Bubble Size Analysis of High Consistency Aerosol Foams and Its Relationship to Foam Rheology

Effects of Container Emptying, Propellent Type, and Time

By LARRY L. AUGSBURGER and RALPH F. SHANGRAW

A photographic technique for the analysis of bubble size in "aerosol" foams is presented and the relationship between bubble size and foam rheology discussed. Within the same formulation, small bubbles are associated with high consistency. Container emptying gives rise to foams of lower consistency due to decrease in bubble density which more than offsets a decrease in bubble size. Synergistic consistency effects resulting from blends of fluorochlorohydrocarbon propellents are due to bubble size. Flammable propellents give rise to foams of lower consistency and decreased stability.

THE PAST few years have seen the introduction of a number of pharmaceutical and cosmetic foams which utilize the unique properties obtained from pressurized systems. These include contraceptives, hand creams, anogenital antiseptics, and antitussive products. The increased interest in foams as a dosage form makes it imperative that a more detailed investigation of basic foam systems be conducted in order that the formulation, packaging, and stability problems may be approached theoretically as well as empirically.

A review of foam formation and evaluation has been presented by Richman and Shangraw (1). These authors investigated the usefulness of rheological measurements in foam evaluation and found them to be extremely informative and reproducible (2–4). Studies were made on the effect of propellent type, propellent concentration, soap type, soap concentration, and additives on foam consistency and stability. In addition, a detailed study was made of the loss of consistency which occurs on container emptying, and a mathematical model was presented which predicted propellent concentrations at different stages and rates of container emptying.

All changes in foam consistency and stability should be relatable to the surface area of the foam bubbles and surface viscosity. The object of the present investigation was to study the effect of formulation, time, and can emptying variables on the bubble size of foams produced by pressurized systems, and to relate these studies to foam rheology.

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EXPERIMENTAL

Formulation and Preparation—The same basic formulation suggested by Richman and Shangraw (2) was selected for this study because it combines relative simplicity with significant practical importance in regard to a typical high consistency foam such as shaving cream. This basic formulation consists of: soap concentrate 8%, stearic acid 5 parts; coconut oil fatty acids 3 parts, triethanolamine 5 parts; additive q.s.; distilled water q.s. ad. 100%. Total—90%; propellant 12/114 (57:43) 10%.

The soap concentrate was prepared by adding, with constant stirring, an aqueous solution of triethanolamine, which had been heated to 80° to a mixture of the stearic acid and coconut oil fatty acids which had also been heated to 80°. The mixture was held at 80° for 20 min. and then allowed to cool to room temperature with agitation. Any additives to be included were added, and the mixture was brought to the desired final weight by dilution with a suitable quantity of distilled water. Temperature, time, and rate of mixing were reproduced as closely as possible, as they may influence the properties of the resultant products.

The soap preparation was weighed into "six ounce" standard coated Spra-Tainer[1] cans which had previously been purged with a few drops of the liquid propellent and Precision 0.020/0.080 valves (Precision Valve Corporation, Yonkers, N. Y.) were crimped into place. As previous work in our laboratories has shown that the presence or absence of normal dip tubes does not affect foam properties; dip tubes were nevertheless omitted because the inversion of cans facilitates the filling of test vessels. The propellent was pressure filled into the test cans by weight. The finished products were then shaken vigorously and placed in a 40° bath water for 20 min. to test for leakage and can distortions. Although water bathing is routinely carried out in aerosol. production to check for leakage and can distortion,

[1] Spra-Tainer, Crown Can Co., Philadelphia, Pa.

little attention has been paid to the effects of time and temperature on the final product, which may be considerable for some formulations. After removal from the water bath, the cans were allowed to remain at room temperature for 3 days prior to testing. All testing was completed on a given batch of test products within 2 weeks of their preparations so as to minimize any possible aging factor.

Bubble size analysis was carried out utilizing an American Optical series 682 apparatus and a Polaroid camera. Foams were placed in a chamber which was formed by gluing a parallel pair of polyethylene strips ($^5/_{16} \times {}^5/_{16} \times 1$ in.) one-half in. apart onto a standard glass slide and covering with a cover slip. The $^5/_{16}$-in. deep chamber allows excess foam to flow freely out of the open ends, eliminating bubble distortion and at the same time providing greater protection against the heating effects of the illuminating lamp. Illumination was provided by an American Optical No. 735C microscope illuminator provided with heat absorbing glass. Exposure times up to 3 sec. were required through the first 5 min. elapsed time. Thereafter, as the foams aged, exposures of less than 1 sec. were sufficient.

Bubble size analysis was made by comparing bubbles against a scale made from a photograph of an object micrometer at the respective magnification used. Measurements were facilitated by performing the operation under the low power of an American Optical (Spencer) Cycloptic series 58 stereoscopic microscope. Bubbles were classified into 10-μ intervals and the average bubble radius was computed. An attempt was made to draw 100 bubble samples. However, in rare instances, it was necessary to take as few as 75 bubbles because of the limitations of magnification ranges available, field size, and actual bubble size.

Foam Rheology—Foam rheology was carried out in the manner of Richman (2) using the Haake Rotovisco rotational viscometer (Brinkmann Instruments, Inc., Westbury, N. Y.). All foams evaluated were studied using the MV III system, consisting of a smooth surface stainless steel rotor with a radius of 1.520 cm. and a height of 6.0 cm., and a stainless steel cup with a radius of 2.10 cm. Use of the 500 Gm. cm. torsion spring gave a working range in shearing stress of 27–5900 dynes cm.$^{-2}$ through a rate of shear range of 1–210 sec.$^{-1}$. Although this smooth rotor has proven to give excellent results with our experimental foams, some slippage was observed whene valuating commercial foam products of still higher consistency. For this reason the MV II P profiled bob system was used which gives a narrower gap between cup and rotor and has a serrated surface which reduces slippage.

Each pressurized container was shaken 15 sec. before filling the cup. The foam was introduced directly into the cup through a 5-in. extension of 0.25-in. polyethylene tubing attached to a Precision foam actuator No. SP115. All samples were removed with the valve in its maximum open position. In order to fill the cup uniformly, the cup was filled from the bottom up by slowly withdrawing the tube during actuation. All measurements were made at 28° so that they might better correlate with bubble data. Except in stability studies, readings were begun 1 min. after the cup had been filled. In operation, the time spent at each shear rate was limited to that necessary to make a dial reading, approximately 5 sec.

The need for reproducible technique in regard to evaluating any foam system cannot be overstressed. Such points as can shaking, rate of discharge, filling of evaluation vessels, time of setting before measurements are run, and temperature are critical. However, once such techniques have been mastered, reproducible results can be obtained. One other caution might be interjected at this point. If the formulation of a product is such that significant sputtering occurs when the foam is discharged, no meaningful physical measurements can be obtained. Sputtering means loss of entrapped propellent, which leads to an increase in foam density and a decrease in foam consistency. Although the literature is full of such data, it is impossible to interpret it even relatively.

Experiment I—The first experiment was designed to determine if bubble sizes and foam aging could be correlated with foam rheology. At the same time it was hoped to determine how these parameters are affected by the well-known can emptying effect. To accomplish these objectives, rheograms and bubble size measurements were made on foams after 1, 5, 10, 20, 30, 60, 90, and 120 min. of standing at room temperature (28 ± 1°). These determinations were made at 1, 10, 20, 40, 60, and 80 percentages of can emptying. Separate cans were used for each time increment. Can emptying was accomplished by removing approximately 5-Gm. samples at 15-min. intervals. Before removing samples the cans were shaken uniformly for 15 sec. Two formulations were studied. The first formulation was the basic formulation without additives, while the second formulation included 1.5% methylcellulose 400. The methylcellulose had previously been shown to have a marked effect on foam consistency and its effect on foam stability was of interest. Generally speaking, foam consistencies and bubble sizes decreased with increasing can emptying as did bubble rates of decay.

Experiment II—This experiment was designed to elucidate the effect of propellents on consistency and stability. Foams were prepared containing 10% of propellents 12, 114, or 12/114 (57:43) in two formulations: plain soap and plain soap with 10% propylene glycol. In addition, a formulation containing the hydrocarbon propellent A-46 with a vapor volume identical to the 57:43 blend of the fluorochlorohydrocarbons (*i.e.*, at 3.99% concentration) was included. This equivalent concentration was obtained using the equation of Gorman and Hall (5). In order to show the variation in bubble data, replicates of the plain soap formulation were studied. Bubble analysis and rheograms were determined for each product at 5% and 75% can emptyings. It was felt that the choice of 75% rather than 80% can emptying would ensure a higher degree of success in obtaining a sufficiently large and uniform sample. Of the fluorochlorohydrocarbon products, those prepared from plain propellents 12 and 114 showed the largest bubble sizes and the poorest consistencies at 5% can emptying. Products using the propellent blend were superior in consistency and showed the smallest bubbles. The addition of propylene glycol generally enhanced consistency, reduced bubble sizes, and reduced their rate of growth. Propellent A-46

generally produced foams with lower consistencies and less uniformity than those obtained using equivalent amounts of the fluorochlorohydrocarbons.

DISCUSSION

The choice of propellent and propellent concentration for use in an aerosol product has traditionally been governed by empirical evaluation of the initial product, stability, and economics. In most cases it has been difficult to ascertain subtle influences of formulation and packaging components and propellent changes due to the lack of sensitive quantitative tests on the final properties of the foam. Rheological evaluation has proven to be an effective means of determining changes in foam consistency, which is certainly one of the most important properties of foams. Furthermore, changes in foams which occur upon aging are reflected in such measurements. It has also been known that the changes in foam properties which occur during use of the product due to can emptying effects may be monitored by rheological measurements. The discovery of the discrete changes in consistency which can be brought about by relatively small changes in formulation raised the interesting question as to what physical-chemical phenomenon these changes could be attributed. It seemed obvious, and later work has borne this fact out, that changes in consistency can be attributed to changes in surface area or changes in viscosity of the interbubble film, probably as reflected in surface viscosity. Taking these influencing factors one at a time, a comprehensive study of the effect of formulation variables on bubble size was undertaken.

The effect of aging of a plain foam on the average bubble diameter at various stages of can emptying is shown in Fig. 1. The same effects on a formulation including methylcellulose can be seen in Fig. 2. Initial bubble radii for both formulations were in the general region of 10 μ. Bubble growth during the first 20 min. was rapid and essentially exponential. However, after 20 min., average bubble diameter seemed to be directly proportional to time.

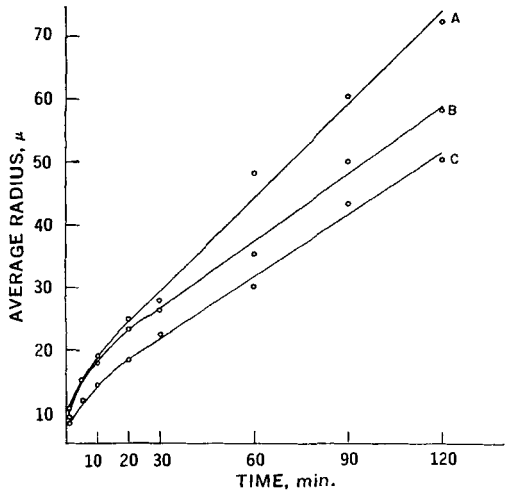

Fig. 1—Bubble growth curves: effect of can emptying (basic soap formulation without additive). Curve A, 10% empty; curve B, 40% empty; curve C, 80% empty.

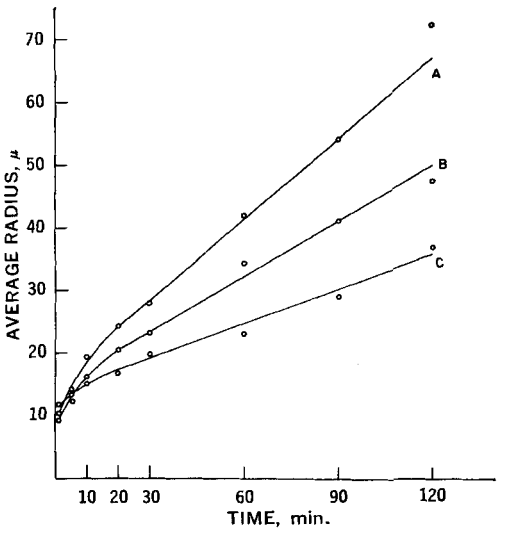

Fig. 2—Bubble growth curve: effect of can emptying (basic soap formulation with 1.5 % w/w methylcellulose 400). Curve A, 10% empty; curve B, 40% empty; curve C, 80% empty.

The initial apparent rapid growth in bubble size can be attributed to the presence of many very small bubbles which would have disappeared after a few minutes. Other factors which may play a role include (*a*) residual vaporiziation of propellent, (*b*) increase in vapor volume as the system equilibrates from cooling effects of propellant vaporization, and (*c*) higher kinetic energy in the initial system and possible greater initial permeability of interbubble films.

One very striking fact can be noted in almost all studies of bubble size *versus* can emptying. The average bubble size decreases as can emptying increases. At the same time the rate of coalescence of bubbles decreases. This means that foams produced at later stages of can emptying are more stable. Smaller bubble size would indicate a larger surface area and thus an increased consistency. However, these results are misleading in regard to the actual consistency of foams, which is known to substantially decrease during container emptying (2). This is due to the fact that although the bubble size decreases, the number of bubbles is also substantially smaller. This lower bubble density results in a much lower surface area which is then reflected in the decreased consistency. The decrease in bubble density helps also to explain the decrease in rate of coalescence of bubbles produced at later stages of emptying. Interbubble films are thicker and diffusion of vapor is thus retarded. These results are consistent with the conclusions of DeVries, who points to the thickness of bubble lamellae as being one of the most important factors in the rate of bubble coalescence (6). The works of DeVries along with that of Clark and Blackman (7) offer an indispensable background for all workers interested in bubble size analyses.

With regard to formulation variables, the plain soap formulation appeared to age more rapidly than the one containing methylcellulose, and this becomes more evident with can emptying. This

can be explained as the result of both a retardation of film drainage and a retardation of drying due to film formation on the surface of the bubbles. As expected, these effects are more significant at later stages of can emptying where the foam is denser.

Foam consistencies decrease with container emptying. Previous works by Richman and Shangraw, and Fong and Shangraw (8) confirmed in this study, show that this decrease in consistency is not directly proportional to can emptying but plateaus in the general region of 40–60% can emptying. It was hoped that bubble size analysis data would show a similar pattern, as it is difficult to envision anything but bubble size playing an important role. However, correlation did not materialize. The plateauing in consistency which occurs in can emptying is thought to be related to the fractionation of propellent blends which occurs as the propellent vaporizes into the headspace. The need for a better understanding of these effects led to the experiment in which the type of propellent was varied.

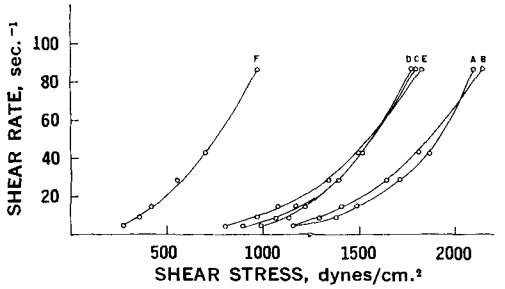

Fig. 3—Pressurized foam flow curves: effect of can emptying on 1 min. rheology (basic soap formulation with 1.5 % w/w methylcellulose 400). Curve A, 1% empty; curve B, 10% empty; curve C, 20% empty; curve D, 40% empty; curve E, 60% empty; curve F, 80% empty.

Bubble analysis can generally be correlated with foam rheology when the formulation remains constant. The effect of can emptying on the methylcellulose formulation may be seen in Fig. 3. As expected, the foam containing the methylcellulose exhibited higher consistencies but the relative drop in consistencies on container emptying remained constant. As initial bubble sizes of the two formulations are very similar, consistency changes are obviously due to viscosity effects, either in the bulk of the liquid, or, more probably, in the surface of the bubbles.

Photomicrographs of a typical shaving cream foam at different times of aging can be seen in Fig. 4. Initially, a shaving cream foam looks more like a gas emulsion since bubbles are essentially spherical. It is only after these high consistency foams have aged for a period of time and the interbubble films have drained, that the classical pentagonal dodecahedrons begin to form.

An interesting insight into the variabilities which can occur in bubble patterns of two high consistency commercial vaginal foams used in contraception is shown in Fig. 5. While one foam appears to be similar to the shaving cream, the other is much more polydisperse and seems to be of poorer quality. The polydisperse foam with thick lamellae shows little change on aging (Fig. 6). The few small bubbles have disappeared as would be expected, but the other bubbles have shown little growth. This photomicrograph points up the basic dynamics of bubble distribution in foams which occurs on aging, *i.e.*, small bubbles become smaller and larger bubbles grow. This is due to the increased curvature of a small bubble which results in higher relative pressure.

The results of the bubble size analysis conducted on foams prepared using different fluorochlorohydrocarbon propellent systems at two stages of can emptying can be found in Tables I and II. Two formulations were employed, one containing

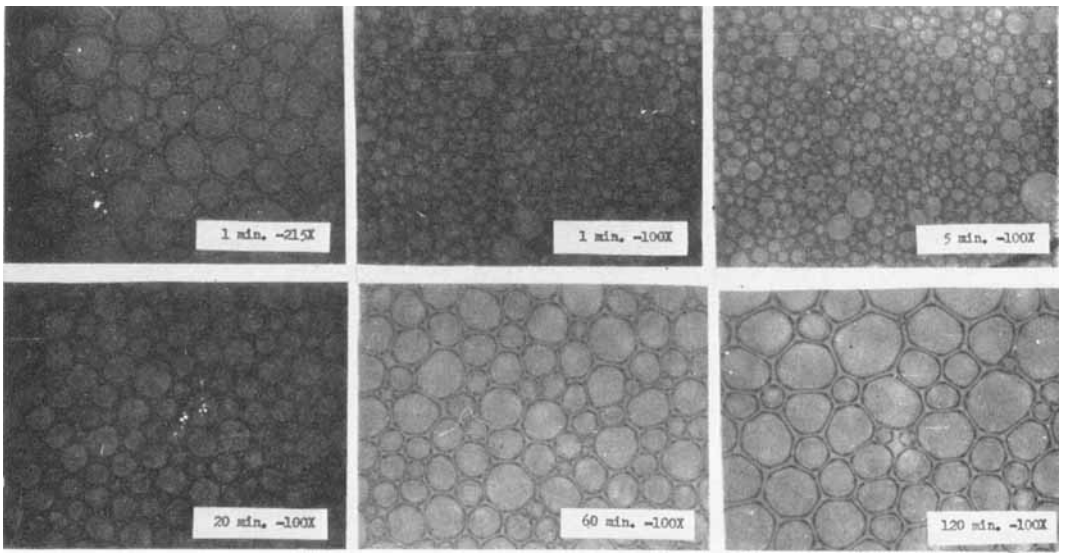

Fig. 4—Photomicrographs illustrating the effect of aging on a typical pressurized shaving foam.

plain soap (with replicate) and one containing 10% propylene glycol. The corresponding flow curves are presented in Fig. 7.

Bubble size data corresponded to consistency curves in all systems, *i.e.*, as bubble size decreased, consistency increased. However, there were some unusual results obtained in regard to bubble sizes produced by each propellent system.

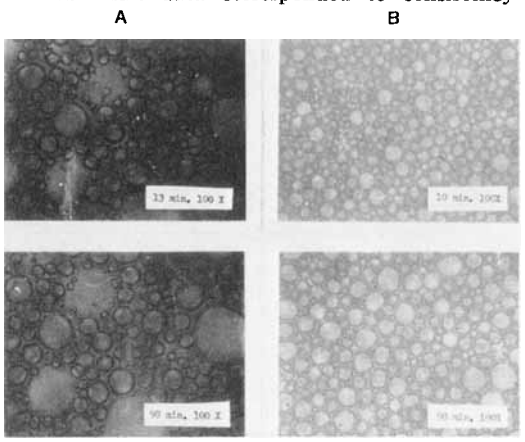

Fig. 5—Photomicrographs comparing commercial pressurized vaginal foams.

Propellent 12 gave smaller bubble sizes and higher consistencies than 114. These consistency data were not in agreement with the preliminary work done by Richman (2), but were reproducible and in line with bubble size measurements. However, the synergistic effect that propellent blends have on foam consistency was apparent in both studies. The reason for this increased consistency can now be seen to be due to the overall decrease in bubble size exhibited by foams prepared from blends. The reason for this decrease in bubble size is not immediately obvious but must be connected to the propensity for solubilized or emulsified molecules of propellent to associate into a single bubble of vapor when pressure is reduced during expulsion of foam.

The changes in bubble size which occur in foam aging can be seen in Fig. 8. Foams containing propellent 12 age extremely rapidly and would be expected to have sharp decreases in consistency.

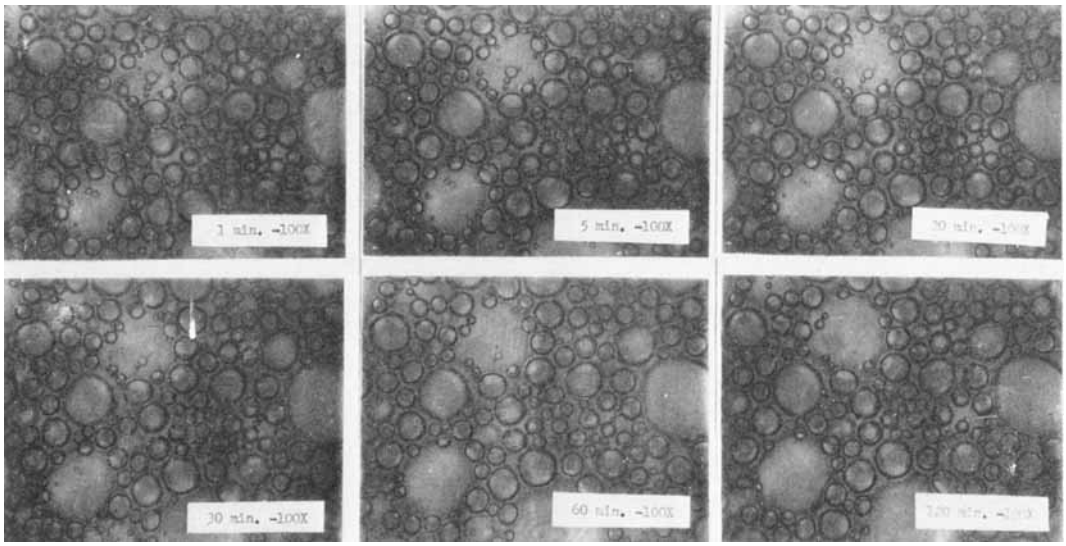

Fig. 6—Photomicrographs illustrating the effect of aging on a commercial pressurized vaginal foam.

TABLE I—EFFECT OF PROPELLENT TYPE ON BUBBLE SIZE OF TYPICAL SHAVING CREAM FOAMS 5% CAN EMPTYING

	Propellent 12 — Radii, μ			Propellent 12/114 (57:43) — Radii, μ			Propellent 114 — Radii, μ		
Time (min.)	Run 1[a]	Run 2[a]	Run 3[b]	Run 1[a]	Run 2[a]	Run 3[b]	Run 1[a]	Run 2[a]	Run 3[b]
1	14.8	15.8	10.5	10.2	9.7	10.6	20.2	21.0	30.2
5	22.4	22.8	15.2	15.3	13.7	12.0	21.1	21.5	31.0
10	27.2	31.6	20.3	21.2	18.2	15.0	21.4	22.7	30.2
20	38.0	37.3	24.8	25.3	22.0	17.1	24.0	24.7	30.6
30	47.0	50.8	31.2	27.1	24.9	21.7	25.1	25.9	31.8
60	63.4	69.4	42.1	36.5	37.3	30.0	28.2	27.7	34.9
90	76.6	83.6	56.0	50.6	51.2	35.0	36.9	36.0	33.9
120	100.2	102.1	64.1	58.0	56.4	43.2	45.2	43.1	34.7
Approx. slopes—linear portions[c] (μ min.$^{-1}$)	0.618	0.625	0.425	0.345	0.344	0.238	0.217	0.182	0.038

[a] Plain soap formulation. [b] Plain soap formulation with 10% propylene glycol. [c] Linear portions taken from 20 to 120 min.

TABLE II—EFFECT OF PROPELLENT TYPE ON BUBBLE SIZE OF TYPICAL SHAVING CREAM FOAMS 75% CAN EMPTYING

Time (min.)	Propellent 12 Radii, μ Run 1[a]	Propellent 12 Radii, μ Run 2[a]	Propellent 12 Radii, μ Run 3[b]	Propellent 12/114 (57:43) Radii, μ Run 1[a]	Propellent 12/114 (57:43) Radii, μ Run 2[a]	Propellent 12/114 (57:43) Radii, μ Run 3[b]	Propellent 114 Radii, μ Run 1[a]	Propellent 114 Radii, μ Run 2[a]	Propellent 114 Radii, μ Run 3[b]
1	10.0	9.4	9.4	7.1	7.6	8.4	6.8	7.5	6.7
5	14.4	15.0	15.4	12.0	10.6	11.9	9.2	9.9	7.7
10	19.5	19.0	17.4	14.2	15.4	16.3	11.9	12.0	9.9
20	26.9	25.4	24.2	19.8	17.5	18.5	13.2	15.1	12.7
30	40.7	40.5	27.1	23.1	21.4	22.5	18.8	17.9	14.7
60	50.8	54.5	37.6	36.9	32.7	29.8	24.4	22.8	18.4
90	72.5	68.1	50.1	48.5	46.0	38.7	29.3	27.8	22.1
120	80.2	84.4	59.4	53.8	55.4	42.3	34.1	35.4	25.9
Approx. slopes— Linear portions[c] (μ min.$^{-1}$)	0.500	0.500	0.357	0.385	0.370	0.285	0.172	0.172	0.125

[a] Plain soap formulation. [b] Plain soap formulation with 10% propylene glycol. [c] Linear portions taken from 20 to 120 min.

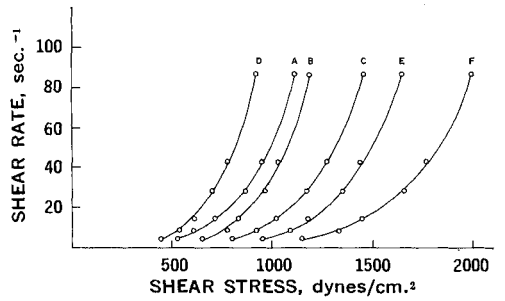

Fig. 7—Pressurized foam flow curves: effect of different nonflammable propellent systems (basic soap formulation with and without 10% w/w propylene glycol—5% can emptying). Key: A, 10% 114–plain soap; B, 10% 12—plain soap, C, 10% 12/114 (57:43)–plain soap, D, 10% 114–10% propylene glycol; E, 10% 12–10% propylene glycol; F, 10% 12/114 (57:43)–10% propylene glycol.

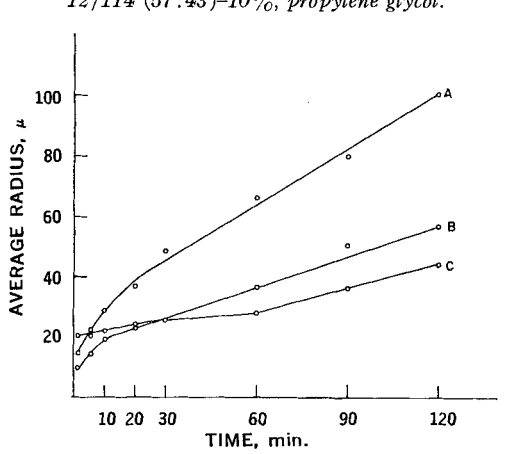

Fig. 8—Bubble growth curves: effect of different nonflammable propellent systems (basic soap formulation without additive—5% can emptying). Key: A, 10% 12; B, 10% 12/114 (57:43); C, 10% 114.

On the other hand, foams prepared from propellent blends and plain propellent 114 have identical bubble sizes after 30 min. All foams containing only propellent 114 age extremely slowly. It seems apparent that propellent 114 does not diffuse as rapidly through the interbubble walls as does propellent 12. The presence of propylene glycol in the formulation gives rise to smaller bubble sizes and slower aging in those foams utilizing propellent 12 or propellent blends. On the other hand, foams produced with propellent 114 have a much larger initial bubble size but show extremely little aging over a 2-hr. period. In all cases, addition of propylene glycol resulted in a decrease in the slopes of the linear portions of the time-radius plots.

One unusual effect is that wherein the propylene glycol formulation actually gives a lower consistency foam than the plain soap when propellent 114 is employed. The effect was so unexpected that additional samples were tested with confirming results. Although the relatively low boiling point and longer time required for vaporization equilibrium to be achieved makes data obtained from propellent 114 less reliable, the effects noted are real and reproducible.

The 75% can emptying data presented in Table II point up the characteristic decrease in bubble size and lower rate of coalescence which occurs in foams produced from the latter part of a pressurized container. This effect is particularly striking in the case of propellent 114 in which the average

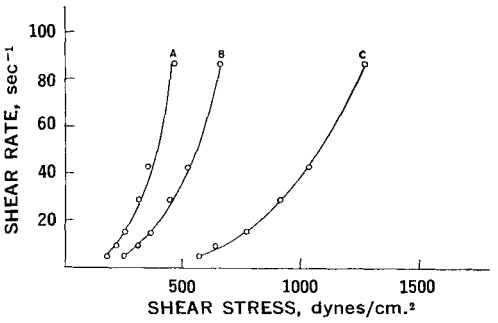

Fig. 9—Pressurized foam flow curves: effect of different nonflammable propellent systems (basic soap formulation with 10% w/w propylene glycol—75% can emptying). See Fig. 8 for Key.

TABLE III—COMPARISON OF PROPELLENT BLEND 12/114 (57:43) AND PROPELLENT A-46 AT MATCHED VAPOR VOLUMES

	Radii, μ							
	5% Can Emptying				75% Can Emptying			
	Propellent 12/114 (57:43)		Propellent A-46		Propellent 12/114 (57:43)		Propellent A-46	
Foam Age, min.	Plain Soap	With Propylene Glycol	Plain Soap	With Propylene Glycol	Plain Soap	With Propylene Glycol	Plain Soap	With Propylene Glycol
1	10.0	10.6	11.6	8.8	7.4	8.4	9.2	8.0
90	50.9	35.0	61.8	48.5	47.3	38.7	48.0	36.6

bubble size is now the smallest of the three propellent systems studied. On this basis, it would be expected that consistency of the 114 foams would not decrease as much as would be expected. The consistency curves shown in Fig. 9 bear out this assumption with the effects in the propylene glycol formulation being particularly striking. The foam obtained at 75% can emptying from propellent 114 in the presence of propylene glycol exhibits a higher consistency than any of the other 114 curves at either level of can emptying. These results have to be relatable to bubble size. Although the reason for this is not clear, the propellent 114 appears to be better emulsified or solubilized in the presence of a large excess of soap. The fact that fractionation of propellents upon can emptying favors the retention of propellent 114 in the concentrate combined with the above effects might serve as a basis for improving foam quality at latter stages of container use. Unfortunately, propellent 114 alone is not energetic enough to serve as the sole propellent in a foam system.

Because it was felt that nonflammable propellents would have a greater application to pharmaceutical foam systems, a great deal of the experimental work was devoted to the investigation of the fluorochlorohydrocarbon propellents. However, the fact remains that economics have resulted in the widespread use of the flammable propellents (isobutane, propane) in cosmetic foams such as shaving creams. A recent analysis of shaving creams of 10 major companies indicates that only two are using nonflammable propellent systems.

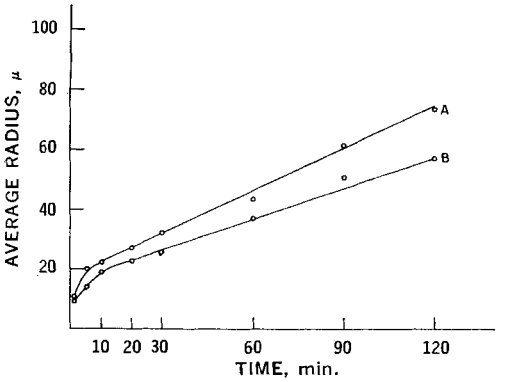

Fig. 10—Bubble growth curves: comparison of flammable and nonflammable propellent systems with matched vapor volumes (basic soap formulation without additive—5% can emptying). Key: A, 3.99% A-46; B, 10% 12/114 (57:43). Slope of linear portion (μmin.$^{-1}$): A, 0.476; B, 0.345.

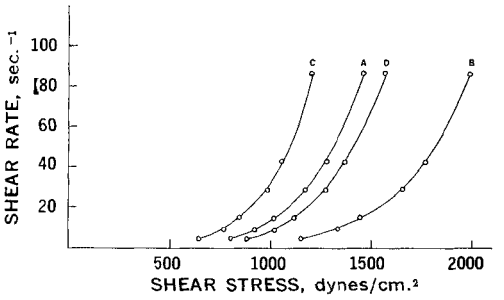

Fig. 11—Pressurized foam flow curves: comparison of flammable and nonflammable propellent systems with matched vapor volumes (5% can emptying). Key: A, plain soap + 10% 12/114 (57:43), B, same as in A + 10% w/w propylene glycol; C, plain soap + 3.99% w/w A-46; D, same as in C + 10% w/w propylene glycol.

A summary of the bubble size data obtained from foams produced from the two propellent systems with matched vapor volumes is shown in Table III. Initial bubble sizes appear to be quite similar but bubble growth is more rapid in the A-46 systems. This increased rate of aging which occurs in the flammable propellent system is illustrated in Fig. 10. It would appear that the flammable propellents diffuse more rapidly through the interbubble walls than do the nonflammable propellents, indicating a slightly lower degree of stability.

The consistency curves shown in Fig. 11 obtained from the two propellent systems indicate the existence of a much greater difference between the foams than was obvious from the bubble data. The A-46 foams gave significantly lower consistencies for both concentrate formulations studied. This decreased consistency in light of fairly equal average bubble diameters can best be explained by looking at a bubble size frequency distribution, as is shown in Fig. 12. The much wider distribution of bubble

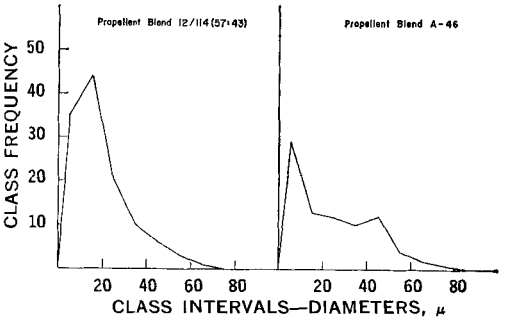

Fig. 12—Initial (1 min.) bubble size frequency distributions: Comparison of flammable and nonflammable propellent systems with matched vapor volumes (basic soap formulation without additive—5% can emptying).

sizes and resultant decrease in total surface area in the A-46 foam helps to explain the lower consistencies. This lack of bubble size homogeneity would seem to indicate that emulsification of A-46 propellent is more difficult. A great deal of work remains to be done on characterization of foams from flammable propellent systems.

SUMMARY AND CONCLUSIONS

(*a*) A photomicrographic technique is described for the determination of the growth of bubbles in aerosol foams which yields a reasonably good degree of reproducibility.

(*b*) Within the same formulation and propellent concentration, smaller bubbles are associated with higher consistencies.

(*c*) As a container is emptied, bubble sizes and growth slopes tend to decrease due to a higher ratio of soap to propellent and thicker interbubble films. Consistency also decreases.

(*d*) The addition of methylcellulose increases foam consistency and decreases the rate of bubble coalescence.

(*e*) Initially, propellent 114 yields larger bubbles than propellent 12. Blends yield smaller bubble sizes than either single propellent. These results are consistent with foam rheology at both 5% and 75% can emptying.

(*f*) The addition of propylene glycol tends to decrease bubble sizes and increase foam consistency except in cases of plain propellent 114 where unusual effects are noted, particularly at 75% can emptying.

(*g*) When vapor volumes are matched, flammable propellents give rise to foams of lower consistency and decreased stability. This is not always obvious when observing average bubble size alone, as bubble sizes are more disperse when A-46 propellents are used.

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Keyphrases

Aerosol foams—bubble size analysis
Bubble size—aerosol rheology
Photomicrographic technique—bubble size determination
Aging foam—bubble diameter effect
Can emptying—bubble size, density
Propellent—foam bubble size

Drug Standards

Determination of Free Salicylic Acid in Buffered Aspirin Tablets

By JOSEPH LEVINE and JOHN D. WEBER

Aspirin and free saliyclic acid are quantitatively released from buffered aspirin tablets, with a negligible degree of hydrolysis of aspirin, by briefly wetting the sample with 98–100 percent formic acid, followed by immediate dilution with chloroform. The salicylic acid is isolated and determined as previously described.

SALICYLIC ACID can be quantitatively separated from aspirin by trapping it as the complex formed with ferric ion on a partition chromatographic column, using ferric chloride (1) or ferric chloride-urea (2) solutions as the immobile phase.

In the application of this procedure to analytical samples, it is essential that the entire amount of aspirin and salicylic acid be dissolved in the mobile phase before the chromatographic treatment, and that the aspirin is not hydrolyzed during the preparation of that solution. These

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