.11
<b>Disperse, then add:</b>				
SCT 200	25.0	2.89	5.0	0.58
Water	20.0	2.40	—	—
Triton X-100	2.0	0.22	—	—
Troysan 192	4.0	0.51	4.0	0.45
Nopco NDW	4.0	0.56	4.0	0.56
Amsco 3077	362	40.22	199	20.32
	<u>1261</u>	<u>110.19</u>	<u>667</u>	<u>38.32</u>

Grind = 6.  
Grind paste = × 0.5.  
Watts = 2.2 AMP × 122 V.  
Grinding time = 1.0 min for 400 mL beaker batch.

$$\text{Energy requirement} = (2.2 \times 122) W \times 2 \times 1 \text{ min} \times \frac{1}{60,000}$$

$$= 0.008947 \text{ (kW-hr)/gal.}$$

maximum cavitation damage for water occurs at vapor pressure of 80 mm Hg, as shown in Figure 2. Also, as the temperature increases, the solubility of gases located in the liquid decreases, and as a consequence of this, the intensity of the shock wave which is formed when a cavitation bubble collapses is enhanced. However, a rise in temperature also brings about a lowering of the surface tension of the liquid and an increase in its saturation vapor pressure, both of which as previously explained diminish cavitation.

Table 6—Formula C—Ultrasonic; Flat Interior

Material	Formula		Total Solids	
	Lb	Gal	Lb	Gal
Water	367	44.04	—	—
Propylene glycol	30.0	3.47	—	—
E. B. solvent	30.0	3.99	—	—
AMP 95	5.0	0.64	—	—
<b>Mix, then add:</b>				
Ti-Pure R-900	200.0	6.4	200	6.4
Atomite	375	16.58	375	16.58
<b>Disperse, then add:</b>				
SCT 200	25.0	2.89	5.0	0.58
Water	20.0	2.40	—	—
Triton X-100	2.0	0.22	2.0	0.22
Troysan 192	4.0	0.51	4.0	0.45
Nopco NDW	4.0	0.56	4.0	0.56
Amsco 3077	231	25.67	127	12.96
	<u>1293</u>	<u>107.37</u>	<u>717</u>	<u>37.75</u>

Grind paste = × 0.5.  
Watts = 2.2 AMP × 122 V.  
Dispersion time = 1.5 min for 400 mL beaker batch.  
Grinding reading = 6.

$$\text{Energy requirement} = (2.2 \times 122) \frac{W}{\text{gal}} \times 2 \times 1.5 \text{ min} \times \frac{1}{60,000}$$

$$= 0.01342 \text{ (kW-hr)/gal.}$$

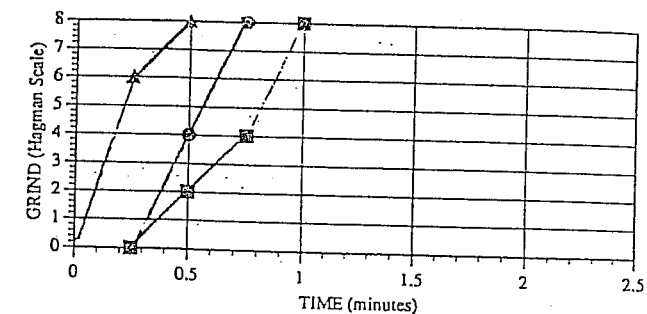
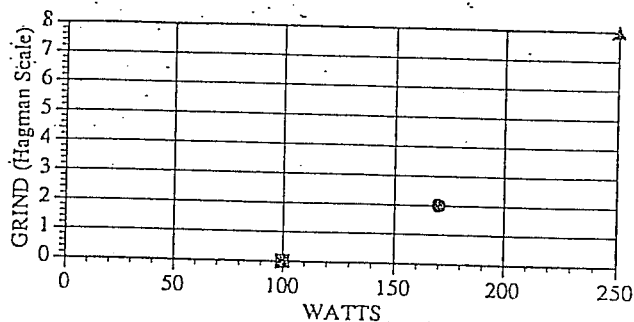


Figure 3—Grind as a function of power at constant time (1 1/2 min). ■—100 W, ●—170 W, and ▲—250 W

Figure 7—Effect of AMP 95. ■—100 W, ●—170 W, and ▲—250 W

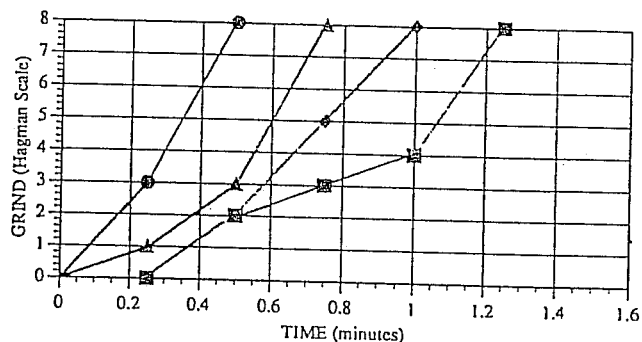


Figure 4—Effect of temperature. ■—25°C, ●—45°C, ▲—65°C, and ◆—85°C

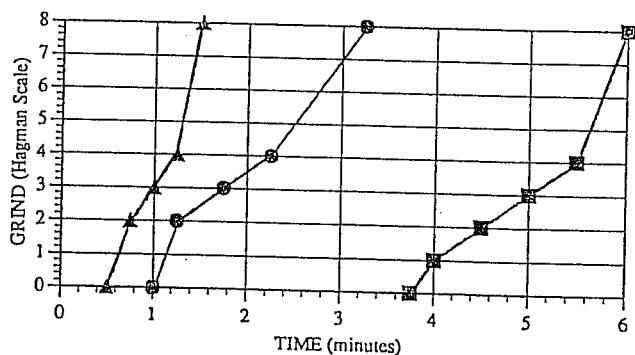


Figure 5—Effect of duration of time. ■—100 W, ●—170 W, and ▲—250 W

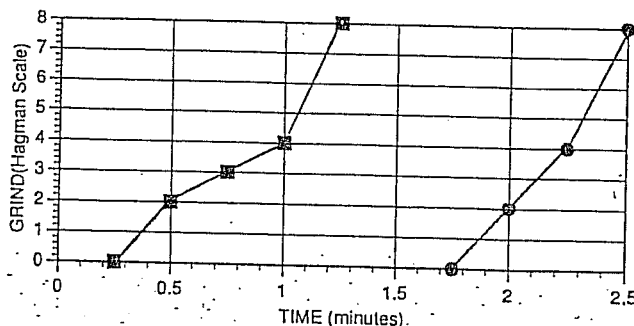


Figure 6—Effect of viscosity. ■—System A, water + TiO<sub>2</sub>, 250 W; ●—system B, water + SCT + TiO<sub>2</sub>, 250 W

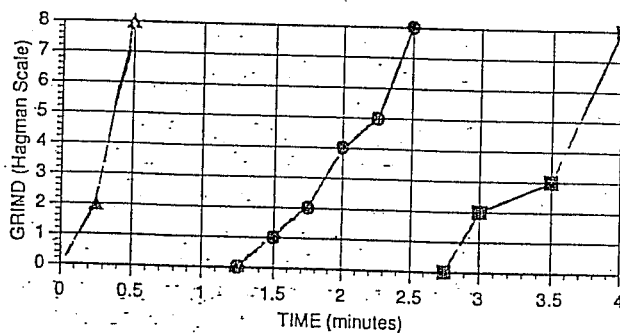


Figure 8—Effect of E. B. solvent, 116 g TiO<sub>2</sub>. ■—100 W, ●—170 W, and ▲—250 W

Table 1—Formula A—High Speed Disperser; Semi-Gloss Interior

Material	Formula		Total Solids	
	Lb	Gal	Lb	Gal
Water	130.0	15.6	—	—
AMP 95	4.0	0.51	—	—
Propylene glycol	30.0	3.47	—	—
SCT 200	25.0	2.89	5.0	0.58
Water	20.0	2.40	—	—
Triton® X-100; pre-mix	2.0	0.22	2.0	0.22
Nopco® NDW	2.0	0.28	2.0	0.22
Troysan® 192	4.0	0.51	4.0	0.45
Mix:				
Ti-Pure® R-900	250	7.3	250	7.3
Grind:				
Nopco NDW	2.0	0.28	2.0	0.28
E.B. solvent	30.0	3.99	—	—
Mix:				
Amsco® 3077	400	44.44	220	22.45
Water	193	23.21	—	—
	1092	105.1	485.0	31.5

Grind = 8.

Grind paste = x 3.

Watts = 6.9 AMP x 121 V.

Grinding time = 5 min.

$$\text{Energy requirement} = (6.9 \times 121) \frac{\text{W}}{\text{gal}} \times \frac{1}{3} \times 5 \text{ min} \times \frac{1.0 \text{ kW-hr}}{60,000 \text{ W-min}} = 0.02319 \text{ (kW-hr)/gal.}$$

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Table 7—Wet Properties

Formula	Disp. Process	%PVC	Specific Gravity		Viscosity		pH
			calc. (lb/gal)	meas.	48 hr (K.U.)	1 mos. (K.U.)	
A.....	conv.	23.17	10.41	10.53	95	99	9.10
	ultra.	23.17	10.41	10.53	95	100	9.00
B.....	conv.	42.51	11.28	11.52	101	108	9.06
	ultra.	42.51	11.28	11.53	100	105	9.02
C.....	conv.	60.78	12.12	12.30	114	116	9.02
	ultra.	60.78	12.12	12.25	115	115	8.96

Figure 2 illustrates the results of measurements of cavitation erosion, which represent cavitation damage, as a function of temperature for a number of liquids. The energy released by cavitation will generally, at first, increase with temperature, then pass through a peak, and finally decrease as the temperature increases still further.

To sum up, the intensity of the cavitation damage to a solid surface depends upon the liquid which is used, and there is a substantial dependence of cavitation damage upon the temperature that the liquid obtains.

### Effect of Duration of Time Test

In the series of experiments to determine how the degree of dispersion depended on the duration of the test, all parameters were kept constant at the following values: content = 200 g H<sub>2</sub>O and 117 g TiO<sub>2</sub> in 400 mL beaker, and power N = 100 W, 170 W, or 250 W. The starting temperature = 25°C, f = 20 kHz. Figure 5 shows the degree of dispersion as a function of the duration time t. It shows that the dispersion effect is weakest initially and then becomes gradually more effective. All the grind readings increase from 4.0 to 8.0 in 30 sec. This can be explained by the fact that there will be a greater number of nuclei at the boundary between the liquid and the pigments, since more surface areas have been exposed. Cavitation depends very strongly on the population and size spectrum of nuclei in the liquid. Another important factor is the temperature increase of the system, which increases the intensity of cavitation, as explained previously.

### Effect of Viscosity

In two experiments to determine how the degree of dispersion depended on the viscosity of the solution, the parameters were kept constant at the following values: power = 250 W, starting temperature = 25°C, and f = 20

kHz. System "A" content = 200 g H<sub>2</sub>O (= 0.9 CPS) and 160 g TiO<sub>2</sub> in a 400 mL beaker. System "B" content = 188.3 g H<sub>2</sub>O + 11.7 g UCAR<sup>®</sup> thickener SCT-200 (= 600.0 cps) and 160 g TiO<sub>2</sub> in a 400 mL beaker. System B takes 50% more time to achieve grind of reading of 8.0 as shown in Figure 6. The effect of viscosity is clear in that it must, at least to some extent, produce dampening and loss of mechanical energy during the growth and collapse process. Consequently, it would be expected that increase in viscosity will decrease the maximum cavity size and the ratio of growth and collapse.

### Effect of Added Amine

In the series of experiments to determine how the degree of dispersion was affected by adding a small percentage of amine dispersant aid, (for example, AMP 95), all parameters were kept constant at the following values: f = 20 kHz, initial temperature = 25°C; and power N = 100 W, 170 W or 250 W. System A content values were: 200 g H<sub>2</sub>O and 116 g TiO<sub>2</sub> in a 400 mL beaker. System B content values were: 198 g H<sub>2</sub>O + 1.87 g AMP 95 + 116 g TiO<sub>2</sub> in a 400 mL beaker.

Figure 7 shows a drastic reduction of time to achieve a grind of 8:0, especially in the low intensities. AMP 95 provides excellent dispersing action because of its chemical structure, comprising an amino group that absorbs on the TiO<sub>2</sub> particle surface and a hydroxyl tail that seeks out the water phase.

### Effect of Ethylene Glycol Butyl Ether

In the series of experiments to determine how the degree of dispersion was affected by adding seven percent ethylene glycol butyl ether, all parameters were kept constant at the following values: f = 20 kHz; initial temperature = 25°C; and power N = 100 W, 170 W, or 250 W. System A content values were: 200 g H<sub>2</sub>O + 116 g TiO<sub>2</sub> in a 400 mL beaker. System B content values were: 186 g H<sub>2</sub>O + 14 g ethylene glycol butyl ether + 116 g H<sub>2</sub>O in a 400 mL beaker.

Figure 8 shows the intensity of cavitation increases at all power levels by the addition of seven percent. Butyl Cellosolve<sup>®</sup> increases the viscosity of water from .92 cps to 1.38 cps, slightly decreases the vp, and lowers the surface tension of water. These tend to lower the cavitation energy. However, the wetting factor of this material enhances the rate of dispersion.

UCAR is a registered trademark of Union Carbide.  
Cellosolve is a registered trademark of Union Carbide.

Table 8—Dry Film Properties

Formula	Disp. Process	Scrub Resis.	Length Anti-Sag	NYPC Leveling	Color Calbizol Violet	Accept. Thalo Blue	Test Iron Oxide	Gloss Measurements	
								Sealed 48 hr	Unsealed 48 hr
A.....	conv.	1582	12	4	+	+	-	35	35
	ultra.	1600	12	4	+	+	-	63	52
B.....	conv.	1120	12	5	+	+	-	2.4	2.5
	ultra.	1150	12	4	+	+	-	3.5	3.8
C.....	conv.	271	12	4	+	+	-	2.2	2.2
	ultra.	280	12	4	+	+	-	2.2	2.2

EXPERIMENTAL

Dispersing Equipment

The ultrasonic source was a Vibra-cell ultrasonic processor, model VC 500, made by Sonics & Materials. The specifications were as follows: 500 W, 20 kHz, dual output, variable power output control, automatic timer with pulser, and 1/2 in. horn. The conventional method consisted of a Cowles Dissolver with a 1/2 hp motor as the power supply. Sample sizes for the ultrasonic process varied between 200 and 400 g. A 400 mL plastic beaker was used with 2-in. Cowles disperser blade. Slight agitation at the slowest speed (380 ppm) was used for the ultrasonic preparations. In the conventional preparations, the sample sizes were approximately one quart. The grinding was at 2300 rpm and the let down was at 720 rpm.

Watts Measurement

The electrical measurement of the power supplied to the ultrasonic and the Cowels was selected as offering the best compromise of precision and flexibility for this investigation. A voltmeter-ammeter combination was used to measure the power supplied.

Dispersion Measurement

The degree to which a pigment is dispersed is assessed by the use of a Hegman grind gage. The fineness of grind provides a direct measurement of the coarsest pigment particles present in a pigment dispersion. The time was measured in seconds. The fineness of grind, as measured by the Hegman scale, was recorded at 15 sec intervals. These data are recorded in Figures 3-9.

Formulations

Three different formulations were utilized, semi-gloss interior, flat exterior, and flat interior paints. Tables 1-3 display these formulations and their calculated physical constants. Tables 4-6 display the ultrasonic formulations. Tables 7 and 8 relate these physical properties to their respective formulations for easy comparison to dispersion data and their final physical properties. The total energy requirement to produce the desired grind is calculated. It is defined at kW-hr/gal.

RESULTS AND DISCUSSION

Effect of Acoustic Pressure Amplitude (Power)

In the series of experiments to determine how the degree of dispersion depended on the intensity, all other test parameters were kept constant at the following values: duration, t=90 sec; bath temperature, T=25°C; content=200 g H<sub>2</sub>O and 117-g TiO<sub>2</sub> in 400 mL beaker; and frequency, f=20 kHz.

Figure 3 shows how the rate of dispersion depends on intensity. As illustrated previously, the pressure in the shock wave increases with increasing R<sub>m</sub>. The maximum radius R<sub>m</sub> that the bubble attains depends on the value of

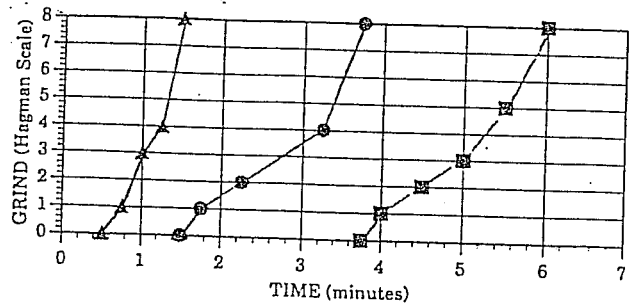


Figure 9—Effect of propylene glycol. ■—100 W, ●—170 W, and ▲—250 W

the acoustic pressure and frequency. The greater the half period, the larger the diameter to which the bubble will expand. In addition, larger amplitudes produce more cavitating bubbles which also, of course, provide more collapsing force.

In summary, the amount of cavitation and its intensity increases proportionately as the acoustic pressure amplitude (power) increases over the cavitation threshold.

The proper amount of power to use in a sonic unit cannot be made since effective power levels will differ with the acoustics of each unit design and with the type and design of transducer.

Effect of Temperature

In the series of experiments to determine how the degree of dispersion depended on the initial bath temperature, T<sub>1</sub> = 25°C and T<sub>2</sub> = 45°C, all other parameters were

Table 2—Formula B—High Speed Disperser; Flat Exterior

Material	Formula		Total Solids	
	Lb	Gal	Lb	Gal
Water	170	22.8	—	—
AMP 95	5.0	0.64	—	—
Propylene glycol	30.0	3.47	—	—
SCT 200	25.0	2.89	5.0	0.58
Water	20.0	2.40	—	—
Triton X-100	2.0	0.22	2.0	0.22
Nopco NDW	2.0	0.28	2.0	0.28
Troysan 192	4.0	0.51	4.0	0.51
<b>Mix then add:</b>				
Ti-Pure R960	250.0	7.3	250	7.3
Atomite®	205.0	9.11	205.0	9.11
<b>Grind:</b>				
Nopco NDW	2.0	0.28	2.0	0.23
E.B.-solvent	30.0	3.99	—	—
<b>Mix:</b>				
Amsco 3077	362	40.22	199.0	20.32
Water	134	16.11	—	—
	1241	110.22	669	38.55

Grind = 6.  
Grind paste = × 1.5.  
Time = 8 min.  
Watts = 6.9 AMP × 121 V.

$$\text{Energy requirement} = (6.9 \times 122) \frac{\text{W}}{\text{gal}} \times \frac{1}{1.5} \times 8 \text{ min} \times \frac{1}{60,000}$$

$$= 0.07483 \text{ (kW-hr)/gal.}$$

Atomite is a registered trademark of Cyprus Mines Corporation.

### Effect of Propylene Glycol

In the series of experiments to determine how the degree of dispersion was affected by adding seven percent propylene glycol, all parameters were kept constant at the following values:  $f = 20$  kHz; initial temperature =  $25^{\circ}\text{C}$ ; and power  $N = 100$  W, 170 W, and 250 W. System A content values were: 200 g  $\text{H}_2\text{O}$  + 116 g  $\text{TiO}_2$  in a 400 mL beaker. System B content values were: 186 g  $\text{H}_2\text{O}$  + 14 g propylene glycol + 116 g  $\text{TiO}_2$  in a 400 mL beaker.

Figure 9 shows propylene glycol does not affect the rate of dispersion at this level. However, a decrease of the rate of dispersion is expected as the percentage of propylene glycol increases, since the viscosity of propylene glycol is 46.7 times that of water.

### SUMMARY AND CONCLUSION

Ultrasonic dispersion of pigments depends on cavitation phenomena. It is defined as the formation and collapse of bubbles. The collapse of large cavitation bubbles leads to the generation of strong pressure pulses which can reach a value of  $10^6$  atm.

The degree of growth and collapse during a given acoustic cycle and the lifetime of the bubble in the acoustic field depends on many factors, such as acoustic pressure amplitude, acoustic frequency, gas content of the cavity, hydrostatic pressure, temperature (which influences the vapor pressure and the surface tension), and the material properties of the liquid (for example, surface tension, vapor pressure, and viscosity).

The work described in the present paper attempts to establish the understanding of the role of these parameters

in the preparation of paints. Hence, an effective and an efficient ultrasonic dispersion can be accomplished in a small beaker.

The criteria for choice of a liquid for maximum cavitation activity would be low viscosity, high surface tension, low vapor pressure and low frequency, and high intensity. Accordingly, water medium as cavitation agents enjoys a marked advantage over the organic solvents. This corresponds favorably with the increasing demand for waterborne coatings.

Ultrasonically made paints show at least equal or better properties, as shown in Table 8. Comparing the time to disperse the pigments to the desired grind, all formulations show the energy consumed by the ultrasonic is from 12.0 to 29% of that used by the Cowles Dissolver. This represents a savings of 71 to 88% of the energy used in dispersion.

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