

Stability of Beverage Flavor Emulsions

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Many of our food items are in emulsion form. An obvious example is milk. It is a natural emulsion of butter fat dispersed in a protein solution. Many of our processed foods are also in the form of an emulsion, for example, mayonnaise, salad dressings, ice cream, whipped cream, coffee whitener, cake batter, frankfurter, and others.

Beverage flavor emulsion is a special type of emulsion. It is prepared in the form of a concentrate and then diluted in sugar solution to make into soft drinks. The soft drinks can be non-carbonated still or carbonated drinks. In soft drinks the flavor emulsion provides flavor, cloudiness and color for the beverage. These properties make the beverage flavor emulsions different from other food emulsions. Although for coffee dairy cream or coffee whiteners are used in the diluted form in the beverage, they are consumed almost immediately, and the stability in the diluted form is of no concern. On the contrary, beverage flavor emulsions are required to have good stability in both concentrate and diluted form of a finished beverage.

Beverage flavor emulsions are oil-in-water (O/W) emulsion. A typical composition of orange flavor emulsion includes orange oils and weighting agents (e.g., ester gum, BVO, SAIB and dammar gum). The water phase usually contains water, hydrocolloid (e.g., gum arabic), citric acid, preservatives and colorings (e.g., FDC colors and β -carotene). Due to the shortage of gum

arabic in the last two years, the other kinds of gums and modified starches are being used as substitutes. Hydrocolloids serve as a stabilizer for the emulsion. Some hydrocolloids, such as gum arabic, also provide emulsifying properties in the emulsion. Citric acid is used for pH control, and benzoate as preservative. Both are for the purpose of preventing microbiological spoilage. Colorings are for aesthetic purposes. In the oil phase, flavor oil is usually composed of essential oils or citrus oils. Weighting agents can also be called density adjusting agents, i.e., materials which are oil soluble with no flavor of their own and have density higher than the flavor oil. As the name implies, they are added to flavor oil to increase the oil phase density and, with the flavor oil, become cloudifiers.

Before 1970, brominated vegetable oil (BVO) was allowed to be used as a weighting agent with no limitation. It was banned in the United Kingdom in 1970 because of concern about the accumulation of bromine in body fat from ingestion of BVO. It was regulated by the Food and Drug Administration (FDA) in 1970 to allow a maximum of 15 ppm in soft drinks in the United States well below the effective level required for a stable emulsion. BVO is a unique weighting agent for citrus oils as it has a very high density of 1.33 gm cm⁻³. It can be easily added to citrus oils to increase the density.^{1,2}

Since the regulation of BVO, it was necessary for the soft drink industry to find other appropri-

ate materials for use as the weighting agents for citrus oils. In the U.S., ester gum is a material approved by the FDA for use as a weighting agent but with a limit of 100 ppm maximum in soft drinks. The chemical composition for ester gum is glyceryl abietate. It is a product derived from wood rosin or tall oil. In Canada, the permitted weighting agent is sucrose acetate isobutyrate (SAIB), and for the countries belonging to the Council of Europe, dammar gum. The shortcoming of all these new weighting agents is that they all have a density much lower than BVO. The density of ester gum is 1.08 gm cm⁻³; SAIB is 1.15 gm cm⁻³, and dammar gum is 1.06 gm cm⁻³. Because of government regulations and the low density of these weighting agents, a stable beverage flavor emulsion in both the concentrate and diluted forms has become more difficult to make than when BVO could be used without limitation.

Problem of Beverage Flavor Emulsion Stability

Since the beverage flavor emulsion is made first in a concentrate form and then diluted several hundred times with sugar solution to make the finished beverages, good stability is required in both forms. The stability requirement often is at least six months or longer.

Because of the low density of the flavor oils, if the emulsion is not properly prepared, it will not have the required stability. The instability of the emulsion in the concentrate and in the soft drinks can be manifest as creaming (ringing), flocculation or coalescence.

Creaming (Ringing)

Creaming is a term adapted from the separation of cream of unhomogenized milk. In the industry the common term for creaming in bottled soft drinks is "ringing" because the flavor emulsion separates from the soda and floats to the top and shows a white creamy ring at the neck of the bottle. Creaming or ringing is related to flocculation. It can be considered as a separation of one emulsion into two emulsions, that is, one is richer in the oil phase than the original emulsion, and the other, just the opposite, is richer in the water phase. When creaming occurs, the emulsion richer in the oil phase rises to the top and forms a creamy layer which is not only unsightly but also indicates the breakdown of the distribution of the flavor oil in the soft drink.

In relation to creaming, two not commonly observed phenomena which have been seen in



bottles of soft drink are "lifting" and "striation." Lifting occurs when the emulsion lifts up from the bottom of the bottle and shows a clear layer of liquid at the bottom. Striation occurs when the emulsion in the bottle shows two or three distinctive layers with different degrees of cloudiness. It looks like the emulsion has classified itself into two or three different particle sizes or density fractions. Lifting and striation only show in bottles which have been held in an upright position undisturbed for a long period of time. They are more often seen in bottles which are kept in the refrigerator where the temperature is low and the Brownian movement and thermal convection of the emulsion particles are less active.

A rather special and rare situation found in soft drinks is "sedimentation." Sedimentation is also called "downward creaming" by the soft drink industry. It happens only when the weighting agents separate from the flavor oil and become precipitates in the soft drinks.

Flocculation

Flocculation occurs when oil droplets of the dispersed phase form aggregates or clusters without coalescence. At this stage, the droplets still retain their original identities. The forces which draw these droplets together to form aggregates are primarily the long range London-van der Waals forces and electrostatic forces around the droplets.³ From the creaming point of view, these aggregates behave as simple large drops, and the rate of creaming is accelerated in systems in which the density difference of aggregates from the continuous phase is sufficiently large. In emulsion concentrate, a perceptible increase in emulsion viscosity can be observed when flocculation occurs. Although flocculation generally changes the physical properties of the emulsion, the particle size distribution remains unchanged. In the soft drink system, the droplet concentration is so low the flocculation often is reversible. The aggregates can be readily redispersed when the interactions between the droplets are weak.

Coalescence

In this step, there is localized disruption of the sheaths around neighboring droplets of the aggregates, and the oil droplets merge together to form a larger droplet. It leads to a decrease of the number of oil droplets and eventually causes the breakdown of the emulsion. When a proper

hydrocolloid is used in the water phase, the breakdown of the emulsion will seldom reach this stage. The reason is that the hydrocolloid, such as gum arabic, has the ability to form film around the oil droplets and, in addition, to provide viscosity in the water phase. More about the film forming property of gum arabic will be discussed later.

Stabilization

For beverage flavor emulsions, the most critical criterion of stability is that in the finished soft drink where the emulsion concentrate is further dispersed in sugar solution. The stability of an emulsion in the concentrate is much easier to achieve than in the soft drinks.

Stokes' Law

The ringing test is the most popular method to evaluate the stability of beverage flavor emulsions in soft drinks. It is a simple test in which bottles of soft drinks containing the beverage emulsion are held in an upright or horizontal position for observation of ringing. The rate of ringing or creaming of a oil droplet in sugar solution may be determined by equating the force of gravitation with the opposing hydrodynamic force as given by Stokes' law:

$$v = \frac{2gr^2(d_1 - d_2)}{9n_2} \quad (1)$$

In equation 1, v (cm/sec.) is the rate of creaming or sedimentation, $g = 980$ (the acceleration of gravity in cm/sec.), r is the droplet radius in cm, d_1 (gms/cm³) is the density of the oil phase, d_2 is the density of the water phase, and n_2 (gm/cm sec.) is the viscosity of the water phase. In an O/W emulsion or a soft drink, the oil density, d_1 , is smaller than that of the sugar solution, d_2 . The resulted sign of v is negative, hence, creaming or ringing will occur.

As an example, orange flavor emulsion is used to make orange flavor soft drinks. Typically the major components of orange flavor, orange oils, have a density of 0.85 gm cm⁻³, and the sugar solution in the soft drink has a density of 1.04 gm cm⁻³ for a 10 percent sugar solution, or 1.048 for a 12 percent sugar solution.

Applying these density data to Stokes' law, the resulted v carries a negative sign, which indicates the emulsion will ring. From Stokes' law, it shows that the rate of a droplet to move to the top, v , is directly proportional to the density differ-

ence of the oil phase from the water phase, and to the square of the radius of the droplet. It is also inversely proportional to the viscosity of the water phase. The equation clearly shows that if one can make orange oils with density as heavy as the sugar solution, then there will be no creaming problem because when $d_1 = d_2$, ($d_1 d_2$) = 0, then $v = 0$.

Since orange oil is lighter than the sugar solution in the soft drink, weighting agents have been added to orange oil to increase the density. For this purpose, brominated vegetable oils (BVO) have been used for many years. There are two types of BVO, with densities of 1.24 and 1.33 gm cm⁻³, and both are effective weighting agents.

The FDA limitation on BVO has forced the beverage industry to use the other weighting agents mentioned before. Among them, ester gum (glyceryl abietate) is the only one approved by FDA to be used in the United States, regulated at 100 ppm maximum in the beverage. With ester gum having a density of 1.08 gm cm⁻³, the limitation makes impossible the adjustment of the density of orange oil equal to that of the sugar solution. An equal weight of ester gum added to orange oil can only bring the density from 0.85 to 0.95 gm cm⁻³, still a big difference from 1.05 gm cm⁻³, the density of a 12 percent sugar solution. According to Stokes' law, an emulsion with these components will cause a ringing problem in the beverage.

Because of consumers' preference for the strength of the orange flavor and the 10-12 percent sugar sweetness level in the beverage, there is little a soft drink processor can do to narrow the density difference between the oil phase and water phase except to make diet drinks. Therefore, if we have ($d_1 d_2$) and n_2 equal to constants, where g , gravity, is also a constant, the rate of an oil droplet to move upward, v , is in direct proportion to r^2 . The guidepost here shows that reducing the droplet size is one way to control the creaming velocity. For instance, in a beverage a particle of 0.1 μm in diameter will travel upward 100 times more slowly in the bottle than a particle of 1.0 μm in diameter. However, a creaming velocity on the order of 1 mm in 24 hours is usually sufficiently counteracted by accidental thermal convection. Application of Stokes' equation to an O/W emulsion at room temperature, with the oil density of 0.8 that of water, and with $n = 0.01$, leads to the conclusion that a particle radius of 0.16 μm will have a flotation velocity of 1 mm per day and will not cream.



Adsorption at Interfaces

Most of the beverage flavor emulsions are made with the water phase composed of gum arabic or other hydrocolloids and water. It has been known for many years that gum arabic solution produces a film at an oil interface.

Shotton and White showed this interfacial film to be visco-elastic, and it seemed to take three to four days to mature to a "solid," possibly multi-layer, film.⁴ They used a technique to inject a drop of paraffin oil into a potassium arabate solution, held the drop at the tip of a micrometer syringe and examined the interfacial film formation under a microscope. After aging the drop for fifteen minutes in the solution the drop was withdrawn. A wrinkled film like a collapsed plastic bag was left indicating the interfacial film consisted of the deposits of gum arabic materials (see figure 1).

Shotton and White further estimated the thickness of the film to be approximately $0.1 \mu\text{m}$, and it apparently possesses some elastic properties. Since gum arabic solution is heavier than water, a layer of hydrated gum arabic will provide an added weight to the oil droplet and actually change the density of the total droplet, that is, the oil droplet plus the gum arabic film, to a higher value. When an oil droplet is smaller, the percentage of weight contributed by the gum arabic layer to the total droplet weight is larger than that for a larger droplet. This further indicates that the oil droplets should be made small in order to benefit more from weight contributed by the gum arabic layer.

The formation of the interfacial film by gum arabic or other hydrocolloid polymers on the oil droplets helps to stabilize the emulsion in one more way: the gum arabic material adsorbed on the surface of the oil droplets prevents oil droplets from coalescence and forming larger droplets which may eventually lead to emulsion breakdown. Thus an adsorbed polymer layer on the droplets holds the droplets far enough apart to keep the van der Waals attraction force minimal.^{3,5} In this way, the droplets will remain dispersed. When an emulsion is sterically stabilized by adsorbed polymers, the mechanism is described as steric stabilization.

In many classic emulsions where emulsifiers or surfactants are used, these emulsifiers are adsorbed on the interface of oil droplets as a closely-packed monomolecular film and reduce the surface tension. Gum arabic performs somewhat the same way but in a much weaker manner. This

is because gum arabic does not have the distinctive polar and non-polar groups that real emulsifiers have.

Electrostatic Interaction

The possibility that electrical charge on particles may be of significant importance to the stabilization of emulsions was recognized early this century, but the elaboration of the electrical theories was only developed in recent years.⁶

In an oil-in-water emulsion, the oil particle may acquire electric charge through the ionization of the adsorbed surface charged group. In beverage emulsions, gum arabic is used in the water phase. Gum arabic is an acidic polysaccharide. The carboxyl ion (COO^-) provides the negative charge. Surface charge may also be acquired through adsorption of the dissolution of small ions in the water phase. Anions have a greater tendency to be adsorbed than cations. Cations are normally more hydrated and prefer to stay in the aqueous bulk solution.

The electrical charge may be acquired also through a possible friction mechanism. An empirical physical rule may be applied to this theory. This rule states that a substance having a high dielectric constant is positively charged when in contact with another substance having a lower dielectric constant.⁷ Since water has a dielectric constant much higher than most of the substances which are likely to be the other phase of the emulsion, oil droplets will probably have a negative charge.

With the presence of the charges on the droplets, these charges give a repulsion force among the charged particles and that force prevents flocculation, thus, contributing to the stability of the emulsion.

As mentioned above, dispersed oil droplets may acquire an electric charge through the ionization of a surface charged group. Oppositely charged ions (counter ions) are preferentially attracted towards the surface; ions of the same charge (co-ions) are repelled from the surface. The region of unequal counter- and co-ion concentrations near the charged surface is called the electrical double-layer. The double layer may be regarded as consisting of two regions: an inner region of strongly adsorbing ions and an outer region where ions are diffusely distributed according to a balance between electrical forces and random thermal motion.⁸

At some point out in the double layer region and corresponding more or less to the potential at

Table I. Stability Characteristics of Anionically Dispersed Systems⁹

<u>Stability Characteristics</u>	<u>Zeta Potential in Millivolts</u>
Maximum agglomeration and precipitation	0 to +3
Range of strong agglomeration and precipitation	+5 to -5
Threshold of agglomeration	-10 to -15
Threshold of delicate dispersion	-16 to -30
Moderate stability	-31 to -40
Fairly good stability	-41 to -60
Very good stability	-61 to -80
Extremely good stability	-81 to -100

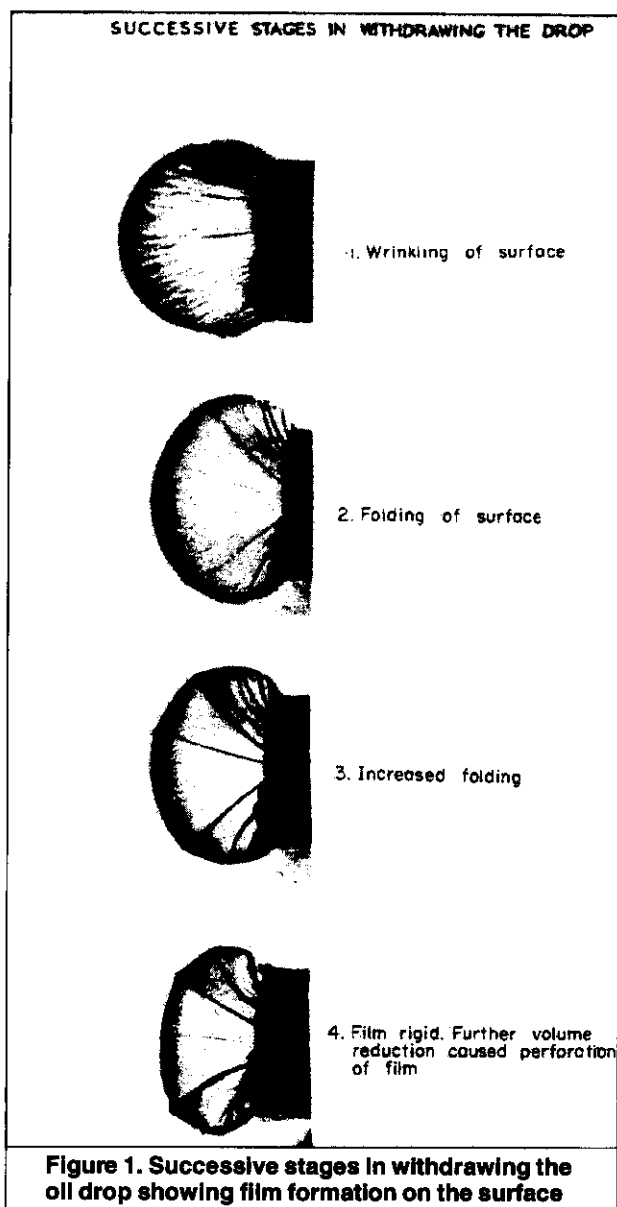
the zone of shear, this electrical potential is called "zeta potential." The determination of zeta potential is extremely important in the study of emulsion stability. It is an important parameter for both achieving emulsion stability and destroying the emulsion stability. According to Riddick, an emulsion should have a zeta potential of -40 millivolt to be stable.⁹

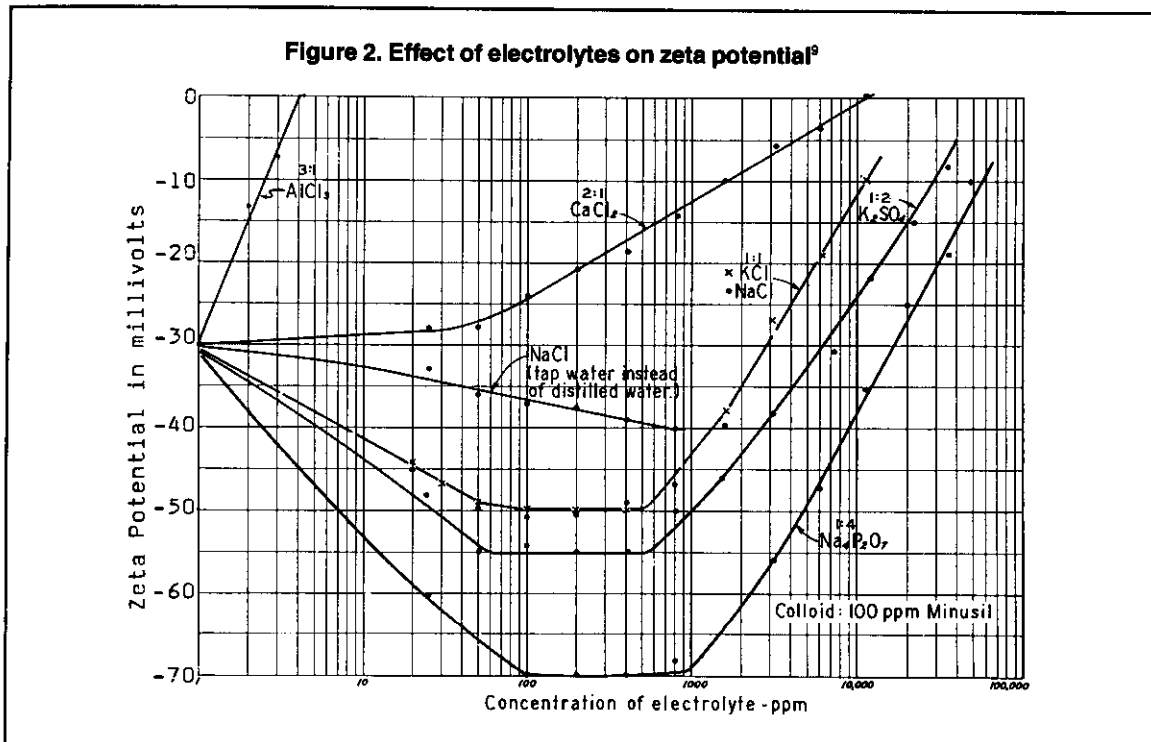
Zeta potential values of less than -15 mv usually represent the onset of flocculation. The threshold region of either flocculation or dispersion exists from about -15 mv. to -30 mv. Values more negative than -30 mv generally represent sufficient mutual repulsion to result in stability.⁹

Gum arabic solutions generally have a zeta potential about -23 mv.^{5,10} However, one cannot categorically state that an emulsion will or will not be stable at a given zeta potential, particularly in the delicate zone of -15 to -30 mv as some other factors should be taken into consideration.

In the case of beverage emulsion, two other important factors are the density difference between the two phases and the droplet sizes. Table I is a general stability table for anionically dispersed systems based on zeta potential as prepared by Riddick.⁹

It should be stressed that zeta potential reflects both the electrolytes' presence in the system and the dissociated ions accompanying the original colloid particles. When a cation electrolyte is added to an emulsion, where its particles carry negative charges, the electrolyte will be adsorbed and neutralize the zeta potential, and cause aggregation to occur due to the van der Waal's-London forces. This electrolyte effect on emulsion stability is much more evident in soft drinks than in emulsion concentrates because





gum arabic is known to produce stable emulsions over a wide range of pH and in the presence of electrolytes.⁴ In soft drinks the emulsion is in a very diluted form and the gum arabic concentration is very low; therefore, the electrolyte effect is more obvious.

In the adsorption and desorption of electrolyte ions causing the change of zeta potential, the higher the valency of the ions the greater the effect of change of zeta potential. Trivalent ions, like aluminum ions, have 10 to 100 times the effect of equivalent concentrations of divalent ions, like calcium ions, which in turn have 10-100 times the effect of equivalent concentrations of univalent ions. The effects of electrolytes on the emulsion stability is shown in Table II and figure

2. Figure 2 shows the dispersion of colloidal silica Minusil in distilled water employing 3:1 to 1:4 electrolytes. The initial zeta potential of the colloidal silica in distilled water is -29 ± 2 mv. The contrast of the shape of curves for the 3:1, 2:1, and 1:1 electrolytes, and that for the 1:1, 1:2, and 1:4 electrolytes is remarkable. For this reason, the soft drink industry usually uses only treated water to make emulsions and the finished beverages.

Particle Size Characterization

To achieve good emulsion stability, it is important to control the oil droplets' particle size. This is especially true for beverage flavor emulsions because their particles have to be stable both in the concentrate and in the diluted finished beverage. This means that one oil phase must be stable in two water phases of different composition. In controlling the particle size, it is important to determine the size distribution of the oil particles in the emulsions. Two emulsions may have the same average droplet diameter and yet have dissimilar stability because of differences in their distribution of particle sizes.

For beverage flavor emulsion, the determination of particle size distribution serves two purposes: one is to estimate the quality of the emulsion concentrate, and the other is even more im-

Table II. Reagent Dosages Required to Bring a Diluted Silica Suspension to Zero Zeta Potential⁹

Type of Reagent	Reagent	Dosage	
		PPM	Millimols
3:1	AlCl ₃	4	-
2:1	CaCl ₂	12,000	700
1:1	KCl & NaCl	22,000	300
1:2	K ₂ SO ₄	55,000	300
1:4	Na ₄ P ₂ O ₇	110,000	450



portant, which is to predict the stability of the emulsion when it is made into the finished beverage.

Many methods are available for the determination of particle size distribution. These methods of particle characterization have been reviewed recently by Groves with emphasis on those methods that are of actual or practical use for the emulsions containing smaller particles.¹² The beverage industry commonly uses the following methods for product development and quality control works: optical microscopic method, light scattering or turbidity measurement and electronic counting.

Optical Microscopy

Optical microscopy for sizing the particles is invaluable for subjective observation because the operator is directly involved. The operator has to judge the size or shape of dispersion based upon past experience.

Since the beverage flavor emulsion is composed of transparent particles dispersed in a transparent liquid, the contrast of the image can be accentuated by using phase contrast techniques. When using phase contrast microscopy, an experienced operator can view and estimate the particles down to $0.5\ \mu\text{m}$ at $1000\times$ magnifications. The routine use of an optical microscope is essential in particle characterization, even if it is used only to examine the state of dispersion without making measurements.

In practice, it is common to use an optical microscope in conjunction with other instrumental analysis. Using optical microscope for objective measurement is very tedious, and human error is inevitable. A statistically significant count, allowing a 5 percent error and a 95 percent confidence level, would need 2960 particles.

Usually the emulsion has to be diluted before being placed on the microscope slide, and Brownian movement of particles usually is a problem for particle counting. Gelatin solution, low viscosity CMC solution and glycerine in water can be used to "freeze" the particles. However, if the emulsion is not distributed evenly in the dilution clumping will occur and error cannot be avoided.

Light Scattering and Turbidity

In general, two types of light scattering methods are used: angular light scattering—the intensity of the scattered light is measured as a function of the scattering angle, and turbidity or

transmittance measurements—the intensity of the transmitted light is measured. In principle, angular light scattering method contains more information, but turbidity measurement is easier to carry out by using a spectrophotometer. For routine particle size analysis or stability tests, where precision is not highly critical, turbidity provides a convenient monitor of the state of the emulsion. However, turbidity measurement only translates to average particle size.

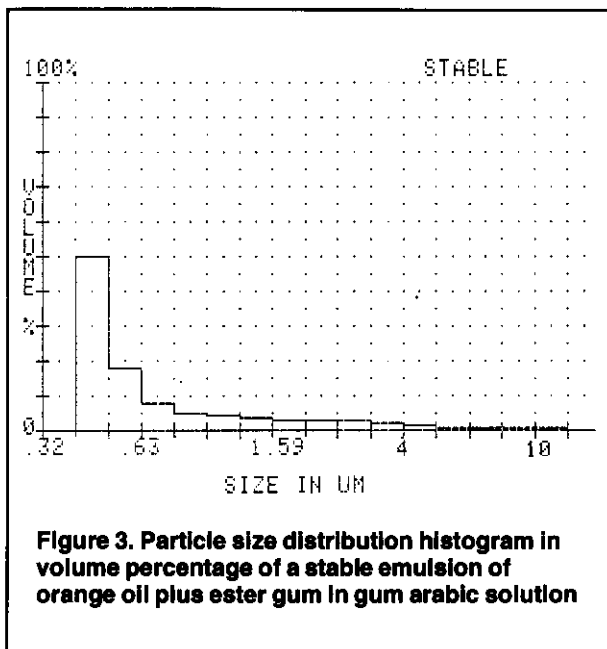
Using a spectrophotometer, light absorption may also be used to provide an estimate of the mean particle size and spread around the mean if measured at different wavelengths. Practical methods are described by Goulden¹³ and Walstra¹⁵ for estimating the fat globules size in homogenized milks.

In the soft drink industry, turbidity or transmittance is used as a quick method to check emulsion quality. Another method is to measure the spectral adsorption of diluted emulsion at two wavelengths, such as 850 nm and 450 nm, or 800 nm and 400 nm, and use the ratio of the readings as stability indices.^{16,17}

Electrical Counting

Particle counting using a Coulter Counter has been known for many years in the pharmaceutical industry.¹⁸ It has been found recently to be valuable for particle size characterization of beverage flavor emulsions. This instrument determines the number and sizes of particles suspended in a conductive electrolyte solution by forcing the suspension to flow through a small aperture and monitoring an electrical current which also passes through the aperture. Electrodes are immersed in the conductive liquid on either side of the aperture. As a particle passes through the aperture, it changes the resistance between the electrodes. This produces a current pulse of short duration having a magnitude proportional to the particle volume. The series of pulses is electronically scaled and counted.

It is generally agreed that the Coulter Counter is capable of making a unique and valuable contribution to the subject of particle size characterization.¹² When the more sensitive model, Coulter Counter Model TA II, equipped with a $16\ \mu\text{m}$ aperture tube is used in an ultra clean electrolyte solution and low electrical noise environment, it will detect particles as small as $0.3\ \mu\text{m}$. This electrical counting technique is much faster than the optical microscopic method. The shortcoming of using such a small aperture tube is that



clogging by gum materials will happen sometimes, and it will take time to clean the aperture and repeat the counting.

In our work on beverage flavor emulsion using the Coulter Counter, we chose to have the particle size distribution expressed in volume percentage per each size category rather than in population percentage. The reason is that volume percentage distinctively shows the quantity in volume of the large particles present in the emulsion. It can be clearly illustrated in the following example. For instance, in one emulsion, there are one thousand dispersed particles of 1 μm diameter, and one particle of 10 μm diameter. By population, this 10 μm particle is only 0.1 percent of the total population. When expressed in volume percentage, this 10 μm particle having 50 percent of the total dispersed particles volume as the volume of one thousand 1 μm particles is equal to that of the one 10 μm particle. In the beverage if this 10 μm particle floats to the surface and rings, it is almost a negligible number in the total population, because it is only 1 in 1001 particles or 0.1 percent of the total population rings. But, in terms of volume, it is 50 percent of the total oil volume in the ring. It is a serious problem.

To set the criteria of particle size distribution for determining the stability of an emulsion, one has to realize that the criterion is different for each emulsion as each emulsion has a different composition which influences the physical and

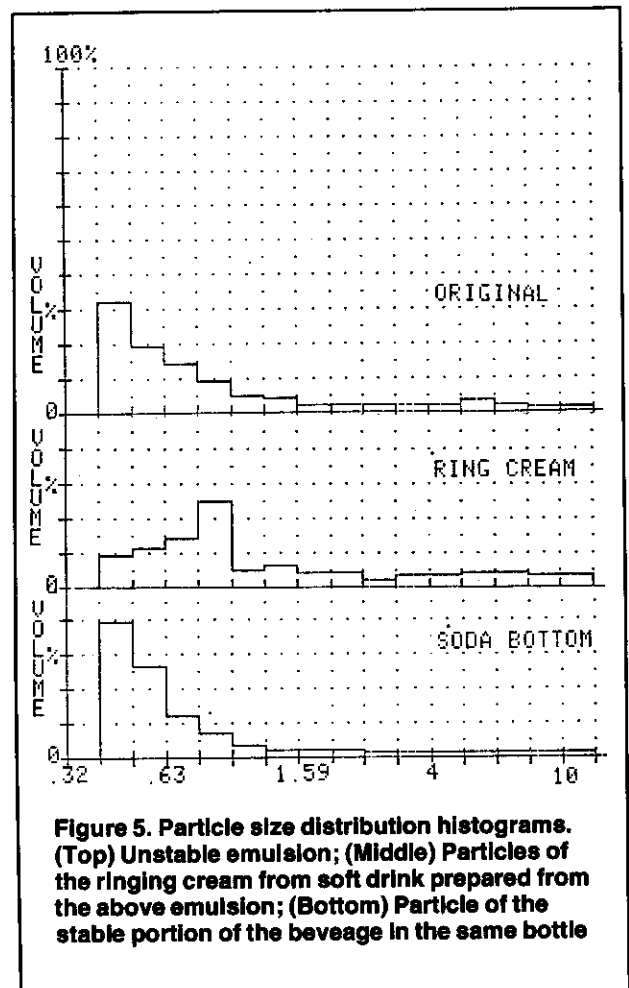
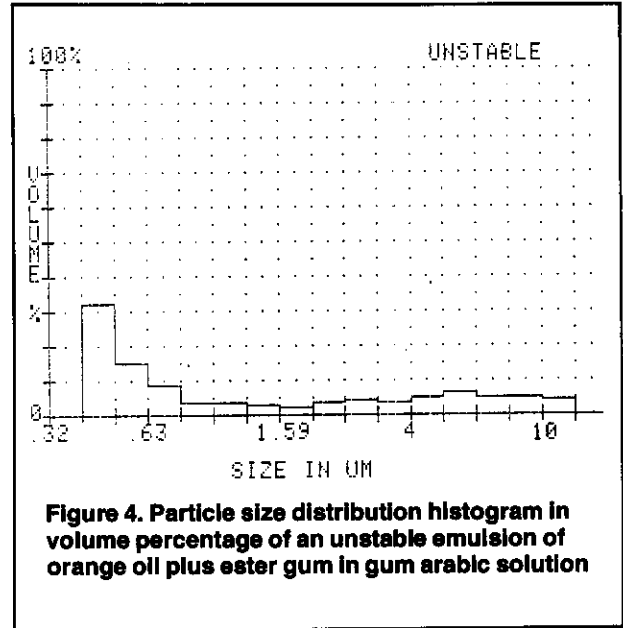
chemical properties of it. Examples of particle size distribution of a stable emulsion and an unstable emulsion of orange oil/ester gum in gum arabic solution type are shown in histograms A and B (figures 3 and 4). In histogram A, the particle volume of the most critical channel, Channel 2 (0.4–0.5 μm), is over 50 percent, and the total volume of Channels 1 to 5 (0.32–1.0 μm) is over 80 percent. In histogram B, the particle volume of Channel 2 is slightly over 30 percent, and the total volume of Channel 1 to 5 is only over 60 percent, and there is a heavy tail portion of particles larger than 1.0 μm .

To set the criterion of particle size distribution for a beverage flavor emulsion, a series of data has to be collected in regard to the relationship of different particle size distribution patterns to different degrees of ringing in soft drink. A "quick and dirty" method to determine the stable emulsion particle size distribution of a beverage flavor emulsion is to prepare a soft drink from this emulsion. Let the bottle stand for a required length of time, such as a week or longer. Then, determine the particle size distribution of the soft drink if there is no ringing, or determine the non-ringing portion of a ringing bottle where the particles are in good suspension as shown in the particle size distribution histogram of the bottom portion of a ringing soda in figure 5.

Instead of using particle size distribution in volume percentage as the criterion for stable and unstable emulsion, Johnson reported the use of the total interfacial area of the particles as determined by Coulter Counter for BVO/ester gum emulsions with satisfactory results.¹⁹ It was determined that at total interfacial area greater than 11 $\text{m}^2 \text{cm}^{-3}$, no ring formed. He also used the total interfacial area data as a guide for improving homogenization performance and adjusting the emulsion composition.

Conclusion

Beverage flavor emulsion is unique. It is prepared in the concentrated form and used in the diluted form. It has to be stable in both the concentrated and diluted forms in which the water phases are of different compositions. The problems of producing a stable emulsion can be solved by application of colloidal chemistry principles. Factors influencing emulsion stability are: the particle size distribution in the emulsion, density balance of the two phases, adsorption of solid film at interfaces and electrostatic charges on the droplets.





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