

PRACTICAL ASPECTS OF EMULSION FORMULATION*

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IT IS THE PURPOSE of this paper to indicate briefly a practical approach to the subject of cosmetic emulsion technology.

In general, emulsions may be said to consist of a dispersion of exceedingly small droplets of a liquid in another equally immiscible liquid. The term oil-in-water emulsion is, therefore, applied to those emulsions in which oil is dispersed in an aqueous medium. Conversely, where water is the dispersed liquid, the resulting suspension is classified as a water-in-oil emulsion. Most cosmetic emulsions fall into the former category; they include vanishing creams, cleansing creams, cold creams, skin and hand lotions, deodorant creams, etc. The so-called tissue, or nourishing, creams are, as a rule, however, water-in-oil emulsions. Emulsions may further be classified into the liquid or solid type. The solid emulsions are formed by oils or fats that are liquid when heated but which solidify when cooled to room temperature.

Another means of differentiating

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emulsions other than by phase classification or consistency is in terms of function. We use the terms "non-vehicle" and "vehicle." Perhaps the simplest way to define these types is by illustration. Although the terminology is admittedly somewhat cumbersome, it is believed that the meaning will become clear. The vehicle type emulsion may be described as an emulsion which in itself does not exert a cosmetic effect, but acts as a carrier for the cosmetic agent. As an example, we may cite suntan, deodorant, and make-up creams. In these preparations, the emulsion is merely used as a vehicle for the astringent in the deodorant cream, the ultra-violet absorbent in the suntan cream, or the pigments in the make-up preparation. The non-vehicle type may be characterized as an emulsion whose basic ingredients—fats, oils, water, etc.—give the desired cosmetic effect. Among the non-vehicle preparations are cold or cleansing creams, hand lotions, night creams, etc. For example, in a cold cream the evaporation of the water phase on the skin surface

gives the characteristic cooling effect. The ingredients of the oil phase afford the desired cleansing, lubricating, and emollient effects. The non-vehicle type emulsion is comparatively easy to formulate and does not present the problems characteristic of the vehicle type.

placed in a jacketed vessel and heated and stirred to around 75-80°C., until melted. They are then stirred into another similar vessel containing a solution of the aqueous ingredients, which have been heated in a like manner, usually at a temperature that is about 5° higher.

Figure 1.—Non-Vehicle Type Creams

	Water	Alkali (Borax Caustic Amines, Etc.)	Oil (Min. or Veg.)	Beeswax	Stearic Acid	Cetyl Alcohol	Lanolin	Absorption Base	Gum	Glyceryl or Glycol Stearate	Humectant (Glycerin, Glycyl, Etc.)	Petrolatum
Cold or Cleansing Creams												
A	29	1	55	15								
B	35	1	50	10		1		1				2
C	55		20					2		15	3	5
Vanishing Creams												
A	65	2			25						8	
B	67	2			20	1	1			3	5	1
C	55	1	5			2		10		25	2	
Tissue or Night Creams												
A	40		25	5		3	10	10				7
B	40		27	5		3		25				
C	55	1	5			2		10		25	2	
Cream Lotions												
A	88	1	1		3	1			1		5	
B	90	1			2		1		1		5	
C	94				1			1		1.5	2.5	

In Fig. 1 are listed a dozen ingredients which are commonly used in the non-vehicle creams. It will be noted that these formulations have a good deal in common, not only as far as ingredients are concerned, but also in the methods of manufacture. With the exception of tissue or night creams, which are emulsions of the oil-in-water type and are handled somewhat as follows. The fatty materials are

Stirring is continued, rather rapidly, until the temperature drops to around 30-40°. Solid creams may be perfumed and packaged at this temperature. Liquid creams, on the other hand, are stirred until completely cooled.

The general procedure for preparing water-in-oil emulsions is somewhat as follows. The fatty materials are stirred and heated together in a jacketed vessel until melted,

and the temperature then regulated to around 45–55°C. The aqueous ingredients are placed in solution and heated to around 55–60°C., or in any event, 5° more than the oil phase. The aqueous solution is then slowly stirred into the oil phase. To avoid phase inversion, each succeeding portion is added only after the previous portion has been emulsified. The resulting emulsion is stirred with relatively slow agitation until completely cooled. It is widespread custom to homogenize water-in-oil emulsions by running them through a colloid mill or similar device for reducing particle size. This procedure is also used, in some instances, for oil-in-water emulsions, but it is not the general practice.

It is seen that emulsions of this category are not too difficult to formulate and that some general rules may be laid down as a guide. The following, although admitting of exceptions, have served the writer for a number of years:

1. The volume (or weight) of the disperse phase should be greater than that of the dispersed phase.
2. All of the ingredients of either phase must be miscible with the other ingredients of that phase.
3. If the stability of an emulsion is dependent on homogenizing or similar specialized mechanical handling, it should be reformulated because it is basically unstable.
4. When other factors permit, it is preferable to formulate an

oil-in-water rather than a water-in-oil emulsion.

The vehicle-type preparations do not admit of the generalizations regarding formulation or manufacturing procedure noted for the non-vehicle types, as the emulsifiers used are more complex and highly specialized. In addition, the additive which gives the desired effect is a stranger to the emulsion scheme and the question of compatibility therefore arises. The task of incorporating the additive in the emulsion is the essence of successful formulation in vehicle-type preparations of this category. We will take the astringent deodorant cream as an example of this type of preparation, and of the difficulties to be encountered. The function of an astringent deodorant cream is to act as a vehicle for the astringent material—usually an aluminum salt. The aluminum salt is said to react with the skin protein and form an aluminum albuminate, the resulting coagulant closing the openings on the skin surface and acting as a bar to epidermal excretions at the site of application. The vanishing cream type of deodorant has gained in popularity and seems to be the most acceptable from the consumer's point of view.

Even at this time, cosmetic formulators new to the problem ask if it is possible to simply add the required amount of an aluminum salt to a standard vanishing cream. Of course, when the ingredients of this type of cream are examined we know

that this is not possible. A concentrated solution of highly acidic salt, such as aluminum sulfate, will break the conventional type of vanishing cream formulated with potassium, or sodium stearates or other saponaceous emulsifiers. Nor will the various glycol or glycerol esters of stearic or other fatty acid do the trick, inasmuch as their emulsifying properties are dependent upon the presence of soap. However, acid stabilized emulsifiers have been developed, some specifically for this particular application. These may be broadly classified into three main groupings: the sulfated, the amide, and the non-ionic types. In some cases, these emulsifiers are used in conjunction with glycerol or glycol stearates, etc., or even in combination with each other. Representative astringent cream formulas are shown in Fig. 2. With these highly

Figure 2.—Typical Astringent Deodorant Creams

	1	2	3
Emulsifier	18*	5†	11‡
Stearic Acid	..	15	..
Cetyl Alcohol	1.5	3	6
Petrolatum	1	3	..
Glycol Stearate	8
Mineral Oil	2.5
Carbamide	5
Humectant	3	3	..
Water	53	52	59
Aluminum Salt	15	18	15
Titanium Oxide	1	1	1

* Sulfated. † Amide. ‡ Non-ionic.

specialized emulsifiers it is possible to formulate stable creams containing sufficient quantities of aluminum or other astringent salts to adequately inhibit the flow of perspiration.

However, there are other aspects of the problem of deodorant cream formulation. All of the commonly used astringent salts, to a greater or lesser extent, have a destructive effect upon fabric, particularly cotton; so that when the user's garment is laundered it is often found that the fabric is completely destroyed near the site of application. It is, therefore, desirable to have a deodorant cream formula contain an inhibitor, which will lessen the tendency of the astringent material to attack fabric. Carbamide, or urea as it is commonly known, which has lately been the subject of some prolonged patent litigation, is widely used as an inhibitor. There are other materials which exert a similar effect.

Some of the more complex amides, for instance, do a good job in very much lower percentages than carbamide. For this reason, it has been suggested that the amide-type emulsifiers are particularly suitable for use in deodorant creams. In some instances, they are used in conjunction with emulsifiers belonging to the other two types as emulsion stabilizers as well as inhibitors. However, it should be noted that while these materials tend to inhibit the fabric-destroying properties of the aluminum salts, there is much evidence to indicate that they also inhibit the astringent properties almost proportionately. It is therefore necessary for the formulator to strike a balance between the inhibiting properties in relation to fabric destruction and

inhibiting factor as affecting the astringent properties of the aluminum salts. Thus, we see the principle of balance or compromise being applied to cosmetic formulation. It is an important principle and is widely applied.

Many cosmetic preparations are classic examples of the combination of opposing forces. The measure of the skill and "know how" of a cosmetic technician is often his ability to achieve this balance. We can carry this concept a bit further in the same type of preparation. The higher the percentage of active ingredient, i.e., astringent material, a deodorant cream contains, the greater its astringent properties, and the more effective will it be as a deodorant preparation. However, we have already noted that we cannot employ any desired percentage of aluminum salt: first, because of emulsion stability; second, because of its effect upon fabrics. A third factor to be considered is the limit of solubility of the aluminum salts. Aluminum sulfate, for example, is quite soluble in water; but it is insoluble in most of the other commonly used ingredients. Since it is necessary for these other ingredients, emulsifiers, etc., to be present in order to form an emulsion, we have available only about 60-65 percent of water, and in many instances the solubility of the aluminum salts is decreased by the presence of the other ingredients. In any event, we end up with a preparation containing a fairly concentrated solution of aluminum salt:

in many instances, a saturated solution. Deodorant creams must last for some time, both on the shelves where they are sold and in the hands of the ultimate consumer. During its shelf life and while it is in use, there is bound to be a certain amount of evaporation of the water content from the surface of the cream, resulting in crystallization of the aluminum or other astringent salt. This development makes the product objectionable in appearance and unsuitable for use.

It has been demonstrated that it is necessary to have a sufficient amount of active ingredient (astringent) to exercise the primary function of a deodorant preparation, i.e., inhibit the flow of perspiration. The greater the amount of astringent salt, the larger the percentage of emulsifier, stabilizer, or inhibitor required. But, the greater the amount required of the last three mentioned ingredients, the smaller the amount of water available as a solvent for the astringent salt. Hence, the need for balance or compromise.

So far, only the formulation difficulties inherent in a typical vehicle emulsion, such as a deodorant cream, have been dealt with. Using the same example, the astringent deodorant cream, it may be of interest to note a few of the manufacturing problems incurred. Employing again, as we must, a general procedure, we note that the fats and oils are often heated together at around 75-80°C., and then stirred until an emulsion is formed. This

emulsion is stirred continuously until cooled, when it resembles a rather stiff vanishing cream. The aluminum salts and other inorganic materials are then added very slowly until dissolved or dispersed. This procedure is critical and somewhat tricky, inasmuch as the addition of the astringent salt results in a rise in temperature, which must be controlled by the introduction of water in the cooling jacket of the mixing vessel or other suitable means. This, together with other factors, might result in an immediate breaking of the emulsion and the spoilage of the particular batch. After the salts are added, the cream is ready for the introduction of perfume, which is added rather slowly in order to insure complete mixing. Titanium oxide is usually included in formulations of this type. For this, and other reasons, it is often considered advisable to mill the entire cream in order to insure distribution of the inorganic material. Here again a note of caution is advised; the peculiar nature of the materials tends to dull the surface of the milling machinery, even after only one run, particularly when colloid mills are employed,

where clearances are rather close.

In view of the foregoing, it would seem that hardly any of the broad generalizations which were applied to the preparation of emulsions of the non-vehicle type can be applied to those of the vehicle variety. Each product in this category has to be considered as an individual problem. The trend, if anything, is for preparations of this sort to become more and more complex. Fortunately, there is an ever-growing number of materials available to which the cosmetic technician may look as a means for solving his problems. These materials are not only increasing in number and variety; they have become more uniform and standardized than they were only a few years ago. By becoming acquainted with the properties and limitations of the latest developments in the field of synthetic emulsifiers and other surface-active materials, the cosmetic chemist will find that he has available a vast potential storehouse of highly specialized tools to assist him in meeting the varied and intricate problems presented in the equally highly specialized realm of cosmetic emulsion technology.