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Mechanisms and Control of Gas Bubble Formation in Cosmetics

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Synopsis—The presence of GAS BUBBLES in cosmetic and pharmaceutical preparations is generally undesirable not only from the appearance viewpoint, but also from physical, chemical, and biological stability considerations. The air bubbles in a product can affect emulsion stability, promote oxidation, and, in some instances, encourage microbial growth. EXTERNAL ENTRAINMENT of gas bubbles can result from mixing, pumping, pouring, jetting operation, or addition of powdered materials. INTERNAL GENERATION of gas bubbles may be due to chemical reactions or physical changes. Each MECHANISM of bubble formation is analyzed and some possible solutions are suggested.

INTRODUCTION

Control of dissolved and undissolved gases in a product is a difficult but extremely important problem in cosmetic and other industries. Some cosmetic preparations, such as shampoos and aerosol shaving creams, are designed to produce many gas bubbles or foam during their usage. In some food preparations the presence of air bubbles is quite essential. However, in most cosmetic and pharmaceutical preparations, the presence of air or other undissolved gases as bubbles is generally undesirable for several reasons. The presence of air bubbles in cosmetic emulsions not only makes the texture appear coarse, but it can also affect the emulsion stability by adsorbing the emulsifier molecules at the air-liquid interfaces (1). The trapped air bubbles in an emulsified cream

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or fluid make-up can sometimes encourage microbial growth or undesirable oxidation. The pinholes in a lipstick or make-up stick can create poor appearance and weaken the stick. Excessive incorporation of air in clear gel-type products can be unsightly or create an under-fill appearance upon standing on the shelf.

Air bubbles in cosmetics are usually introduced during the manufacturing processes. If the viscosity of the product is low, the incorporated air can escape into the atmosphere in a short time and the product will become bubble-free. If, on the other hand, the product is very viscous, the rate of escape may be very low. From the rheological viewpoint, if the product has an appreciable yield value, the entrained bubbles may not escape at all and the product can remain aerated during the entire shelf life. Control of gas bubbles is thus particularly important in plastic or thixotropic products. However, since there are many ways by which gas bubbles can be incorporated during numerous manufacturing steps, control of gas bubbles in finished products is not always easy.

The purpose of this paper is to examine in detail the common sources and mechanisms of bubble incorporation into cosmetic preparations during various manufacturing processes, and to suggest preventive methods.

Discussion

External Entrainment

The mechanisms of bubble formation often encountered in cosmetic preparations may be divided into two main categories: bubble incorporation due to the entrainment of the external gas and bubble formation due to the internal generation of gases. There are many variations but four important operations which are frequently the sources of external entrainment of air will be discussed.

Mixing Operations

Broadly speaking, mixing covers agitation, blending, homogenizing, and milling; it is probably the most common source of air incorporation into products such as facial creams, hand lotions, and liquid make-ups.

Propeller or turbine mixers are widely used to process flowable liquids or emulsions. Depending on the mixer speed, impeller size, and location, a vortex such as the one illustrated in Fig. 1 can form. If the vortex

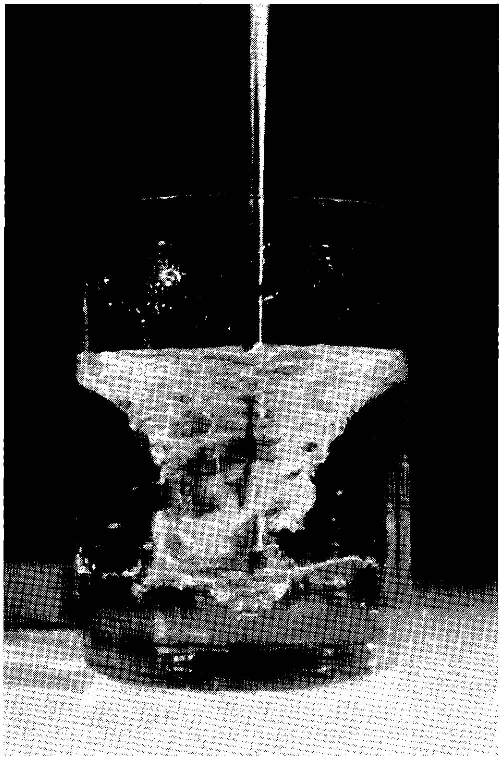

Figure 1. Aeration from vortex formation

is deep enough to touch the impeller, the surrounding air may be sucked in to form bubbles.

Except when it is desired to deliberately aerate the fluid or to draw the floating material from the fluid surface (e.g., to wet the dry pigments placed on the fluid surface), the formation of vortex is generally undesirable as it will reduce the mixing efficiency. A reduction in the mixer rpm may eliminate the vortex but it will also reduce the intensity of mixing and shear force required to obtain a fine emulsion. In a jacketed kettle, it may also reduce the rate of heat transfer and result in poor cooling or heating of the batch.

One way to suppress the vortex and, at the same time, increase the mixing efficiency is to use baffles (2). In a cylindrical tank, for example, four vertical baffles, each one-tenth tank diameter in width, placed equally around the tank will serve such a purpose. The baffles reduce the tangential velocity component but increase the radial and vertical flow. The effect is that the fluid does not merely rotate around the tank axis

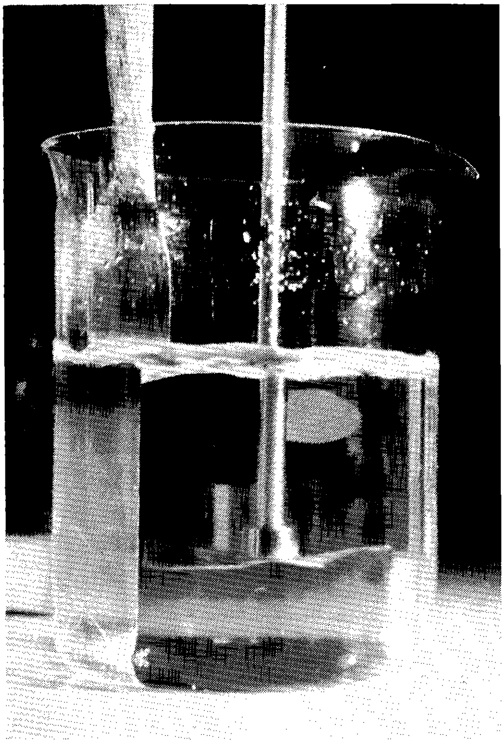

Figure 2. Elimination of vortex by using spatula

but is mixed uniformly. The photograph in Fig. 2 was taken at the same mixer rpm as Fig. 1 but a spatula was placed on one side of the beaker to serve as a baffle. The absence of vortex and bubbles clearly indicates the effect of the baffle.

Another effective way to suppress a vortex when using a propeller mixer is to tilt the mixer and place it off-center. This will create a difference in the velocity with which the fluid impinges on the tank wall and a vortex formation is thus prevented without using baffles.

Some high-speed mixers are provided with built-in baffles to minimize the chance of surface turbulence and aeration. However, it is generally better to place the head of a high-speed mixer as near the bottom of the process kettle as possible to avoid air entrapment.

In the cosmetic industry, an anchor-type mixer is often used to process high-consistency creams. Although such a mixer is normally operated at a relatively low rpm, air entrapment is still possible if the mixer tips are not completely covered by the product. As illustrated in Fig. 3,

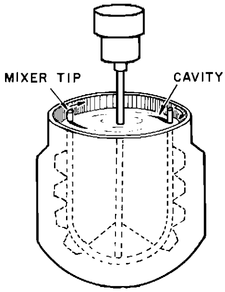

Figure 3. Cavity formation in anchor-type mixer

if a high-consistency cream is made in such a kettle, the uncovered mixer tips can create cavities behind them as they rotate in the cream. As these trailing cavities are filled, air is often trapped and dispersed into the product. For a similar reason, a planetary-type mixer can often incorporate a considerable amount of air into a viscous product during its operation.

The above problem in an anchor-type mixer can be solved simply by increasing the batch size or shortening the mixer tips so that they are completely covered by the product. But in other cases, it may be necessary to redesign the mixer so that such cavities are not created during the mixer operation.

Paddle mixers are also widely used in the cosmetic industry to process creams and pastes. In using these mixers, the fluid level should be sufficiently high to cover the paddle completely so as to avoid excessive surface turbulence. A processing kettle equipped with a paddle mixer fitted with scraper blades plus a built-in high-speed homogenizer is quite ideal in processing cosmetic emulsions and suspensions. If necessary, such a kettle can be designed to operate under vacuum for a completely air-free operation.*

In some instances, bubble entrainment in a mixing process can be minimized by revising the manufacturing procedure. For example, the emulsifiers used in making a cream can often encourage bubble formation during the emulsification. Since cosmetic emulsions are usually made at a high temperature at which the viscosity of the emulsion is usually low, most of the entrained bubbles can escape during the initial stage of emulsification. To obtain a good emulsion, mixers or homoge-

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* Eppenbach Agi-Mixers made by Giffor-Wood Co. of New York, N.Y., have these features.

nizers should be turned on at high speeds during this stage. After the cooling water is turned on, the temperature of the batch will drop and the viscosity as well as the yield value of the emulsion will increase greatly. Since any entrapped air will have difficulty in escaping after this period, further mixing should be done with extreme care to avoid permanent aeration. If possible, the thickeners used to increase the viscosity of the emulsion should not be added at the beginning but should be added after emulsification to allow the entrained air bubbles to escape (3).

Pumping

Considerable air can be entrained sometimes when a cosmetic preparation is pumped from one kettle to another. A common source of aeration in this type of operation is a leakage in some section of the line. This can be due to a faulty gasket, a poor connection, or pinholes in the line. The entire line should be inspected for the source of leakage. Since air incorporation is most likely to take place in the suction side of the pump, it is generally advisable to connect the pump as closely to the originating tank as possible to reduce the number of connections in the suction side and thus reduce the chance of air entrainment.

Jetting and Pouring Operations

The jetting operation discussed here is a discharging of a fluid through a nozzle, tube, or hose, into atmosphere or through atmosphere into another fluid. In this sense pouring may be considered as a special kind of jetting. These operations are very common in cosmetic processing as in the filling of a hand lotion or liquid shampoo into bottles, transferring of a batch of cream from a kettle to a storage tank through a rubber hose, or pouring of a drum of liquid into a batch in a process tank as illustrated in Fig. 4. These jetting or pouring operations can often be the source of the troublesome air bubble entrainment.

In some cases the solution may be fairly simple. For example, the problem of air entrainment in transferring into a storage tank (Fig. 4, B) can be corrected by pumping the material through the inlet located near the tank bottom. A more careful pouring or use of a deflective plate to force the fluid to fall along the kettle wall can also minimize the problem of pouring in Fig. 4, C. However, sometimes the problem is much more complex and requires thorough analysis before an intelligent solution can be prescribed.

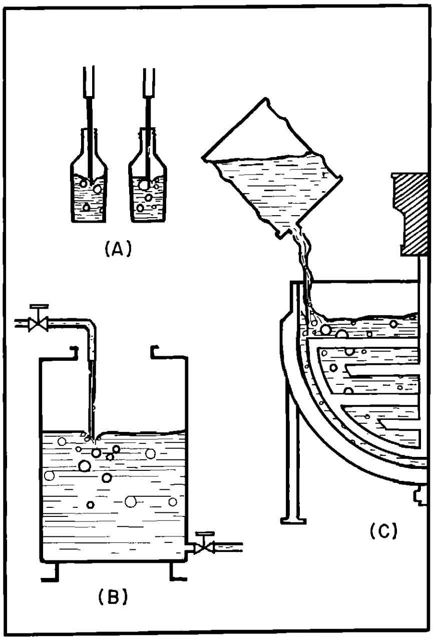

Figure 4. Air entrainment by jetting and pouring

To explain the basic mechanism of air entrainment by jets, Fig. 5 illustrates the discharge of a fluid through a small nozzle into a vessel containing the same fluid. The jet in (A) is perfectly smooth and there is no air in the receiving fluid. The jet in (B) is discharged at a much higher rate and the jet surface is irregular and the surrounding air is trapped by the jet surface and carried into the bulk. The jet in (A) is evidently in a laminar flow and (B) is in a turbulent flow.

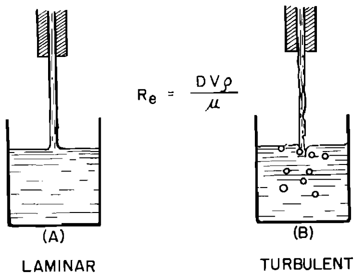

Figure 5. Laminar and turbulent jets

In fluid mechanics, transition from a laminar flow to turbulent flow is related to a dimensionless parameter, Reynolds number defined as follows:

$$\mathrm{Re} = \frac{DV\rho}{\mu}$$

where

Re = Reynolds number
D = diameter of the jet
V = linear velocity of the jet
ρ = density of the fluid
μ = viscosity of the fluid

When the nozzle is perfectly smooth and sufficiently long the transition from a laminar flow to turbulent flow normally occurs at a Reynolds number above 1000. However, if the nozzle is not smooth, straight, or too short, the jet may be in a turbulent flow at a lower Reynolds number. The turbulent jets can often be the cause of bubble problems in cosmetic processing.

For example, if a hot lipstick is poured from a kettle into a mold through a valve with a short exit pipe, the product may become aerated. This is because, in flowing through the valve and elbow, the velocity distribution in the pipe becomes irregular causing uneven stream surface which traps the surrounding air as the liquid plunges into the mold. By lengthening the exit with a smooth pipe, it is possible to smooth the jet stream and reduce the chance of air entrapment by turbulence. The length of the tube required to smooth the flow of a jet is dependent on the jet velocity as well as the physical properties of the fluid. Generally, the lower the viscosity, the longer the pipe length should be to achieve a smooth laminar flow.

A similar troublesome bubble entrainment problem can occur in the filling of cosmetic preparations using a straight circular nozzle. The modern filling machines generally operate at a very high speed and the discharged fluid can easily be in turbulent flow depending on the nozzle design and fluid properties. Often the lengthening of the filling nozzle alone will not solve the problem completely.

Reduction in the filling rate is a possible solution but it will affect the production rate. One possible way to minimize the turbulent entrainment is to use a filling nozzle with a larger diameter. For example, by doubling the jet diameter, D, while keeping the volumetric flow rate constant, the linear flow rate, *V*, will be reduced to one-fourth. Therefore, the Reynolds number for the larger jet will be only one-half of the smaller jet and this might bring the jet to the laminar flow region to avoid turbulent bubble entrainment.

The above calculation assumes no change in the fluid viscosity as the jet nozzle is enlarged. This will be true only with a Newtonian fluid, the viscosity of which is independent of the rate of shear. Many cosmetic preparations are non-Newtonian and particularly emulsion products are generally pseudoplastic or thixotropic, i.e., shear-thinning. Shear-thinning means that the viscosity decreases with increasing rate of shear. Because of the high linear velocity, the shear stress on the fluid while it flows through the nozzle is much greater in the smaller nozzle than in the larger nozzle. Therefore, if the fluid is shear-thinning, the viscosity of the fluid in the smaller jet may be much smaller than the viscosity of the same fluid flowing through a larger nozzle. Naturally, this will make the Reynolds number even greater in the smaller jet and increase the chance of aeration when the filling material is thixotropic. For these reasons, filling nozzles with very small discharging holes should be avoided. In addition to the bubble problem, discharging of a thixotropic emulsion through very small openings can sometimes cause a permanent viscosity breakdown.

At times, the turbulent bubble entrainment problem can be solved by varying the filling temperature. Many cosmetic creams are filled at an elevated temperature for practical or aesthetic reasons. Since the viscosity of an emulsion is usually low at a high temperature, reduction of filling temperature may increase the fluid viscosity and hence reduce the Reynolds number at which the product is filled.

The mechanism of bubble formation by jet discussed above involves turbulent entrainment. However, depending on the physical properties of the fluid, the discharging rate, and the geometry of the nozzle, air can be entrained even when the jet is perfectly smooth and in laminar flow (4). The author investigated the bubble entrainment by such laminar jets using high-speed photography and, as illustrated in Fig. 6, this is due to the formation of a very thin film of gas which breaks away to form air bubbles in the product. Figure 7 is a photograph taken by the author showing formation and breakup of such a cylindrical air film.

This type of bubble entrainment by a laminar jet is quite common in the filling of viscous cosmetic preparations. The author has ob-

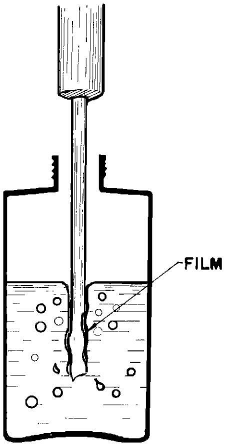

Figure 6. Laminar bubble entrainment

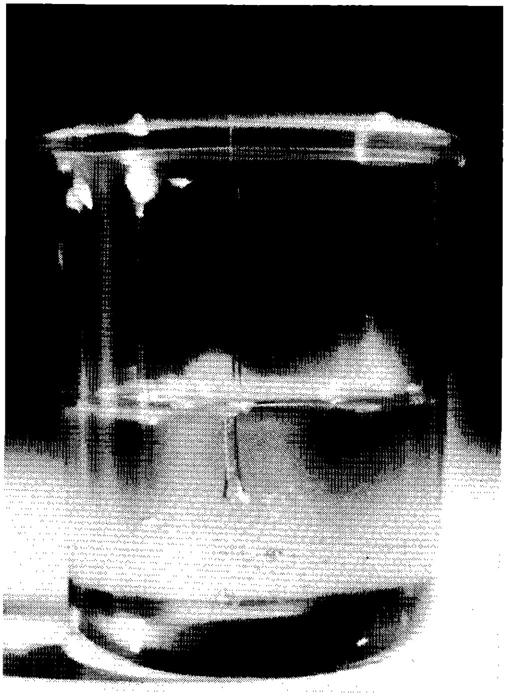

Figure 7. Gas film formation

served this type of entrainment from a perfectly smooth, viscous jet at a Reynolds number as low as 10. Furthermore, a study on such laminar jets indicated that bubble entrainment could occur only when the linear velocity of the jet exceeded a critical value termed "minimum entrainment velocity." By keeping the jet velocity below this value, laminar jet entrainment by the film breakup mechanism can be eliminated. The minimum entrainment velocity is a function of the jet diameter as well as the physical properties of the jetting fluid. For Newtonian jets having a uniform velocity profile, the following correlation was found by the author (5):

$$\mathrm{We} = 10\ \mathrm{Re}^{0.74}$$

or

$$\left[\frac{DVe^2\rho}{\gamma}\right] = 10\left[\frac{DVe\rho}{\mu}\right]^{0.74}$$

where

We = Weber number
Re = Reynolds number
D = jet diameter at the point where jet meets the receiving fluid
Ve = minimum entrainment velocity
ρ = density of the liquid
γ = surface tension of the liquid
μ = viscosity of the liquid

By solving the equation for minimum entrainment velocity, the following equation is obtained:

$$V_e = 6.22\,\frac{\gamma^{0.794}}{D^{0.206}\,\rho^{0.206}\,\mu^{0.587}}$$

From this equation, it can be seen that a fluid with low surface tension and high viscosity will have a low minimum entrainment velocity and will be likely to trap air by this mechanism. A solution to this problem is then to use a nozzle and filling rate such that the linear jet velocity is always below the minimum entrainment velocity.

In some instances, a reduction of jet velocity may not be possible or desirable and it may be necessary to solve the problem by redesigning the filling nozzle. Ideally, the filling nozzle should be submerged into the receiving fluid at all times to avoid air entrainment; however, this is not always possible in modern filling machines. Some filling nozzles are designed so that the jets do not hit the receiving fluid vertically, but rather they are to flow along the wall of the bottle being filled (Fig. 8). Among those illustrated, (B) and (C) are usually the better types from the aeration viewpoint. The type (D) with multiple pinhole orifices can sometimes introduce more air than a vertical nozzle because of the high jetting velocity.

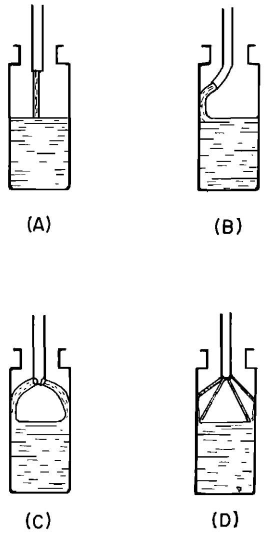

Figure 8. Various types of filling nozzles

(A) Vertical nozzle (B) J-shaped nozzle
(C) Double-orifice nozzle (D) Pinhole nozzle

Incorporation of Powdered Material

In cosmetic processing, as in the preparation of a liquid eyeliner or fluid make-up, it is often necessary to add powdered materials such as pigments or zinc stearate into a liquid. During such addition, the powders can carry into the liquid bulk an appreciable amount of air which may remain in the product.

This is a very common problem but not always easy to solve. Often a slow and uniform addition or a sprinkling of the powders rather dumping them into the batch will ease the problem. If the batch undergoes viscosity changes during the manufacturing process, the powdered material should be added when the batch is at its lowest viscosity (e.g., while the batch is hot) so that any entrained air bubbles can be freed.

If a very large quantity of powdered material must be incorporated in a batch and if the resulting mixture has a very high consistency, the use of a vacuum mixing kettle may be necessary. For example, a toothpaste is most commonly made in a vacuum mixing kettle or it must be passed through vacuum deaerating equipment afterwards to remove the entrapped air bubbles. A molten lipstick bulk is sometimes processed in an evacuated kettle to free entrapped air bubbles. The use of such a vacuum kettle for a lipstick would be unnecessary if the formula can be revised to lower the yield value of the bulk at its molten state. In some applications, the presence of air is undesirable only because it contains oxygen. In such instances, nitrogen or other inert gases can be used to purge the air from the enclosed system.

One possible way of mixing a batch of a fluid without air entrainment is to recirculate the fluid with a pump. An in-line mixer or homogenizer can be added in the line to achieve effective mixing or dispersing. To be effective, however, care must be taken to make sure that the circulation is not localized. Such a system is quite ideal for many applications but the consistency of the batch cannot be too high.

Internal Generation of Bubbles

All of the mechanisms discussed so far involve entrainment of the external air, i.e., the air which was not originally present in the liquid bulk. However, in addition to these mechanisms, it is also possible to form bubbles through internal generation of gases.

Chemical Reaction

As in a fermentation process, many gases can be generated through chemical reactions. If a viscous product undergoes a chemical reaction during the manufacturing operation or shelf life resulting in a liberation of a gas, the product can obviously contain gas bubbles. Although there are many chemical reactions which can lead to the liberation of gases, most cosmetic ingredients are relatively inert and this type of generation is relatively uncommon.

Physical Change

In the absence of a chemical reaction, gas bubbles can sometimes form as a result of a physical change. For example, if the change in environment is such as to produce a decrease in the solubility of the dissolved air in a product, the excess air can be liberated as air bubbles.

Such a solubility decrease can occur not only as a result of temperature increase or pressure decrease, but can also occur in the mixing of two or more liquids. If the mixing results in a decrease of the solubility of the dissolved gas, a liberation of the gas will take place until a new equilibrium is established.

According to the experiments conducted by the author, this mechanism is believed to be responsible for air bubble formation in the preparation of hydroalcoholic Carbopol®* gels (6). Mixing of ethanol and water, both saturated with air, will result in a decrease of the air solubility and liberation of air bubbles. The liberated air can escape from an ethanol-water solution readily. However, if an alcohol dispersion of a Carbopol resin is neutralized with an aqueous solution of triethanolamine, the liberated free air bubbles may be trapped in the newly formed gel because of the high viscosity and yield value of the Carbopol gel. Thus, no matter how much care is taken to avoid externally entrained air, it is still possible to form air bubbles by this mechanism.

To obtain a bubble-free gel, one can disperse Carbopol in a preblend of alcohol and water and neutralize the dispersion only after all the excess bubbles are freed. Since unneutralized Carbopol dispersions have very low viscosities, air liberated before neutralization can readily escape from the system. To hasten the bubble liberating process, the unneutralized dispersion should be agitated with a mixer. A more rapid way to remove the excess bubbles is to pass the unneutralized dispersion through an ultrasonic machine. By a cavitation process, the excess air can be removed quickly and the gel obtained by neutralizing such a dispersion can be completely bubble-free, provided that no external air is entrained during the neutralization process.

Finally, another possible mechanism of bubble formation as a result of a physical change is due to a density change. As some melted waxes are solidified, a density increase may occur and result in a product shrinkage. Sometimes, the wax mixture may harden before the full shrinkage takes place and result in a gradual formation of bubbles. An example would be the solidification of paraffin-based hair pomades. In some formulations, the full shrinkage may take several days resulting in the pulling away of the product from the sides of the jar or formation of many bubbles. Sometimes a gradual cooling rather than a rapid cooling

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* Carboxy vinyl resins, B. F. Goodrich Chemical Co., Cleveland, Ohio.

will minimize the problem but frequently a reformulation will be required to solve the problem completely.

CONCLUSIONS

Control of gas bubbles in cosmetic preparations is a difficult but extremely important problem in making stable and aesthetically pleasing products. Although there is deaeration equipment* designed to remove gas bubbles from the products, it is generally best to prevent the gas from getting into the products in the first place. As has been shown, once the mechanisms and sources of bubble formation are correctly identified, it is generally possible to prescribe solutions which will prevent or minimize the problem.

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* Examples, Versator® from Cornell Machine Co., Springfield, N. J., or Sontrifuge® from Teknika Inc., Dayton, Ohio.