



Homogenizer Handbook

Processing of Emulsions and Dispersions



SPX®

From its very genesis APV has maintained an impeccable reputation for excellence worldwide. That standard is perpetuated in the field of homogenization and high pressure pumps. It's a tradition that dates back to our invention of the homogenizer in the early 1900's.

That desire to excel in saving our customers is demonstrated today through innovative design, precision, engineering, quality manufacturing and attention to detail within the APV Homogenization Division.

This division, created by the unifying of APV Gaulin and APV Rannie introduced a new era in the industry. With more than 155 years of combined experience, APV provides our customers with the greatest resource for homogenization technology ever assembled. While maintaining the distinguishing characteristics and unique designs of the GAULIN and RANNIE products, APV is now able to offer a much greater range of equipment and service from a single source.

The APV Homogenizer group is ready to meet the challenges facing us in this exciting, technologically advanced world, and we look forward to serving you!

World Leader in Homogenization Technology

Introduction

The APV Homogenizer Group designs and manufactures high-pressure homogenizers (operating pressures up to 29,000 psi/2000 bar) for the processing of emulsions and dispersions. Emulsions with viscosities up to several thousand centipoises, cP, (for the premix) may be processed, and a particle size as small as 0.1 micrometers is possible by selecting the correct unit. The same viscosity and particle size limitations apply in the case of dispersions. Naturally, there will be some overlapping as to viscosity and particle size governing the choice of equipment; but, in general, the operating parameters are sufficiently well defined that an intelligent choice for evaluations may be made.

Emulsions and dispersions encompass a significant number of products that would seem to be, on cursory examination, totally unrelated to each other in form, substance and method of production. However, a more detailed investigation would reveal that many basic elements are common to all emulsions and dispersions. To discuss the topics of homogenization and emulsification in a reasonable space, it is necessary to relate these processes to the basic elements of emulsions and dispersions. Therefore, although this handbook will examine general conditions of homogenization, it is reasonable to expect that most emulsions and dispersions will respond in similar ways to these processing techniques.

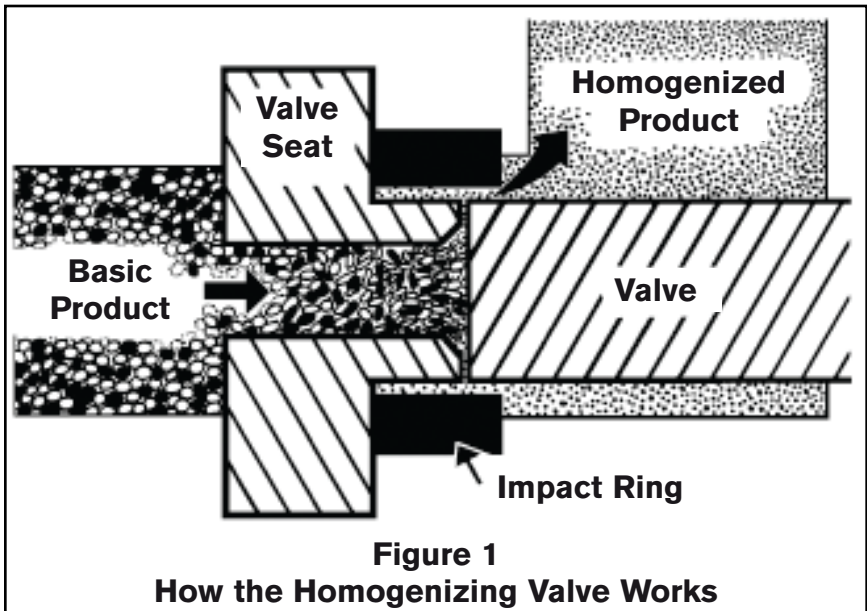
Homogenization

At the World's Fair in Paris in 1900, Auguste Gaulin exhibited his invention... a process for "treating" milk. According to the literature and publications of that time, the word "homogenized" was first used to describe milk treated by the Gaulin machine. Therefore, the terms "homogenization" and "homogenizer" historically relate to the process and equipment developed by Gaulin. The homogenizer basically consists of a positive-displacement pump to which is attached a homogenizing valve assembly. The pump forces fluids through the homogenizing valve under pressure. (This is described in more detail later in the handbook.) The term "homogenization" refers to the process or action that occurs within the homogenizing valve assembly. Today, the terms "homogenization" and "homogenizer" are quite often incorrectly applied to devices that subject a liquid mixture to conditions different from that in the Gaulin and Rannie homogenizers. In this handbook, "homogenization" and "homogenizer" refer to the action and equipment related to the classically recognized homogenizer.

Homogenizers

To understand how the homogenizer works and what it does, it is first necessary to trace the path of the liquid mixture through the homogenizing valve. Figure 1 shows a plug-type homogenizing valve and standard valve seat. The unhomogenized product enters the valve seat from the pump cylinder at a relatively low velocity but at a high pressure. For example, the velocity may be about 10 to 20 feet per second (3.05 to 6.10 m per second). The pressure for this example will be at 3000 psi (206.8 bar). This pressure is generated by the positive-displacement pump and by the restriction to flow caused by the valve being forced against the seat by an actuating force. The positive-displacement pump provides a relatively constant rate of flow and, therefore, will generate the required pressures as the flow area between the valve and seat is increased or decreased.

The liquid flows between the valve and seat at high velocity. In this case at 3000 psi the corresponding velocity would be about 500 feet per second (152.4 m per second). As the velocity increases, the pressure decreases producing an instantaneous pressure drop. The liquid then impinges on the wear ring (impact ring) and is finally discharged as homogenized product. The time it takes for the liquid to travel across the face of the valve seat and to undergo homogenization is less than 50 microseconds (5 one hundred thousandth of a second). Therefore, it can be seen that a large amount of energy is dissipated in a very short time, producing a large energy density in the liquid.



The theories of homogenization that have been presented over the years have attempted to relate this high-energy transition in the valve to the results observed in the product. In the past, it was difficult to prove or disprove many of these theories, but it appears that turbulence is the primary mechanism.

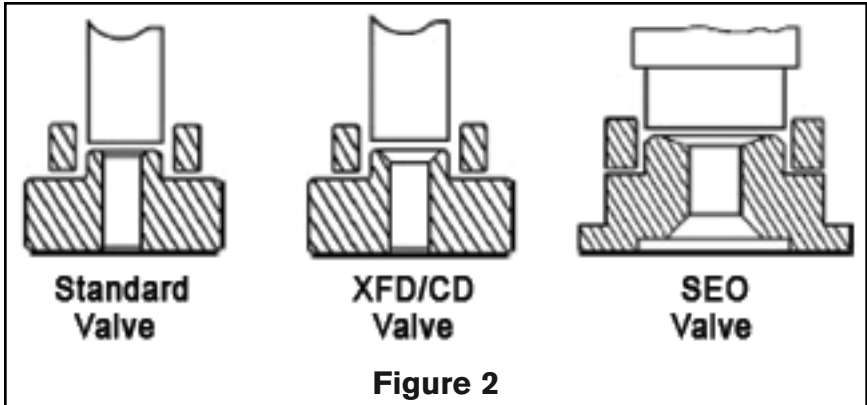
First, something should be said about shear in the valve, because many people assume that shear is the main mechanism of homogenization. When using the term “shear”, one is describing the elongation and subsequent breakup of a dispersed phase droplet because of different fluid velocities surrounding the droplet. The classical theories of shear effects, as related to the flow profile in the homogenizing valve, are difficult to justify. Two arguments against shear are (1) the viscosity of many fats and oils is greater than the maximum value allowed for shearing of the droplets, and (2) the flow profile in the valve is such that the bulk of the liquid does not experience large velocity gradients due to the boundary layer effects.

In the cavitation theory, the liquid encounters intense cavitation because of the large pressure drop through the valve. When the pressure drop is large enough, the vapor pressure of the liquid exceeds the ambient pressure causing formation of vapor bubbles (cavities in the liquid). When the cavitation bubbles implode (collapse of the cavities), shock waves are generated in the liquid. These shock waves break apart the dispersed droplets.

The second homogenization theory, relating to turbulence, suggests that the energy dissipating in the liquid generates intense turbulent eddies. These eddies would be of the same size as the average droplet diameter. The intense energy of the turbulence and localized pressure differences would tear apart the droplets, reducing their average size. Flow visualization experiments by this author using a modeled quartz homogenizing valve revealed that, for emulsions, droplet size reduction is produced by turbulent flow. Backpressure or second-stage pressure suppresses cavitation but maintains turbulent flow. Droplets passing through the turbulent jet at the discharge from the gap are immediately disrupted by intense turbulent flow.

Types of Homogenizing Valves

Different types of APV homogenizing valves are available for various applications. Figure 2 shows some of the configurations of these valves. Through experimentation and experience, it has been found that certain valve geometries are more efficient at homogenization than others, depending on the product or process involved.



The standard flat valve (SV) is used in a variety of food, dairy and chemical applications. This valve is available in different sizes; and the conditions of operation, such as homogenizing pressure and flow rate, would determine which size would be appropriate. Standard valve material is a nonferrous alloy; however, tungsten carbide or ceramic would be provided for highly abrasive applications.

By putting a large inner chamfer on the flat valve, a short-land valve seat is produced. The “land” is the surface on the seat that the fluid passes over as it travels through the gap between the valve and the seat. The XFD style and SEO style encompass the short-land seat. The short-land valve is beneficial when processing milk or similar emulsions with the lowest homogenizing pressure possible to achieve normal shelf stability. This valve usually requires a second-stage valve or a backpressure device. The short-land valve design is incorporated into the patented APV Micro-Gap® homogenizing valve, which is more fully described in Technical Bulletin #65.

The cell disruption (CD) valve, a version of the XFD, represents a unique variation of the short-land design in a single-stage configuration. This valve is most efficient for processes that require the disruption of single-cell microorganisms and the subsequent release of proteins and enzymes. This application is discussed in detail in the booklet, “Cell Disruption by Homogenization”.

The SEO valve is used for high-pressure applications (over 10,000 psi/690 bar) and for critical emulsions, where small and uniform particle size is needed, such as liposomes and intravenous emulsions.

Variations in valve configuration, such as piloted versus plug, do not significantly affect the action or efficiency of the valve in most cases but are only related to mechanical aspects of the valve such as alignment and incorporation of hardened surfaces.

Colloid Mill

The following information will be useful for current colloid mill users. The APV colloid mill is different from the homogenizer in its action on the processed fluid. The operation of the colloid mill is similar to the mechanism of a basic mill configuration; the work on the product takes place between a stationary part (stator) and a rotating cone (rotor). The premix is fed into the area between the rotor and stator by centrifugal force. With its high peripheral speed, the rotor generates high shear fields within the fluid in the working area. Because of the unique features of the APV colloid mill, the distance between the rotor and stator can be adjusted to optimize the energy imparted to the product.

By decreasing the distance between the rotor and stator, shear on the product is increased; however, there are limitations in this procedure. Decreasing the gap will also substantially decrease the flow rate and increase the temperature rise in the product. Figure 3 shows a new design for a patented APV colloid mill (US Patent 6,305,626).

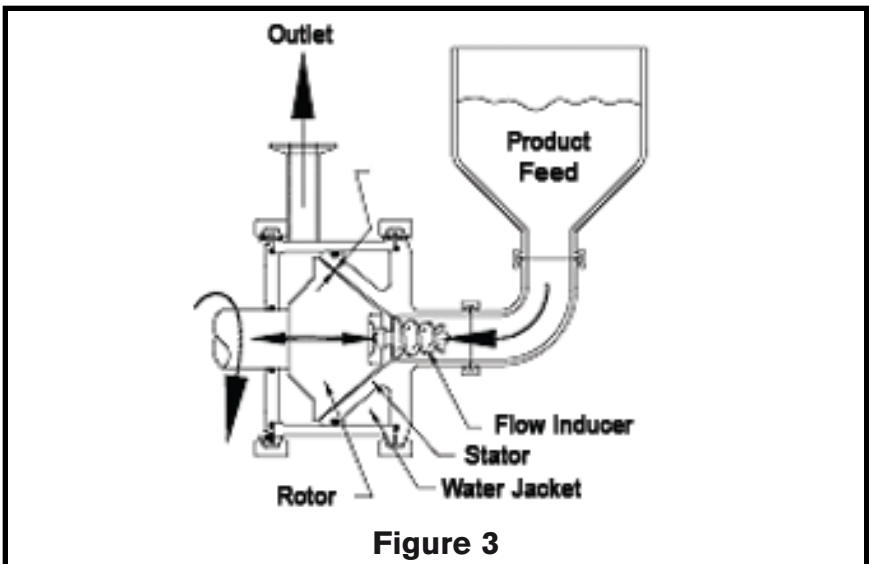


Figure 3

Emulsions

An emulsion may be defined as a heterogeneous system consisting of two immiscible liquid phases in which one is intimately distributed in the other as minute droplets whose diameters exceed 0.1 micrometer. In most cases one of the liquids is water. A third component, known as a surfactant (emulsifying agent or stabilizer), must be present in amounts adequate to prevent coalescence of the dispersed phase.

When oil is dispersed as extremely fine droplets in a continuous phase of water, the emulsion would be oil-in-water (O/W). Emulsions of this type are water-dilutable. When the oil is the continuous phase with the water distributed in the oil as small droplets, the emulsion is water-in-oil (W/O). This type of emulsion is not dilutable with water but will dilute with oil.

The type of emulsion that forms is greatly dependent upon the nature of the surfactant used in the formulation. The surfactant or emulsifier selection is a science in itself and will not be detailed here. However, it can be briefly stated that the two main functions of the surfactant are (1) reducing the interfacial tension of the oil and water phases, and (2) preventing agglomeration, coalescence and breaking of the dispersed droplets after they are formed.

Obviously, the amounts of the various ingredients making up an emulsion can vary significantly, due to the large number of diverse emulsion products. However, it has been determined that the amount of surfactant required for an emulsion in many cases is dependent on the energy level (kW/amount of product) used to generate the emulsion. For example, homogenizing at 4000 psi (275.8 bar) can generate a certain type of emulsion with 0.25% emulsifier. This would be equivalent in average droplet diameter to an emulsion generated by a high shear mixer using 2.25% emulsifier. Therefore, the high-energy input of the homogenizer allows a reduction in amount of emulsifier and, thereby, a substantial savings in emulsifier costs. This phenomenon is more fully explained in Technical Bulletin #61.

Emulsifiers

The selection of a suitable emulsifying agent is a complex process. An emulsifier is categorized in relation to the balance that exists between the oil-soluble (lipophilic) portion and the water-soluble (hydrophilic) portion of the molecule. Combinations of emulsifiers are sometimes used to achieve the desired balance. For references concerning techniques for selection of emulsifiers, as well as composition, I recommend contacting companies that sell emulsifiers.

If the emulsifier is 25% oil soluble and 75% water soluble, then an emulsion prepared with this particular agent will usually be an oil-in-water type; that is, the oil is dispersed as extremely fine droplets in a continuous phase of water. If the emulsifier is 75% oil soluble and 25% water soluble, then the reverse will be true. It will tend to form emulsions of water-in-oil, where the oil is the continuous phase with the water distributed in the oil as small droplets. Of course, the amount of solubility in the oil and water phases, as mentioned here, is only approximate but it is used to illustrate Bancroft's Rule. Simply stated, this rule is that the phase in which the emulsifying agent is more soluble will be the continuous one.

There are four general types of emulsifiers: non-ionic, which do not impart a charge to the dispersed phase; cationic, which impart a positive charge to the dispersed phase; and lastly, amphoteric, which will impart either a positive or negative charge, depending upon the pH of the system. At the present time there are several thousand emulsifying agents commercially available. With this large number of possible agents, formulating a new emulsion and determining the best emulsifying agent from cost and efficiency standpoints could require months of work. Fortunately, all of the agents would not have to be tried, since one would select different hydrophilic/lipophilic blends and then work with the agents in a particular chemical grouping, consistent with the blend characteristics.

For a complete listing of emulsifications and their manufacturers I suggest "Detergents and Emulsifiers", printed by McCutcheon's Publications, 175 Rock Road, Glen Rock, New Jersey 07452, www.gomc.com/mccutcheons. New editions are printed yearly. For those interested in the theory of emulsions, see the listings in the bibliography.

After having selected the optimum emulsifying agent for a particular system and adding it to the emulsion formulation, one may ask, "How does it work?" The emulsifier's oil-soluble end dissolves in the surface of the oil with the water-soluble portion of the molecule sticking out into the aqueous phase, so that the oil droplet would resemble a pincushion. We now have a system with a large number of small oil droplets bounded by an emulsifier, and all of these droplets have a charged layer surrounding them (electrical double layer).

Even with nonionic emulsifiers and water-in-oil emulsions, it is believed that some degree of charge potential may exist around the droplet, possibly generated by a frictional mechanism. With certain stabilizers such as gums, a protective, uncharged coating forms around the droplet. This emulsifier layer, therefore, either because of its charge effect or purely physical barrier, causes repulsion between droplets when they approach within a certain distance of one another. This layer helps stabilize the emulsion, because it minimizes flocculation and coalescence of the droplets. Stokes' Law predicts how the creaming rate is affected by the physical characteristics of the emulsion. The rate increases as particle size increases (it is proportional to the square of the radius); increases when the two immiscible phases have a large difference in density and decreases when the viscosity of the outer phase is large. Therefore, the emulsifier minimizes creaming, because flocculated droplets represent bigger particles that separate faster. Also, by preventing coalescence it minimizes breaking of the emulsion, which results in the formation of two separate phases (demulsification).

If the oil concentration (O/W) is increased to a level where there is so much oil surface that the particles are physically touching, then a point will be reached where the viscosity of the emulsion rises very rapidly. A further increase in oil concentration will result in a breaking or an inversion of the emulsion to a water-in-oil type. Due to the large surface that can be developed with the homogenizer, 65% oil is generally the upper limit; although in some cases where extremely small particle sizes are present, 50% may be the upper limit. With the colloid mill, where the average particle size is larger and the surface area produced is not as great as for the homogenizer, it is possible to make emulsions of 80 to 85% oil phase.

Why Use Mechanical Energy to Process Emulsions?

Dispersions require the application of mechanical energy for preparation. Emulsions, however, can be prepared spontaneously; that is if sufficient surfactants and cosurfactants are mixed with an oil, the oil can be poured into water and a stable emulsion prepared (microemulsion). This type of system, however, requires a unique combination of ingredients and a large amount of emulsifier. Since one can "trade off" mechanical energy for the chemical energy, of an emulsifier, the mechanical energy at \$.03 to \$.08 per kilowatt hour is much cheaper than an emulsifier at \$5 to \$50 per pound; then, here is the case for high energy equipment. If one were to use a standard mixer, the emulsifier level could be reduced to approximately 5 to 10% as opposed to the 25 to 50% required for a spontaneous emulsion.

If we move to still higher energy levels; for example the colloid mill, the emulsifier level could probably be reduced to 2 to 5%; with the still higher energy level of the homogenizer, it can be reduced to as low as 0.2%.

These figures are approximate, since they depend on the total surface area to be developed and the efficiency of the particular emulsifier. They do represent, however, typical reductions that are possible by substituting mechanical energy for chemical energy.

Dispersions

For this booklet a dispersion (or suspension) is defined as solid primary particles, agglomerates or aggregates distributed uniformly throughout a continuous medium. These particles or group of particles can range in size from 0.001 micrometers on up to greater than 1 micrometer. A dispersion is colloidal if at least one dimension is between 0.001 micrometer and 1 micrometer. ¹ The term "aggregate" refers to a group of particles that are more tightly bound to each other than are particles in an agglomerate. The particles in an aggregate may be joined at their faces or attached to each other by salt bridges. In an agglomerate the particles are less strongly attached at their edges and corners. An agglomerate or "...floc may be considered as a loose, irregular, three-dimensional cluster of particles in contact in which the original particles can still be recognized".²

When making these definitions, most authors are referring to dispersions of powders or pigments. However, because of the diversity of applications involving the homogenizer and colloid mill, it is sometimes difficult to describe all homogenized dispersions by a simple definition. Nevertheless, many of the dispersions encountered can be related to these definitions.

A few examples of the dispersions processed in the homogenizer or colloid mill reveal the variety of these applications:

Antacid	Talc	Ink
Cellulose	Mica	Carbon Black
Antiperspirant	Teflon	Calcium Stearate
Clay (Bentonite, Kaolin)	Dyes	Titanium Dioxide
Ketchup	Fumed Silica	Barytes (Barium Sulfate)

These applications involve solids of one type or another dispersed into a liquid; but, obviously, some are very different from the standard pigment-type dispersion. The basic steps involved in preparing a dispersion can be given as follows:

1. Wetting (involves the displacement of air from the particles by the continuous phase);
2. Physical separation (separation of particles by some mechanical means);
3. Stabilization (preventing the tendency for re-agglomeration).³

The wetting step simply involves adding a dry powder or pigment to a liquid and mixing in the solid phase, generating a release of air from the bulk packing of the solid. Sometimes, chemical wetting agents are needed to facilitate this step.

The second step of the process involves the use of mechanical equipment. The type of equipment to be used depends on the result desired and on the physical nature of the dispersion. The types of machines used for this step in the process include mills (ball, pebble, sand, roll, high-speed impeller or colloid) and the homogenizer.

The last step involves use of the appropriate chemical surface-active agents to retard strong re-adhesion of the particles to each other but not necessarily to avoid all flocculation. In some cases it is desirable to have some very weak flocculation occur in a pigment dispersion, so that, when the pigment settles, hard packing does not occur but low energy mixing/agitation will re-disperse the solids. The evaluation and selection of chemical agents goes beyond the scope of this handbook but are thoroughly covered in many reference sources.⁴

The process of dispersing is sometimes called “milling” or “grinding”, especially in the paint and ink industries; but, in actuality, particle grinding does not occur.⁵ The dispersion process most often involves the reduction of agglomerates to their primary particle size. “It is generally accepted that primary particles of pigments are not significantly reduced during conventional dispersion processes.”⁶ If the desire is to grind particles, then the ball mill would be used: “...comminution of pigment particles during the dispersion process is unlikely to be achieved by any machine other than the ball mill”.⁷

The type of equipment used would depend on the characteristics of the product and process. For very high viscosities (over 5000 cP) the ball, roll or impeller mill would be used. The sand mill could be used in a continuous, low viscosity system. The APV colloid mill could be used in a continuous system, a closed system, an aqueous or a non-aqueous system at Newtonian viscosities below about 5000 cP. The homogenizer could be used under all these same conditions as the colloid mill; but it is most efficient at lower viscosities (less than 2000 cP), and it can generate a smaller primary particle size than the colloid mill, because of its higher energy input.

The classic Gaulin or Rannie homogenizer, consisting of a high-pressure pump and homogenizing valve assembly, disperses particles through turbulence and possibly impact generated in the homogenizing valve. This action is very different from the effects produced by the previously mentioned equipment. However, the dispersion process in the homogenizer still involves the reduction of agglomerates or aggregates to the primary particle size. "Actual breakdown of small, compact crystals (by most dispersing equipment) is not likely. One exception is a powder in the form of needle-shaped crystals."⁸

Considering the fluid action of the homogenizer to be turbulence, the break-up of these agglomerates can be described by the following statement. "The primary mechanism leading to disruption of the extended flocs by turbulence is pressure difference on opposite sides of the floc which cause bulgy deformation and eventual rupture. The pressure differences are due to the random velocity fluctuations of turbulent flow."⁹

The homogenizer can produce a very fine dispersion of particles, in many cases to the primary particle size. All of the dispersion material passes through the homogenizing valve, subjecting the total product to the high energy density in the valve. This means that homogenization would be more energy efficient than a device such as a ball mill, which requires long processing time and has an efficiency of less than one percent. Most of the energy is lost in the friction of the ball charge and is converted into heat.¹⁰

The homogenizer also makes unique changes to many products. It defibrillates the fibers of cellulose to increase its water-binding tendency. It changes the size of the tomato fibers in ketchup to produce a thicker product. It can cause delamination of clay-like products, separating the plates in the mineral, presenting more surface area to the liquid phase.

It disperses fumed silica, so that a better gel matrix can occur. Pigments, such as titanium dioxide and carbon black, are dispersed uniformly by the break-up of agglomerates.

Through experimentation and experience, certain conditions of homogenization that affect dispersions can be described. Some of these involve equipment and some involve the process. In many cases, high pressure is beneficial in producing a good dispersion. However, sometimes increasing pressure beyond a certain value does not produce a progressively better product. This is because there exists a threshold energy to disrupt the agglomerates. Once this energy is reached, the primary particle size is obtained, and no change in particle size is observed, despite increasing the pressure. A titanium dioxide dispersion would be an example of this effect.

Multiple passing may be necessary for some dispersions, if no threshold energy is reached or if unique physical changes are being made in the product such as occurs with cellulose.

Experimentation suggests that as the Newtonian viscosity of the continuous phase increases, the efficiency of homogenization decreases. For a carbon black dispersed in a high viscosity resin, this may mean that high pressure and more than one pass are required for a good dispersion.

The use of an effective surfactant can make significant changes in the quality of a dispersion undergoing homogenization. The surfactants can do this by reducing the energy required to disrupt the agglomerates or by stabilizing the formed dispersion. For example, a small amount of sodium hexametaphosphate will produce a finer dispersion of calcium carbonate than will a large amount of an ineffective emulsifier. An effective surfactant can also dramatically reduce the viscosity of a dispersion by preventing interparticle adhesion, either through charge or steric repulsion.¹¹

Results from limited experimentation suggest that the geometry of the homogenizing valve and seat can affect the dispersion process. A valve configuration that improves the efficiency of emulsification will also benefit dispersion process. Therefore, because Gaulin and Rannie use the optimum designs and materials for their homogenizing valves, efficient emulsification and dispersion will occur.

Incorporated into the design characteristics of the valve is the material of construction.

Because solids in liquids are significantly more abrasive than emulsions (liquid-in-liquid), the use of wear-resistant material on the surfaces of

the homogenizing valve and seat and on the pump valve seats is usually required. The degree of abrasiveness of the dispersion is very much dependent on the size, shape and hardness of the particles. A material that is hard (for example, on the Mohs Scale) may not be as abrasive with a particle size below one micrometer as it would be if the particles were five micrometers. Also, cubic crystals may be more abrasive than flat plates or thin needles. Therefore, it can be difficult to predict how abrasive a material will be without actual test data.

There are many different methods available for analysis of particle size or quality of dispersion. Each of these techniques will reveal an aspect of the dispersion that another may not. Therefore, it may be useful to combine more than one method to obtain a total picture of quality. These techniques will not be covered in any detail here, but they are described more fully in other reference sources or suppliers' literature. These methods include: grind gauge; microscopy; sedimentation techniques, either gravitational or centrifugal; laser diffraction; optical turbidity; photon correlation spectroscopy; hydrodynamic chromatography and, of course, simple shelf-life settling.

Energy Levels Developed

The following example demonstrates the intense energy dissipated through the homogenizing valve. At a pressure of 3000 psi (206.8 bar), a fluid moves through the valve at an average velocity of 500 feet per second (152.4 m per second) with a residence time of 50 microseconds. This results in a dissipated energy density of about 1000 kW-per-centimeter-cubed. This demonstrates how the homogenizer concentrates high energy into a small fluid volume.

The colloid mill has an energy level equivalent to the homogenizer at 500 to 1000 psi (34.5 to 68.9 bar) and is capable of preparing an emulsion having an average particle size in the one to five-micrometer range or a dispersion of comparable quality. With some products the colloid mill is adequate for the emulsion quality required. There are always exceptions to these generalities, which is the reason for the availability of our Customer Service Laboratory.

Parameters for Selection of Equipment

In general, the same parameters apply for both emulsions and dispersions, insofar as the selection of equipment is concerned. The principle parameter would be the viscosity of the material as processed, categorized either as Newtonian (real) or non-Newtonian (apparent).

Newtonian viscosity remains constant with increasing shear rate. A non-Newtonian fluid (thixotropic and pseudo-plastic) in contrast is, by way of example, a latex paint with a high apparent viscosity when the system is subjected to a low shear rate; but, when under a high shear rate, it has a low apparent viscosity. In the case of non-Newtonian fluids, the actual viscosity under a high rate of shear is the one that should be considered. A final type of non-Newtonian viscosity that has to be considered is the category dilatant/rheoplexic, where the material is fluid at rest but rapidly generates high viscosity when shear is applied. Normally, dilatant/rheoplexic systems cannot be handled with high-energy equipment; one exception being crude clay dispersions. These dispersions have been prepared with the homogenizer.

For further information on the rheology of emulsions, we would suggest Sherman's "Emulsion Science" (see bibliography) or an equivalent book covering this topic.

For guidance, we suggest the following Newtonian viscosity ranges in centipoise for optimizing efficiency (viscosity of the internal phase or premix).

Homogenizer	1 to 1000
Colloid Mill	1 to 5000

The homogenizer shows a drop in efficiency between 200 cP and 1000 cP; but, normally in this range, either the homogenizer or colloid mill may be used. The homogenizer becomes less efficient when the dispersed-phase viscosity is greater than 500 cP.

With the colloid mill there is no decrease in efficiency with an increase in viscosity, but the power requirements can become so high as to make it impractical to use the mill for viscosities above 5000 cP. If the rotational speed of the mill is reduced, considerably higher viscosities may be handled.

Other parameters that must be considered in determining the selection of equipment would be factors such as temperature rise and the physical properties of the ingredients. In systems containing volatile solvents, where the material must be maintained under pressure, the homogenizer would be more satisfactory than the colloid mill. If the product must be pumped through a heat exchanger after processing, the homogenizer would eliminate the necessity for an additional pump, which is often required with the colloid mill.

Normally, the temperature rise in the homogenizer is considerably less than in the colloid mill.

The temperature rise (°F) through the homogenizer can be estimated by the following equation:

$$\Delta T = \Delta P / 40.42 C_v d$$

where ΔP is in psi, C_v is specific heat (BTU/lb°F) and d is density (lb/gallon). With water at 1000 psi (68.9 bar) homogenizing pressure, a rise of 3°F (1.7°C) would occur. For water, $C_v = 1$ and $d = 8.330$ (at 70°F).

Requirements For Good Homogenization

The flow profile through the homogenizing valve has been discussed previously, but it is also beneficial to examine what occurs to the dispersed oil droplet as it undergoes homogenization. The homogenizing pressure represents the level of energy applied to the liquid as it goes through the valve. A portion of this energy subdivides the droplet, but most of it is converted into heat after homogenization is completed. It is estimated that less than 0.1% of the energy is used for emulsification. This is based on the work needed to create the new, total surface area in the final emulsion. This is not to say that the energy is wasted, because the subdivision of the droplets is a complex process, and homogenization requires this total energy level to initiate the action. For example, the initiation step may involve bringing the liquid to a certain velocity before efficient homogenization can occur.

Interfacial Tension

The work required to break down the droplet (of low viscosity) is a function of the interfacial tension (related to the surfactant) and also the diameter of the droplet. Reducing interfacial tension makes it easier to disrupt the droplet. As the droplet diameter decreases, the surface forces become more dominant in relation to bulk forces and resist disruption to a greater extent. This means, of course, that more energy is needed to reduce the droplet when its diameter becomes smaller. This is reflected in the shape of a curve comparing average droplet size to homogenizing pressure. As the homogenizing pressure increases, the average droplet size decreases; however, the rate of change in droplet diameter also decreases. Therefore, the curve is logarithmic and starts to level off at high homogenizing pressures. Although high homogenizing pressures are used, there is a limit to the size reduction with a single pass of the dispersed phase possible in the homogenizer. Some sources have suggested that this limiting value is about 0.1 to 0.2 μm .

Premix

The condition of the premix to the homogenizer is one of the most important factors influencing good homogenization. As was stated above, the energy needed to reduce a large droplet would be less than that needed to reduce a small droplet. Therefore, to make more efficient use of the high-energy homogenizer, the droplets should be reduced to as small a size as possible with low energy mixing, before going to the homogenizer. In this way, the energy of homogenization is not expended on reducing extremely large droplets to a medium size range, when this can be done with low energy equipment; but, rather, the homogenizer should be used to reduce medium-size droplets to a small-size range.

As a general estimate, the premix should contain droplets that are all under about 10 μm . The uniformity of the premix can also be important, because a very broad distribution of droplet sizes could lead to a homogenized product with a broad size distribution or a bimodal distribution.

Oil Content

Considering the previous description of the energy transfer in the valve, the effect of increasing oil content can also be visualized. With the same energy density in the liquid, the increased oil content will mean that each droplet experiences a smaller portion of the total energy. The result will be that the emulsion quality may not be quite as good when the oil percentage is increased. The homogenizer is most efficient when the oil percentage is less than 50%; and, of course, the efficiency is improved as the percentage is decreased below this level.

Dispersed Phase Viscosity

Experimentation has demonstrated the relationship between homogenizing efficiency and the viscosity of the dispersed phase or continuous phase of the emulsion. In an oil-in-water emulsion, the Newtonian viscosity of the oil is very important with respect to the efficiency of homogenization and, thereby, the average particle size generated. When measuring average droplet size versus increasing oil viscosity (for example, between 2 to 200 cP) at constant homogenizing pressure, experimentation found that the average droplet diameter steadily increases as viscosity goes up. Therefore, the homogenizer will produce its best results when the oil viscosity is low, usually less than 200 cP.

Sometimes this can be achieved by heating the emulsion premix or the oil phase to lower the viscosity of the oil. In some cases, such as resin emulsions, the oil phase is dissolved in a solvent that substantially reduces the viscosity of the oil phase. The only reasons for operating the homogenizer with a high temperature premix would be to lower the viscosity of the dispersed phase, to keep the dispersed phase in a molten state or to maintain aseptic processing conditions.

Continuous Phase Viscosity

It has been found that the continuous phase viscosity will also affect homogenizing efficiency. In this case it appears that the efficiency drops off as the viscosity increases to about 100 cP and then levels off. This phenomenon may relate to the mechanism of homogenization and is not totally understood at this time. In summary, then, it can simply be stated that the homogenizer will operate most efficiently when the dispersed and continuous-phase viscosities are low.

Particle Size Distribution

Many applications require a very uniform droplet-size distribution in the generated emulsion, either for control of creaming rate or for some physical action or characteristic required of the emulsion. This can be accomplished in the homogenizer by passing the product through the valve more than once. Because the homogenization process is random in nature, the size distribution follows a log-normal distribution curve. This means that the curve is asymmetrical and contains a “tail” representing oversized particles. With one pass through the homogenizer, there is a certain probability that not all particles are subjected to the same intense energy of homogenization; therefore, a portion of the particles passes through without being as reduced in size as others. Another pass through the homogenizing valve increases the probability of these large particles being reduced. Therefore, multipassing through the valve narrows the particle-size distribution. The benefit of multipassing diminishes after nine passes. The effect of multipassing is shown in Figure 4.

Two-Stage Homogenization

The two-stage homogenizing valve (a Gaulin invention) has been available for many years. Experimental testing has suggested that the primary function of the second-stage valve is to influence intensity of the homogenization effect in the first stage.

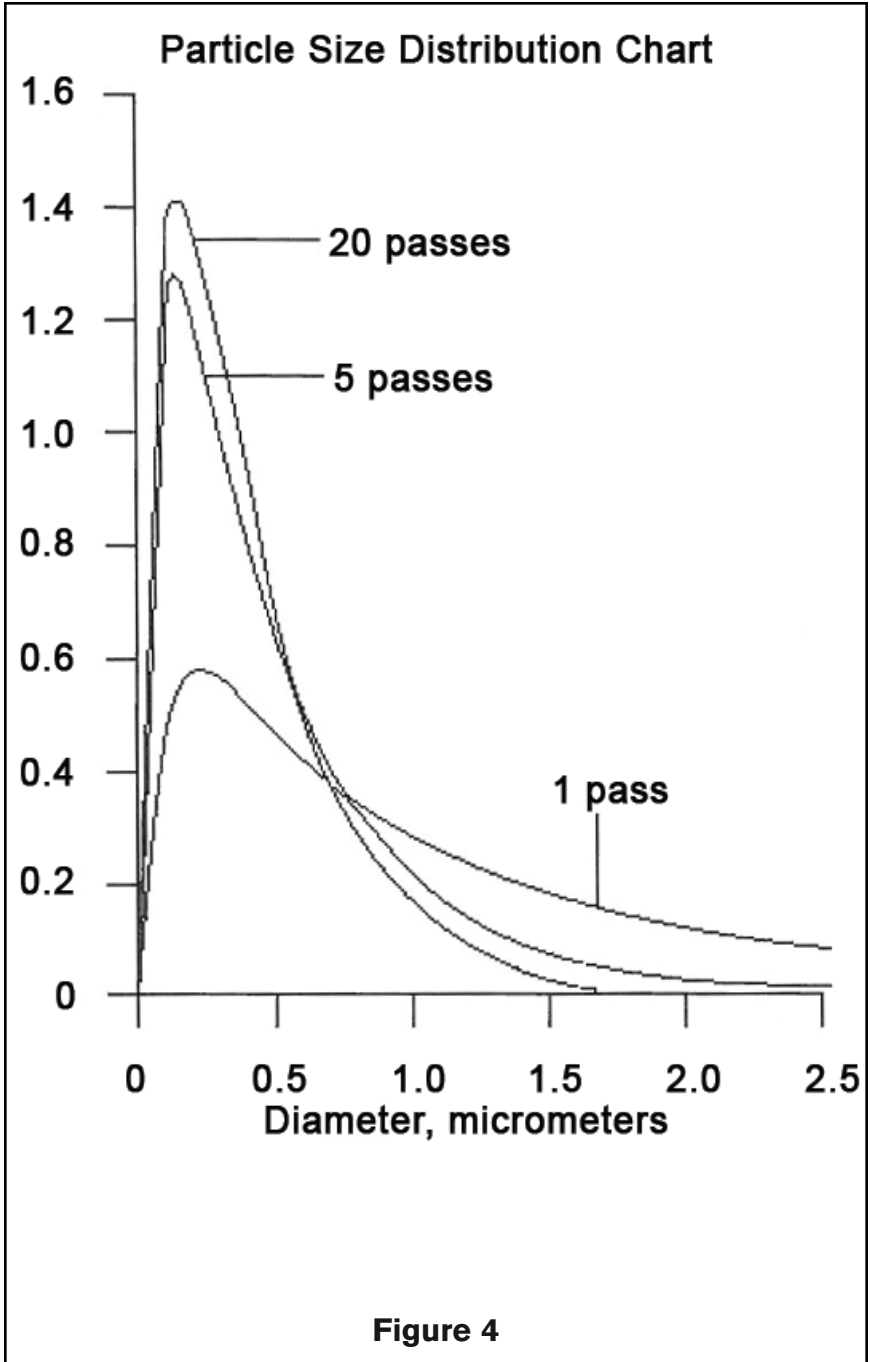


Figure 4

Therefore, the second-stage valve in a sense “fine tunes” the homogenization process. For emulsions, the second-stage backpressure suppresses cavitation, eliminates two-phase flow and intensifies turbulence. In oil-in-water emulsions, it has been found that the ideal second-stage pressure should be between 10 and 15% of the total homogenizing pressure. It has been demonstrated with milk that passage through the second-stage valve, itself, while maintaining a low backpressure as stated above, does not significantly alter the condition of the emulsion. Sampling the milk both before and after the second-stage did not reveal any substantial change in emulsion quality. The second-stage valve is a convenient means for applying the correct backpressure to the first-stage homogenizing valve. A homogenizer processing milk at 2000 psi (137.9 bar) with a two-stage valve may produce milk emulsion quality equivalent to a single-stage valve operating at 2200 to 2300 psi (151.7 to 158.6 bar). If the homogenizer has a maximum operating pressure of 2000 psi, then the two-stage valve configuration extends the achievable homogenization quality beyond that produced by a single-stage valve set at this maximum pressure.

There may be some products other than simple emulsions that have complex formulations that may be affected by the second-stage with respect to appearance or viscosity. The effect of the second-stage valve is further described in Technical Bulletin #58.

Homogenizing Efficiency Measurements

To determine the ideal operating pressure of the homogenizer on a particular product, the first step is to establish a method of evaluation. The method may be viscosity increase or decrease, average particle size (as determined microscopically or by instrument), rate of separation, grind gauge or change in physical appearance, just to mention a few. The next step is to collect samples from the homogenizer at different pressure settings; for example, every 1000 psi (or less, if a narrow pressure range is being evaluated). Finally, using the appropriate method of analysis, the evaluated parameter is compared or plotted versus homogenizing pressure and the lowest pressure that generates the required product can then be selected. For some products it may be desirable to determine the effect of multipassing at an appropriate pressure.

Applications For Gaulin And Rannie Equipment

Gaulin and Rannie homogenizers are used in the processing of many varied products. A listing and description of them all would be beyond the scope of this handbook; however, in order to demonstrate the diversity of the products benefiting from homogenization, a few will be briefly described.

The homogenizer is extensively used, of course, in the dairy industry for the processing of milk products and ice cream mixes. In the food industry the homogenizer is used for processing dispersions such as ketchup and tomato sauce, for emulsions such as orange oil and beverage emulsions and for frozen whipped toppings. It is also used in the production of cream soups.

In the pharmaceutical field, the homogenizer is used for dispersions such as benzoyl peroxide in cream or lotion bases, for antacid dispersions and for vitamin suspensions. Homogenization at high pressures is required for perfluorocarbon emulsions (also known as a blood substitute), intravenous emulsions and liposomes.

In biotechnology, the homogenizer is used for disrupting microorganisms, such as *E. coli* or yeast, to release active enzymes or proteins. The chemical industry uses homogenizers for the preparation of a myriad of products. Some dispersion applications include titanium dioxide and other pigments, clay and talc dispersions. Emulsion applications include wax and silicone oil (usually for low-viscosity oils).

The following information will be of interest to current colloid mill users. The colloid mill is used in a number of applications. Different products should be evaluated individually on the mill to determine the best balance of flow rate, temperature rise and emulsion quality. Food applications include the production of mayonnaise and salad dressings. Formation of silicone oil emulsions (high viscosity and high oil content) and processing of photographic gelatin are two chemical applications. Cosmetic products such as hand cleaners and skin creams have been successfully processed. These are just a few of the mill applications.

In general, the mill is best suited to products that are of high viscosity and high oil content. Because the energy level in the mill is much lower than that in the homogenizer, it does not “overwork” those high oil systems that can be overworked by the homogenizer. This effect is probably due to the type of particle-size distribution and average particle size generated by the mill. The total surface area of the emulsions made on the mill is not as large as that generated by the homogenizer; therefore, the surfactants can better accommodate this surface area, resulting in a more stable emulsion system.

Footnotes

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