

Manufacture of Emulsions by Shock Cooling

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A "shock cooling" technique was used in the preparation of five emulsion systems. The shock cooling was achieved by atomizing a heated blend of the emulsion phases in a laboratory model spray dryer operating with chilled inlet air. The test preparations included an o/w hand lotion, an o/w baby lotion, a w/o cold cream formulation, and an o/w cosmetic lotion containing suspended pigments and hydrophilic ointment U.S.P. The physical stability of the shock cooled products was equivalent to or improved over that of control materials processed by conventional slow cooling techniques. The results of this study suggest that the principle of shock cooling has value in large scale manufacture of emulsions.

EMULSION SYSTEMS containing waxes, fats, and other low melting solids are widely used as dermatological vehicles and in the formulation of numerous cosmetic creams and lotions. The products generally are prepared by the fusion process. Briefly, this involves emulsifying the molten oil phase with the aqueous phase at elevated temperature (approximately 65 to 75°), then slowly cooling the product to room temperature. During the cooling step, the emulsion is agitated continuously to insure uniform rates of congealing throughout the batch (1-4).

Slight variations in the conditions of manufacture can alter final emulsion characteristics significantly (1, 5). The rate at which the hot emulsion is cooled has been reported to be particularly critical (2, 6). Boylan, DeKay, and Banker have shown, for example, that products with increased thixotropy and decreased stability result as the rate of cooling is increased (7). Strianse noted that emulsion viscosity increased with increasing rates of cooling (8); others have reported that the reverse effects can occur (5).

It has been generally accepted that rapid cooling promotes the crystallization and separation of the higher melting components of the emulsion. This in turn results in products which are "grainy" and unstable (9, 10). Consequently, slow congealing techniques, although time consuming, laborious, and inefficient, are commonly employed in practice.

A reanalysis of the congealing process and of the results obtained with nonaqueous systems

(11-14) suggested, however, that crystal growth and similar effects could not occur if the emulsions were subjected to instantaneous rates of cooling. The present study was undertaken, therefore, to evaluate the feasibility and potential applications of shock cooling techniques in the manufacture of emulsion systems.

EXPERIMENTAL

Materials.—Five emulsion products were studied in the present experiments. The test systems included hydrophilic ointment U.S.P., an o/w baby lotion, an o/w hand lotion, a o/w pigmented lotion (cosmetic make-up base), and a w/o cold cream formulation. The formulas for the latter four products are given in Table I.

All raw materials were U.S.P. or N.F. quality unless otherwise indicated.

Methods of Preparation.—Each emulsion was prepared by heating and melting the components of the oil phase together at approximately 65° and adding this phase to the preheated aqueous phase held at the same temperature.¹ The resulting emulsions were blended at this temperature for 5 minutes using a loop-type agitator rotating at low speed.

The hot emulsions were divided into two equal portions. The first portion was shock cooled. This was accomplished by spray congealing the emulsion in a Nerco-Niro laboratory model spray dryer² operating with the inlet air chilled to approximately 14°. To maintain the inlet air at this temperature, a supply of dry ice was placed at the mouth of the inlet duct and allowed to vaporize into the inlet air stream. The centrifugal wheel atomizer was run at full turbine air pressure (6 Kg. per cm.).² Liquid feed rates to the atomizer were maintained at 150-200 ml. per minute and resulted in outlet air temperatures of less than 20°. The products were collected by cleaning the walls of the spray dryer with a rubber spatula.

The remaining portion of each hot emulsion served as the control preparation and was congealed by the conventional procedures. This involved the slow

Received June 3, 1963, from the Department of Pharmaceutical Technology, Warner-Lambert Research Institute, Warner-Lambert Pharmaceutical Co., Morris Plains, N. J.

Accepted for publication July 16, 1963.

Presented to the Scientific Section, A.Ph.A., Miami Beach meeting, May 1963.

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¹ The order of addition was reversed for the baby lotion and cold cream.

² Nichols Engineering and Research, Inc., New York, N. Y.

TABLE I.—COMPOSITION OF TEST EMULSION SYSTEMS

Phase	Component	% by Wt.				
		Baby Lotion	Hand Lotion	Pigmented Lotion	Cold Cream	
Oil	Lanolin	1.00	
	Cetyl alcohol	2.00	1.25	0.50	...	
	Span 80 ^a	2.10	
	Tween 80 ^a	4.90	
	Liquid petrolatum, light	34.00	4.00	25.00	56.00	
	Silicone 200 fluid ^b	5.00	
	Glyceryl monostearate	...	0.50	0.50	...	
	Stearic acid	...	1.00	
	Atlas G-3694 ^a	...	0.50	
	Spermaceti	0.50	12.50	
	White wax	12.00	
	Aqueous	Methylparaben	0.20	0.15	0.20	...
		Emcol E 607-S ^c	...	1.00
Glycerin		...	1.00	
Propylparaben		0.05	...	
Sodium lauryl sulfate		0.63	...	
Titanium dioxide		12.00	...	
Cosmetic red oxide, A6205 ^d		0.50	...	
Sodium borate		0.50	
Water		q.s.	100.00	100.00	100.00	

^a Atlas Powder Co., Wilmington, Del. ^b 350 Centistoke viscosity, Dow Corning, Inc., Midland, Mich. ^c Emulsol Corp., Chicago, Ill. ^d Kohnstamm and Co., Inc., New York, N. Y.

cooling of the product to room temperature with continuous mixing applied by the loop-agitator. Approximately 1 hour was required to complete the preparation by this technique.

Evaluation.—Shock cooled products were compared with their controls immediately after preparation and periodically during a 2-month storage interval at 5, 37, 45°, and room temperature (27°). Samples were subjected also to freeze-thaw conditions for 1 week by cycling between -5° (16 hours) and 27° (8 hours). All products then were transferred to room temperature storage and observed during an additional 4-month interval.

Shock cooled materials and their controls were compared by microscopic, visual, and tactile evaluation. Overall physical characteristics, including phase separation, color, texture (graininess), and spreadability were observed routinely.

The rheological characteristics of each product when first prepared and after 6-month storage at room temperature were measured using an Epprecht viscometer.³

RESULTS AND DISCUSSION

Each of the five emulsion systems was prepared successfully by the shock cooling technique. No difficulties were experienced in congealing the emulsions in the spray dryer; the entire cooling operation was completed in less than 5 minutes.

Table II summarizes the characteristics of the shock cooled and control products. The shock cooled emulsions were smooth, free of graininess, lighter in color, and generally more uniform than the control materials.

Improved dispersion and a greater reduction in the particle size of the discontinuous phase was obtained by the shock cooling technique. This was particularly evident with the cold cream formulation. The majority of particles observed in the shock cooled product ranged between 2 and 7 μ in diameter. The emulsion prepared by the conventional slow cooling method showed particle sizes ranging from 10 to 40 μ . Since aliquots of the same

hot emulsion were used in preparing both products, the changes in particle size necessarily occurred during the cooling operations. In the spray congealing technique, the hot emulsion was subjected to atomization and dispersed into droplets with small particle size. This, in effect, represented a form of homogenization and accounts for the reduced particle sizes found in the spray congealed preparation. The particles observed in the control product (cold cream) were not uniformly spherical, thereby suggesting that some size enlargement (due to crystallization or agglomeration) may have occurred during the slow cooling process. There were no microscopic or macroscopic indications that abnormal amounts of air had been trapped in the spray congealed products. Therefore, the lighter color of the shock cooled products was attributed to the decreased particle size.

In two of the five test systems (the hand lotion and the cold cream), the spray congealed products were less viscous than their controls. The situation was reversed with the pigmented lotion and the baby lotion. For the latter formulations, shock cooling also resulted in (qualitative) changes in the level of thixotropy. Hydrophilic ointment showed similar flow characteristics when made by either process.

These results are inconsistent with considerations of particle size and presently are not fully understood, particularly because of the rheological studies reported by others for similar products (7, 15). The short holding time involved in the shock cooling process led to a reduction in evaporation losses (of water) from the emulsions; this may account, in part, for the observed differences in product rheology.

Products prepared by spray congealing generally were more stable than their control preparations. This was particularly evident in the case of the three lotion products.⁴ Except for the hand lotion (made by either process), all products were stable under freeze-thaw and 5°. Storage at 45° most clearly

³ Manufactured by J. W. Fecker Division, American Optical Co., Pittsburgh, Pa.

⁴ These preparations represented formula modifications of products with known and satisfactory stability. The emulsifier systems were altered deliberately to insure that the influences of shock cooling would be readily apparent.

TABLE II.—CHARACTERISTICS OF SHOCK COOLED AND CONTROL PREPARATIONS

Product	Cooling Procedure ^a	Particle Size, μ ^b	Color	Texture and Appearance ^c	Rheology ^d	Separation ^e		
						45°	37°	RT
Baby lotion	SC	2-5	Off-white	U	MV, NT	3	2/	0/
	C	5-10	Tan	NU	ST	3	3/	0/
Hand lotion	SC	2-5	White	U	LV, NT	1	1	1
	C	2-10	White, off-white	U	NT	2	2	2
Pigmented lotion	SC	2-10	Pink	U	MV, ST	2	1	0
	C	5-30	Dark pink	SA	NT	3	2	2
Cold cream	SC	2-7	White	U	LV, MT	1	0	0
	C	10-40	White, off-white	NU	MT	1	0	0
Hydrophilic ointment	SC	7-15	White	U	MT	1	0	0
	C	10-25	White	U	MT	1	0	0

^a SC = Shock cooled; C = control (cooled slowly). ^b Particle size range included approximately 75% of total particles counted. ^c U = Uniform and smooth; NU = nonuniform; SA = "streaky" application. ^d MV = More viscous than control; LV = less viscous than control; NT = negligible thixotropy; ST = slight thixotropy; MT = marked thixotropy. ^e Phase separation observed after 2 months at 45° and 37° and after 6 months at room temperature, respectively. 0 = No phase separation; 1 = trace indications of separation; 2 = moderate separation; 3 = severe separation. / Mold growth occurred at these conditions.

demonstrated the comparative improvements in stability obtained in the shock cooled products. The degree of phase separation observed after long term storage at 45°, 37°, and room temperature served as the final measure of relative stability. These observations are summarized in Table II.

The rate of cooling established in the spray congealing process was approximately 13° per 0.1 second.⁶ This rapidity of chilling precludes extensive crystal growth, and it is, therefore, not surprising that smooth and grain-free products were obtained. In fact, it appears that the slow cooling rates commonly used in preparing emulsion systems would promote rather than inhibit crystallization and the separation of components.⁶ By applying sufficient agitation to the system, however, nucleation rather than crystal growth can be induced. The required rates of agitation clearly would be governed by the congealing rates employed and vice versa. These considerations help explain the critical relationships which exist in emulsion manufacture between the rates of cooling and the intensities of agitation (1, 5).

The results of the present study showed that stable and pharmaceutically elegant emulsions can be prepared by shock cooling. The spray congealing procedure used to demonstrate the feasibility of this approach was fast, simple to control, and reproducible; nevertheless, the equipment requires modification for large scale manufacturing operations. The necessity for modification is primarily because the conventional spray dryer, as used in this study, is not designed for use with liquid and semi-solid (outlet) products. For example, it was difficult to clean the unit, and the accumulation of product on the chamber walls and in the outlet air duct work limited the amount of product that could be processed in a single run. The results suggest, however, that other spray congealing units (more appropriately designed for emulsion applications) or the use of other shock cooling techniques which minimize particle growth during the cooling step⁷ will be valuable in commercial applications.

$$^6 \text{ Approximately: } \frac{175 \text{ Gm. (feed)}}{\text{minute}} \times \frac{(65^\circ - 20^\circ)}{\text{Gm.}} \times \frac{1 \text{ minute}}{60 \text{ seconds}} = \frac{130^\circ}{\text{second}}$$

⁶ It is recognized that controlled degrees of crystallization are desired in some cosmetics in order to impart "pearlescent" and other characteristics.

⁷ Such as the use of chilled ointment mills operating at close clearance.

CONCLUSIONS

The results of this study demonstrate the feasibility of applying shock cooling techniques in the preparation of emulsified creams and lotions. The shock cooled products were prepared by spray congealing and compared favorably in all respects with the products made by conventional slow cooling procedures. The improved stability and texture characteristics and the absence of "graininess" in the spray congealed materials was noteworthy, particularly because of the generally accepted impression that rapid cooling is detrimental to emulsion systems. Although the spray congealing procedure used in the present experiments was simple to control, reproducible, and resulted in very high cooling rates, its usefulness for commercial applications is limited. These experiments do suggest, however, that other shock cooling procedures, more appropriately designed for large scale manufacturing operations, hold promise for future application.

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