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Low-energy emulsification—I—principles and applications

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Synopsis

The amount of energy normally expended in commercial processing of a cosmetic emulsion is far greater than the amount theoretically required. Whether the emulsion is made by a batch process or semicontinuous process, thermal energy is first supplied to heat the ingredients and mechanical energy is then provided for mixing and emulsification. Additional mechanical energy is expended to cool the product and the heat removed is generally discarded.

A considerable saving in energy is made possible by a more effective usage of thermal and mechanical energy in emulsification. A substantial saving of thermal energy can be achieved by a careful determination of emulsification temperature and by a selective heating of the ingredients. The method discussed here basically involves making an emulsion concentrate which is later diluted with the remainder of the external phase at room temperature.

In addition to conserving energy, the proposed LOW-ENERGY EMULSIFICATION technique also offers a great advantage in reducing the processing time and equipment cost. In some instances, the energy cost for processing an emulsified lotion can be reduced by 50 per cent while the production efficiency can be increased by 100 per cent.

INTRODUCTION

The recent natural gas shortage in the United States has again clearly demonstrated the importance of energy conservation. One area of cosmetic processing that has not been critically examined in terms of energy requirement is emulsion processing. Compared to the energy actually required, a considerable amount of energy is wasted in a typical plant operation of emulsion manufacturing.

In 1965, the author proposed a technique of emulsification referred to as "semicold processing" that was designed to allow manufacturing of emulsion products with a partial heating of the raw materials (1). Since then, the author has tested this technique on numerous emulsified and nonemulsified products in production scale with favorable results. The method allows not only a conservation of thermal and mechanical energies, but also a substantial increase in manufacturing efficiency and a reduction in operating expenses without any compromise in the product quality. In some cases, it is also possible to reduce the capital expenditure on process equipment when planning an expansion of production capacity. The main purpose of this paper is to outline the basic

principle and to point out possible areas of application in practical processing of emulsion products.

ENERGY CONSUMPTION IN EMULSION PROCESSING

In the conventional processing of cosmetic emulsions, the oil- and water-soluble ingredients are usually heated in two separate kettles as illustrated in Figure 1. There are two forms of energy input: thermal energy (TE_1 , TE_2) for heating and mechanical energy (ME_1 , ME_2) for mixing and homogenizing (ME_3). It can be shown by energy balance that, in a typical plant production, only a small fraction of the energy input is utilized in actual emulsification, *i.e.*, to break up liquids into small droplets.

If city water is used for cooling the batch, the thermal energy removed during the cooling stage is generally discarded along with the water. If chilled water is used and recycled, additional energy is required by the compressor in the refrigerated system to remove and discard the heat. The majority of mechanical energy supplied dissipates as friction and turns into heat and noise.

An estimate of the total energy utilized *vs.* the energy wasted in a conventional processing of emulsions can be made by calculating the theoretical energy requirement. For illustration, it is assumed that one is making a 1,000 kg batch of a certain O/W emulsion consisting of 25 per cent mineral oil, 5 per cent surfactant and 70 per cent deionized water.

The theoretical energy requirement for emulsification is, of course, dependent on the effectiveness of the surfactant as well as the droplet size distribution of the final emul-

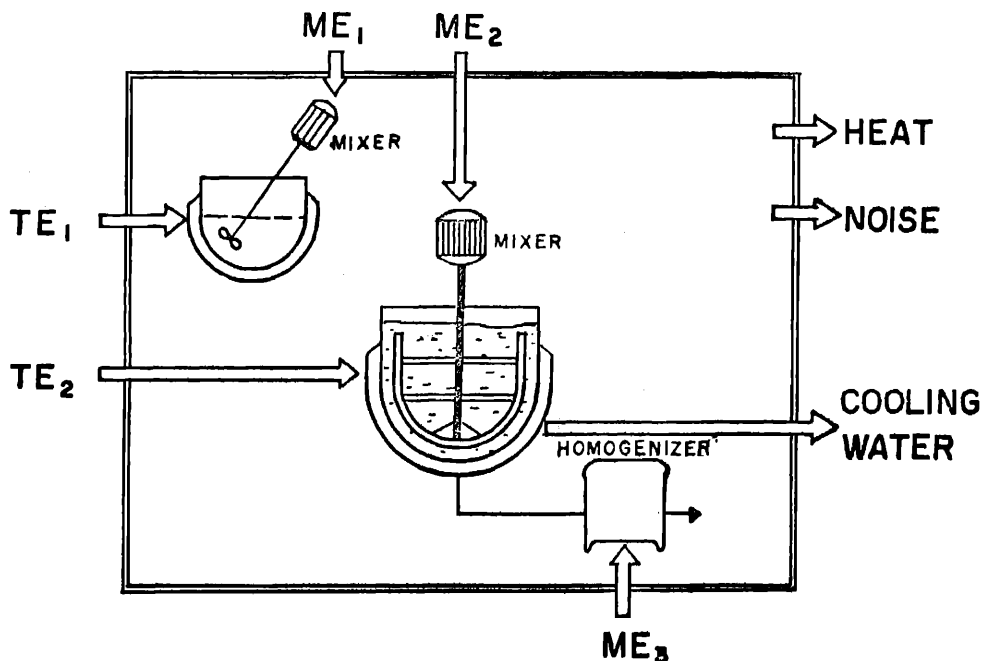


Figure 1. Energy input in emulsion manufacturing

sion. The theoretical amount of work required to break the liquids into droplets can be calculated from the interfacial tension and the change in surface area given by the following equation:

$$W = \gamma \Delta S$$

where W = work done
 γ = interfacial tension
 ΔS = change in interfacial area

For illustration, it is assumed that the interfacial tension is 2 dyn/cm and the final emulsion droplets are spherical, having a uniform diameter of 1μ . Taking the specific gravity of mineral oil as 0.85, the minimum energy requirement calculated from the above equation is 0.84 Kcal per 1,000 kg of the O/W emulsion. This value represents, of course, a theoretical minimum assuming no internal friction. The actual requirement is expected to be much greater.

The amount of energy consumed in making such an emulsion in a plant scale will be also dependent on emulsification method, emulsification temperature, etc. For this calculation, the following assumptions are made:

Mixer power: one horsepower for each of the two mixers

Mixing time: 90 min per batch for both mixers

Homogenizer power: 5 hp

Homogenizing time: 20 min

Room temperature: 20°C

Emulsification temperature: 80°C

Heat capacity: 1 cal/g, °C for all raw materials and finished emulsion

Batch size: 1,000 kg

The results of the calculation based on the above parameters are given in Table I.

The combined mechanical energy input is close to 3,000 Kcal and the thermal energy consumption is 60,000 Kcal or about 95 per cent of the total. Clearly, compared to the theoretical requirement, a typical plant processing of an emulsion consumes a far greater amount of energy. It should be evident that if one can devise a way to make the emulsion cold, 95 per cent of the energy consumption in this example can be immediately saved.

Hot emulsification is wasteful not only from the energy viewpoint but, even more importantly, from the consideration of production time and efficiency. The time required

Table I
Energy Input

Energy Source	Energy Consumption (Kcal)	Percentage Total
(a) Two Mixers	1,920	—
(b) Homogenizer	1,067	—
Total Mechanical Energy	2,987	5
Total Thermal Energy	60,000	95
Total Energy Input	62,987	100

Table II
Processing Time

Operation	Time (minutes)
Preparation	50
Heating	30
Emulsification	30
Cooling/Mixing	60
Homogenizing/Pumping	30
Clean-up	40
Total	240 = 4 hr

for each operation in a typical batch processing of the emulsion cited in the example is given in Table 2.

The total processing time in this example is 4 hr, including clean-up and preparation. It is to be noted that 30 min is spent on introducing the thermal energy and 60 min is spent on removing it at a latter stage. Therefore, a total of 90 min of valuable processing time can be saved by adopting the use of a cold method.

LOW-ENERGY EMULSIFICATION

Even though the advantages of a cold process are quite obvious, the popular use of waxy raw materials in cosmetic emulsions presents a serious problem in practical processing. Even with the use of a homogenizer, stearic acid and most waxy substances would not emulsify properly in cold water. Hence, a completely cold emulsification is feasible only with limited emulsions consisting solely of liquids or liquid-soluble ingredients.

However, in most cosmetic emulsions the total amount of waxy material is generally below 20 per cent and sometimes only 3 or 4 per cent; a question can thus be raised as to the necessity of heating the entire 100 per cent of the ingredients in order to obtain a good emulsion. The basis of low-energy emulsification is to combine the advantage of cold emulsification with the practical necessity of hot emulsification by selectively applying heat to a part of the ingredients.

Figure 2 illustrates the common batch processing of a cosmetic emulsion. If the final emulsion is an O/W type, the internal phase generally consists of oils and waxes. The external phase is made up of water and water-soluble components. In this figure, h and H represent the heat supplied to the internal and external phases respectively. After emulsification, the batch is generally cooled to room temperature by either circulating cooling water in the kettle jacket or passing the warm emulsion through a heat exchanger. Neglecting the small amount of heat lost to the atmosphere, the frictional heat and the heat of mixing, the heat that must be removed from the emulsion is $h + H$.

In a low-energy method illustrated in Figure 3, instead of heating the entire external phase, only half of the water phase is first heated to make a concentrated emulsion; the remaining half is then added at room temperature. The energy supplied to the external phase is now half of the original value and the heat which must be removed later is only $h + 0.5 H$.

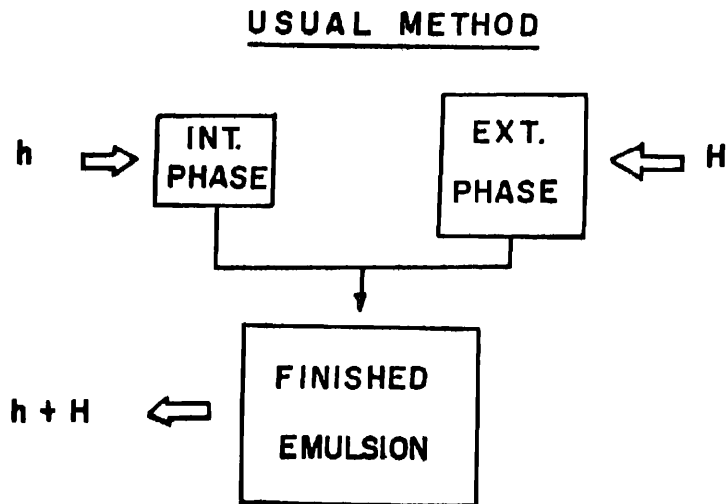


Figure 2. Conventional emulsion processing

In this example, only 50 per cent of the external phase was withheld for cold dilution, but there is no reason why one cannot withhold 70 per cent or more to achieve an even greater energy conservation. In some instances, a portion of the oil phase can be withheld and added cold later if it consists of mostly liquid materials such as mineral oil. One limitation is that one cannot withhold too much so as to make the processing of the concentrate difficult or the subsequent dilution impossible.

There are many different ways to apply the low-energy emulsification technique. One method (Figure 4) uses two kettles and an automatic metering valve. The entire oil phase is heated in one of the kettles and the first portion of water and water-soluble ingredients is heated in the other kettle. Depending upon the desirability of a phase

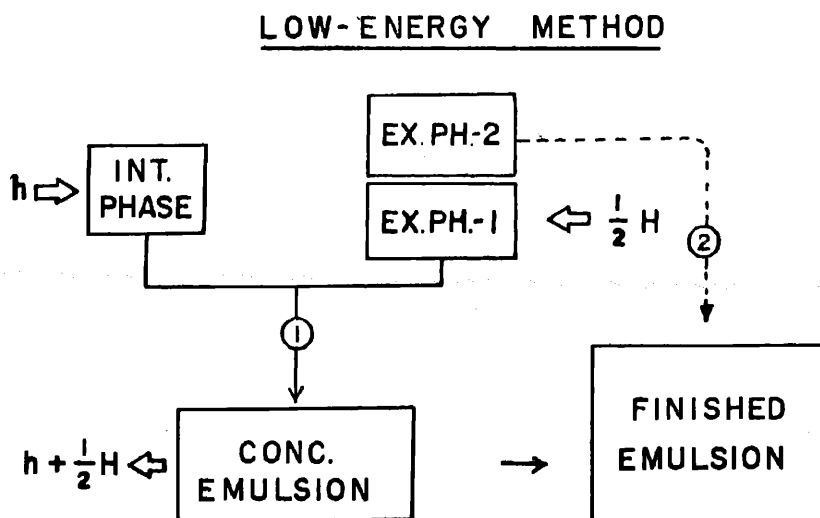


Figure 3. Low-energy emulsion processing

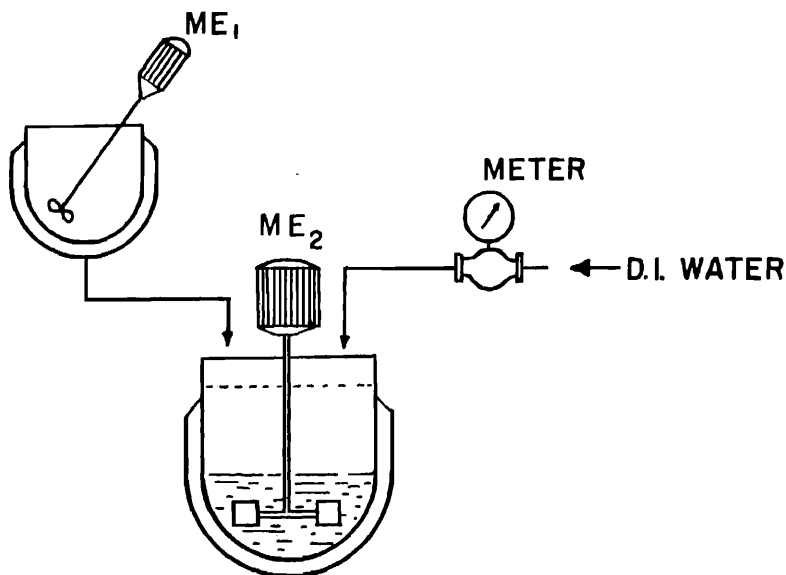


Figure 4. Two-kettle low-energy emulsion processing

inversion, the oil phase can be added to the aqueous phase or *vice versa* to form the emulsion concentrate. While the concentrate is being cooled, the remainder of the cold deionized water can be metered in to complete emulsification.

The saving of thermal energy is quite obvious in such a technique. The mechanical energy, ME_1 , supplied to the upper kettle remains unchanged, although ME_2 , supplied to the lower processing kettle, may be somewhat different. In the low-energy method, the initial emulsion is more concentrated and generally more viscous. On the other hand, there is less material in the kettle in the first stage so that the total mechanical energy consumption will not be much different. If anything, the over-all mechanical energy consumption will be less for the new process since the cooling time is shortened considerably by the addition of the cold water.

The low-energy technique illustrated here involves a two-step operation. It may appear, at first, that it would take longer to process a batch in comparison to the conventional, one-step procedure. According to the author's experience, the low-energy method actually requires much less processing time and in many situations this benefit may be even more desirable than the conservation of energy. The reason is that the most time-consuming part of making a commercial emulsion is often the cooling of the batch. Particularly if the product is very viscous or if the cooling water is not very cold, the time required may be very long. In the new process, the addition of less energy at the beginning means removal of less heat during the cooling period, resulting in a substantial shortening of the heating and cooling time. Moreover, the dilution step can be carried out during the cooling stage by simply metering the cold, deionized water so that there is no extra time consumed in the second stage.

APPLICATION OF THE LOW-ENERGY METHOD IN PRODUCTION EXPANSION

In addition to saving energy and processing time, the low-energy technique described here may be applied in some instances to save capital expenditure in planning a produc-

tion expansion. Since only a portion of the batch is heated in the low-energy method, it is possible to use a smaller kettle for processing or, in some instances, use the existing kettle to make larger batches to save capital expenditure on expensive, jacketed kettles.

The economy of such a technique should become apparent by considering the following examples. Consider a company which now has a 200-gallon tank to make a certain, low-solids, O/W moisturizing lotion. For illustration, it is assumed that the time required for each compounding operation is the same as the time presented in Table II. Suppose that it is now desired to increase the production capacity of this product by 2.5 times to meet the increased sales demand. One proposal calls for purchasing a new 500-gallon process tank and increase the batch size by 2.5 times. However, a careful engineering study would soon reveal that the purchase of a new kettle alone would not guarantee a proportional increase in the actual production if the conventional emulsification technique is used.

The reason should become apparent if the time required for each operation (Table II) is carefully examined. The preparative time includes the time required for weighing the ingredients and for metering or weighing the deionized water in the aqueous phase. Since the batch size is now 2.5 times greater, it will take longer to catch the deionized water. The heating and cooling times will be much greater, not only because more material is involved but also because of the fact that the larger the kettle, the smaller will be the heat transfer surface per unit volume of the material. The time spent on homogenizing and pumping will increase proportionally to the batch size.

If it now takes 4 hr to complete a 200-gallon batch, it will likely take six or more hours to process a 500-gallon batch. This means that only one batch can be produced in an 8-hr work day and one can expect only a 25 per cent increase in the actual production capacity.

Naturally, there are ways to speed up each compounding step to allow completion of the 500-gallon batch within the 4-hr limit, but this will require more equipment. For example, the time required to catch the deionized water can be shortened by installing a larger ion-exchange unit. The cooling time can be shortened by using a rotary, scraper heat exchanger. The homogenizing/pumping time can be shortened, but larger equipment will be needed. It is evident that a considerable capital expenditure will be required for the proposed production increase.

By far the more economical way of meeting the need would be to adopt a modified, low-energy technique (Figure 5). The idea presented here is to make an emulsion concentrate in the existing 200-gallon kettle and then dilute it in a 500-gallon storage tank equipped with a mixer.

The method is particularly ideal if the emulsion has a relatively small internal/external phase ratio. Since only 200 gallons of the material is heated, the heating and cooling times will not increase. The time needed for pumping and homogenizing will be the same. The only operation that may require extra time is the metering of 300 gallons of deionized water. However, a relatively inexpensive, automatic metering valve can be conveniently used to meter the water while the concentrate is being processed in the kettle so that no extra time will be required in this operation. It should not present any difficulty to complete two 500-gallon batches of this emulsion in an 8-hr period using such a technique. The only new equipment required for this process is a 500-gallon storage tank. A storage tank, however, is much less expensive than a jacketed, stain-

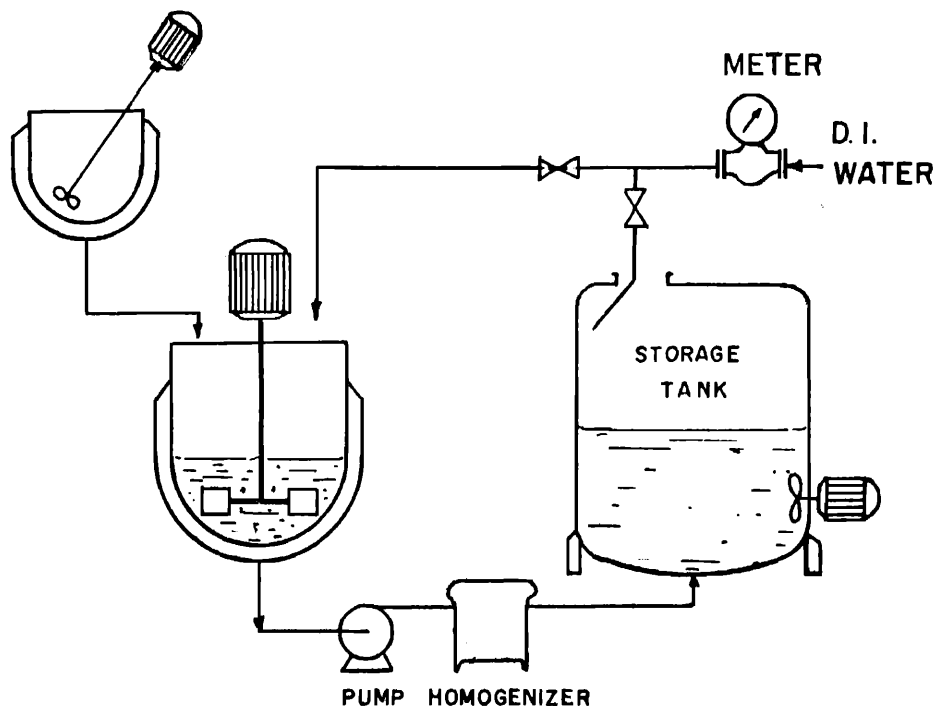


Figure 5. Modified low-energy emulsion processing

less steel kettle of the same capacity. Therefore, from both the production efficiency and equipment cost viewpoints, the low-energy method is far superior to the conventional method of emulsification.

LIMITATION OF LOW-ENERGY TECHNIQUE

There are, naturally, some limitations on the application of the proposed low-energy technique. One of the important points to be considered is that mixing becomes difficult if the viscosity of the concentrate is too high. There is therefore a limit as to how much of the external phase one can withhold. Fortunately, most emulsions, even fairly concentrated, are flowable at the elevated temperature at which emulsification is normally carried out.

The second limitation is that, upon dilution, a stable emulsion of desired properties must be obtainable. The desired properties may be the correct texture, opacity or certain rheological properties. This requirement is, of course, very important from the marketing viewpoint. However, since emulsion is an extremely complex system with so many physical factors which can affect its properties, it is not easy to define the conditions which will satisfy this requirement.

The only reliable way to determine if the method works satisfactorily would be to conduct carefully controlled pilot batch experiments. It is important to remember that there is no set way to carry out low-energy emulsification. There are many variations possible and with some imagination many advantages can be derived.

In some instances, for example, the presence of a thickener can make the concentrate very thick and cause difficulty in mixing. Sometimes such a problem can be solved by dispersing the thickener in the diluting water. If the viscosity of the thickener is pH-dependent, it may also be possible to avoid the problem by carefully controlling the pH of the concentrate. Phase inversion of the concentrate can cause a problem if the emulsion does not invert to the desired type upon dilution. Sometimes such a problem can be prevented by changing the order of phase combination or the surfactant location (3).

It should be cautioned that since cold deionized water is used in the second stage, this water must be sterilized to avoid microbial contamination.

CONCLUSION

Although the technique described here lends itself readily to the processing of low-solids, O/W emulsions, there is no reason why it would not apply to W/O emulsions or nonemulsified products. In fact, the method works very nicely on processing most shampoos and even some make-up preparations. The author has successfully tested a W/O system by withholding parts of both the oil and aqueous phases. However, such a technique becomes rather involved and requires experience to handle properly.

In using the technique, it is well to reexamine the emulsification temperature used. In many instances it is possible to lower the emulsification temperature without affecting the emulsion quality. In some emulsions stabilized with nonionic surfactants, it may be desirable to keep the emulsification temperature above the PIT (phase inversion temperature) in order to facilitate emulsification.

In some cases, considerable savings in mechanical energy used in mixing or homogenizing can be achieved by changing emulsification method. Elimination of energy-consuming high-shear equipment can be frequently accomplished by optimizing emulsification conditions. For example, without changing formulation, it is sometimes possible to significantly facilitate emulsification by controlling the emulsifier location or presolubilizing the oil phase (4). In some instances, one can obtain a finer emulsion using the low-energy technique than using a conventional method.

The author has conducted considerable basic work on variables affecting droplet size distribution and stability of emulsions prepared by this low-energy technique. A paper dealing with these aspects will be published in the future.

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