

# ULTRASONIC EMULSIFICATION

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The pharmaceutical and cosmetic industries are now employing the energy of ultrasonic vibration as a more efficient means of creating: (1) *stable emulsions*; (2) *emulsions which have optimum viscosity*; (3) *emulsions with desirable particle size*, and (4) *thorough distribution of solid particles in a liquid*

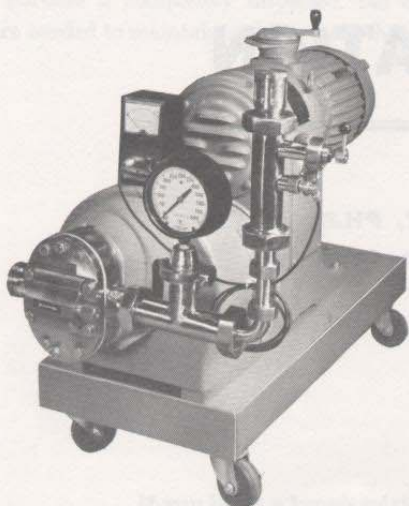


FIGURE 1

"Rapisonic" quick-strip, sanitary homogenizer takes only 10 minutes to take apart, clean and assemble those parts in contact with the liquid. Its stainless steel construction is particularly suited to pharmaceutical and cosmetic processing. It is capable of handling a wide range of emulsion systems, both oil-in-water and water-in-oil.

*medium (dispersions) . . . or any system requiring high-intensity mixing.* Results for those systems have been difficult to predict in advance with conventional equipment.

Emulsions and dispersions are often made with low-cost, propeller mixers, involving a time-consuming process and producing a low-quality product with large particle size. Other equipment used are colloid mills and piston homogenizers, which do produce a good emulsion, but with low throughput, high power requirements, high maintenance costs, and perhaps undesirable heating effects. Equipment is also bulky and has a high initial capital cost.

## Ultrasonics

Today, ultrasonics equipment, capable of being engineered to almost any capacity and for use in any

liquid processing system, is available. Such equipment is being used by more and more drug and cosmetic manufacturers as an efficient, low-cost system for producing excellent emulsions and dispersions.

Low-cost, large-volume production is achieved through a unique concept creating ultrasonic energy mechanically. Such equipment is manufactured by Sonic Engineering Corporation, Norwalk, Connecticut. The equipment is used widely to manufacture such drug and cosmetic products as antacid emulsions, antibiotic salves and ointments, and a broad variety of lotions, cold creams, and other cosmetics.

The processing technique is so unique that a descriptive word had to be coined to describe it . . . sonolation. Sonolation equipment should not be confused with transduced piezoelectric devices, operating in the ultrasonic frequency range, which tend to be limited in output and very expensive. The sonolator produces high intensity energy at lower cost, and the economic advantage rises sharply as process volumes increase.

The Sonic unit used in the drug and cosmetic industry is the "Rapisonic," a quick-strip, stainless steel system which meets the requirements of the pharmaceutical and cosmetic industries (Figure 1).

How does a sonolator operate? Liquids are pumped through a specially-shaped orifice at pressures of 150-350 psi at high linear velocity against a stainless steel blade cantilevered in the jet stream. The dimensions of the blade, together with the other dimension parameters of the system, are such that the system operates in the ultrasonic range. As the liquid jet flows over the blade, it causes the blade to vibrate very rapidly . . . to produce an ultrasonic "note." When the system reaches a steady state, a cavitation field is generated at the leading edge of the blade. Cavitation pressure fluctuations of up to 60 tons/sq. in. are generated. It is within this cavitation envelope that the high energy mixing effect is produced. (See Figure 2).

If dissimilar liquids are passed through the sonolator, a fine-particle (Fig. 3, page 823) emulsion results. If finely divided solids and liquids are passed through, a dispersion results.

Key design parameters which lead to large increases in acoustic energy are proper blade-to-orifice distance, and back pressure. The sonolator is designed to permit best selection of these important factors while the unit is running. The sonolator can thus be "tuned" for every system. This affords the processor an opportunity to maximize acoustic energy for any

particular system, and provides him with a far more fundamental processing tool than simply a mixing tool for manufacturing emulsions or dispersions.

Water-in-oil emulsions are ideal for the sonolator. Traditionally, to secure this type of emulsion, water is added slowly to the oil phase until the desired end product is achieved. Water must be added very carefully, and even with every precaution the emulsion may invert to an oil-in-water emulsion. The entire mixture will be wasted.

With a sonolator, this danger is eliminated. The procedure is done semi-automatically, blending the separate phases by recirculating the oil, and adding water until it is distributed uniformly throughout the entire batch . . . without fear of inversion.

Often the rate determining step of a particular process involves an energy interchange through a liquid-solid interface. This leads to other applications such as liquid-solid extraction, where the objective is to expose the solids to the liquids efficiently in order to effect the extraction. The rate of extraction is dependent on the rate at which the liquid-solid interface energy exchange takes place. Sonolation can increase the exchange rate markedly.

For example, a manufacturer of vanilla extract uses of Sonic "Dispersionic" unit to speed the extraction of vanilla from the bean. Extraction time has been cut from 48 hours to one hour.

Gums and film-formers, such as Veegum, acacia, and tragacanth, have a tendency to ball up when dispersed in water. Development of maximum viscosity with conventional mixers may require many hours.

The "Rapisonic" is ideal for dispersing these thickening agents or gums. Intense acoustic vibrations quickly penetrate the exterior of the dry gum particles, wetting them and mixing them completely. The sonolator permits rapid development of viscosity and

eliminates hours of mixing and results in a very stable emulsion (Figure 5).

### Advantages of Sonolation

The cosmetics industry was one of the first to take advantage of ultrasonic emulsification and dispersing. Here are the reasons why sonolation has become so popular in the industry:

1—*Simple, low-cost design*—Initial equipment costs are a fraction (often one-fourth or one-fifth) of the outlay required for conventional equipment with similar output. Installation of a sonolation system often merely means the replacement of an existing unit without change of production methods. A premix can be transferred by the sonolator to storage or recirculated into the same container.

2—*Low operating costs*—

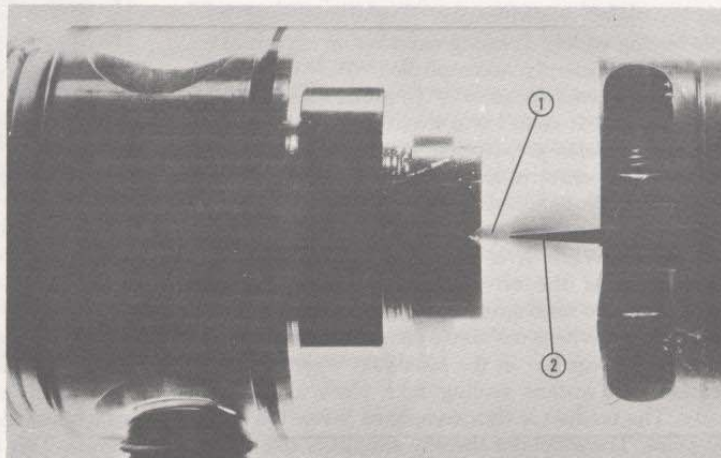
a. *High efficiency*—Because all the energy is developed and used right in the liquids, high operating efficiency results. High production rates are achieved with a fraction of the power needed for conventional mixing equipment. Liquid output-power input ratio of Sonic equipment is the highest of any high intensity mixing equipment used in emulsification, dispersion, super-mixing or reaction.

For example, Chesebrough-Pond's, Inc., Clinton, Connecticut, recently replaced two piston-type homogenizers with a "Rapisonic" unit of Sonic Engineering Corporation for producing "Dry Skin Cream" (Figure 4, page 824), with the following results:

- a. Achieved production rates of 480 gph, compared with 150 gph with the piston-type units.
- b. Saved 20 per cent in power requirements.
- c. Realized a 60 per cent saving in space.
- d. Reduced maintenance.
- e. Eliminated one pump needed from storage to filler hopper.

FIGURE 2

Laboratory strobe photo shows Sonic sonolator in action. This processing system is one of the highest intensity mixing devices available, mechanically generating ultrasonic acoustic energy to produce emulsions and dispersions . . . and to achieve faster reactions in many heterogeneous phase systems. Liquid to be processed is pumped through a special orifice forming a flat, high-pressure jet stream (1). This jet impinges on the edge of a flat blade (2), causing it to vibrate at ultrasonic frequencies. Cavitation takes place continuously, producing violent local pressure changes. These are put to work immediately in the liquid, with virtually no loss of energy.



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Piston-type homogenizers required an 8-hour day to achieve six 1000 lb. batches. The "Rapisonic" at Chesebrough-Pond's makes a 5000 lb. batch in three hours. The only maintenance required involves routine six-month blade replacement and motor lubrication. "The 'Rapisonic' has been operating well for the past year with only minimum maintenance," according to the company, "producing excellent emulsions at low cost."

b. *Low maintenance*—The sonolator has but two moving parts . . . the vibrating blade and the pump. The blade operates indefinitely without replacement, and when needed, replacement cost is negligible. Pump maintenance follows standard pump procedures—with standard care. This is in direct contrast with piston-type homogenizers, which require frequent replacement of expensive parts, and take hours to tear down for maintenance or repair.

c. *Easy cleaning*—The two moving parts of a sonolator system consist of a precision pump and the vibrating blade, both of stainless steel and easily cleaned. The sanitary model ("Rapisonic") can be taken apart, cleaned and reassembled in 10 minutes.

3—*Versatility*—Along with its highly efficient production of emulsions and dispersions, the sonolator can be used for inline mixing and pumping duties. Residual pressure downstream makes the system available for pumping to the processing station . . . spray dryer, heat exchanger, or filling station. Because of its portability, it can be moved from one area to another with ease, making it possible to process many different formulations at different production locations within the plant.

4—*No pre-mix necessary*—Producing emulsions without pre-mix saves time and labor and is an exclusive feature of sonolator, permitting continuous and controlled emulsification. Emulsification of liquids having widely differing densities or viscosities is easily and efficiently accomplished.

5—*Faster emulsification*—"To make a good emulsion stir until it cools" is a traditional technique in cosmetic emulsion preparation. It is usually necessary to lower temperatures slowly, with continuous mixing to maintain stability.

With the sonolator, processing temperatures may be reduced. While it is necessary to heat the solid components of the oil phase above their melting points, use of a semi-automatic system makes it often unnecessary to heat the water phase. The two phases are brought together in the sonolator, where emulsification and instant cooling take place simultaneously. The product is thus completed below the critical point. The sonolator thereby simplifies the entire process, and can eliminate some heating and cooling steps, usually associated with batch premix methods.

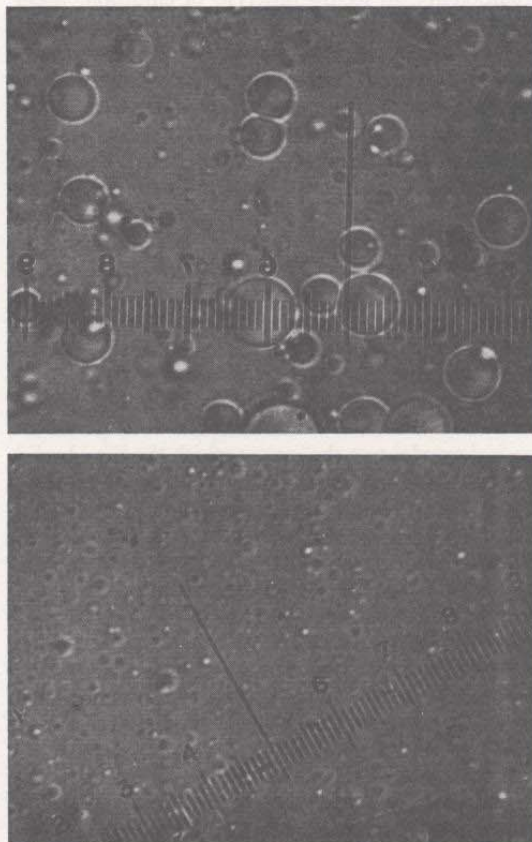


FIGURE 3

Samples of flavor emulsion before (above) and after sonolator. Magnification is 1000 times. Note fine particle size resulting from one pass through the sonolator. Each small division on the scale is one micron.

High intensity sonolator permits process simplification. Often time-consuming steps can be reduced or eliminated because of mixing efficiency.

For example, a manufacturer of liquid make-up was having difficulties in achieving a thoroughly dispersed pigmented emulsion. Using a propeller mixer to achieve a pre-mix, he then treated the entire batch extensively, through a 3-roll mill. Red and white streaks of iron oxide and titanium dioxide appeared when the make-up was spread as a film. The process was time-consuming, as well.

With sonolator, this company is now getting a thoroughly dispersed pigmented emulsion. A 100-gallon batch is made by adding the water phase and surfactants, then pigments are added directly as a pre-mix. This low-viscosity slurry is then recirculated through a "Dispersonic" unit, which disperses the pigment particles completely. Oil is added, the mix-

ture is again circulated, and an emulsion plus a dispersion result.

6—*Materials economy*—The very high energies produced by cavitation afford a means of creating smaller particle size (Figure 3), and in a great many cases allow a reduction in the amount of emulsifying agent.

7—*Crystallization*—In normal crystallization techniques (in penicillin production, for example), a solution containing materials to be crystallized are super-saturated either by cooling or by evaporation and then seeded. Because seeding may be initiated non-uniformly, resultant crystal growth proceeds at different rates at different nuclei sites, and resulting crystals may show a very broad and uneven crystal size distribution.

With the sonolator the cavitation bubbles generated at the leading edge of the vibrating blade act as sites for crystallization and eliminate the need for seeding in the usual manner. Because of their great number, very small bubbles form very uniform and usually small crystals. In some instances, there is evidence of both a more orderly growth of crystal

FIGURE 4

"Rapisonic" homogenizer at Chesebrough-Pond's (lower right) and storage tanks and valves are key elements in processing of "Dry Skin Cream." Operator has left hand on sonolator, inside which are located the vibrating blade and jet-stream orifice of the "Rapisonic." This unit replaced two piston-type homogenizers, and at the same time tripled production speeds.

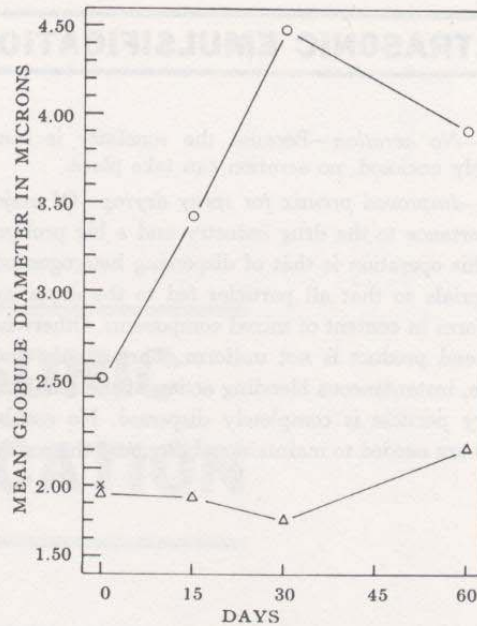
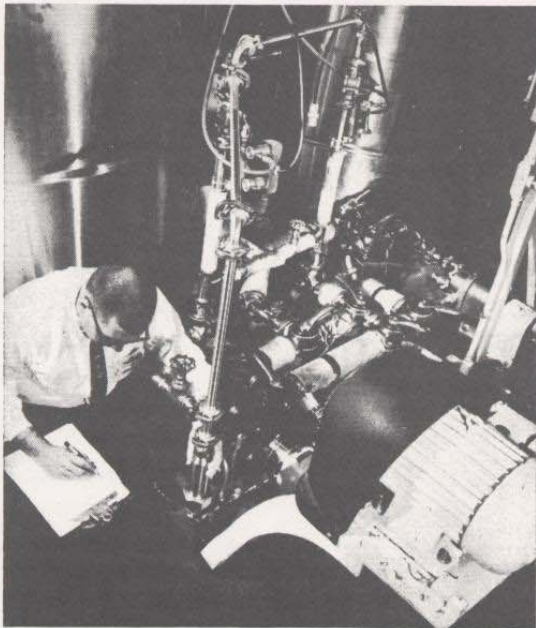


FIGURE 5

Mean globule diameters of stored mineral oil-acacia emulsions. Emulsifying apparatus: O, colloid mill; Δ, ultrasonic generator (sonolator).

faces, and development of a purer crystal.

In their natural state, vitamins are oily. In many cases, in such products as animal feed, food supplements, etc., it is desirable to produce them in the dry state for formulating. Vitamin particles must be of a certain size for proper absorption into the bloodstream. If too large, they will not be absorbed too readily by the system, and if too small, more surface area is exposed, and the vitamin is rendered more susceptible to degeneration by oxidation.

Under the previous system, a pre-mix was made, using a tank with a propeller mixer, then the pre-mix was run through a colloid mill. Considerable heat was generated, and air was incorporated into the mixture. The resulting product was unstable, and of the wrong particle size. It took the company one hour to make 100 gallons of product.

With a "Rapisonic" sonolator, proper uniformity is achieved with reproducible results. Sonolated material has been proven to have greater shelf life and greater bioability. Production has been increased by 300 per cent; it now takes but half an hour to make 200 gallons.

The procedure is this: A water emulsion is first made of the vitamin, synthetic starch is added, and then the mixture is dried on rolls. The resultant powder is Vitamin A, encapsulated in the dry starch, with the water evaporated.

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8—*No aeration*—Because the sonolator is completely enclosed, no aeration can take place.

9—*Improved premix for spray drying*—Of major importance to the drug industry and a big problem in this operation is that of dispersing heterogeneous materials so that all particles fed to the dryer are uniform in content of mixed components. Otherwise, the end product is not uniform. Through the intimate, instantaneous blending action of the sonolator, every particle is completely dispersed. No emulsifiers are needed to maintain stability, and the sonola-

tor pump can be used to feed directly to the spray dryer.

Finally, many areas of application for sonolation have not yet been touched on. Important work is proceeding on speeding up chemical reactions and polymerization. Synthesis of biologically delicate materials is feasible. New process lines to add flexibility and automation to the drug and cosmetic manufacturer's flow sheet are being developed to utilize this unique processing tool, often with elimination of batch processing. ■

**If you have a tough mixing problem that needs a fresh approach,  
call or write.**



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