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Received for review July 24, 1972. Accepted December 6, 1972. Reference to brand or firm name does not constitute endorsement by the U. S. Department of Agriculture over others of a similar nature not mentioned.

Whey Solids as Agricultural Foam Stabilizers

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"Chemifoams" were custom-tailored using whey solids and a mixture of whey solids and animal hide glue as stabilizers for protecting row crops from frost and freeze damage, as carriers for herbicides, fungicides, defoliants, and the like, as foam covers after soil fumigation with gaseous fumigants, and as foam markers. A foam containing 1.5% whey solids and 2% Hi-X, a surfactant, was a satisfactory carrier for the herbicide Karmex. A heavier more durable foam containing 1% whey solids, 1% animal hide glue, 0.3% Natrosol, and 2% Hi-X, called the "standard formulation," showed improved persistence of 200% at 36°F, 386% at 68°F, and 400% at 73°F. Several variables affecting the standard were examined. Reduction in temperatures caused an orderly increase in persistence of the foam. Maximum persistence was obtained at 67 and 73°F for 2-day-old formulations and at 36°F for 3-day-old formulations. Adjustment of pH to any but the normal pH of the standard was found unnecessary. Optimum concentrations of Natrosol and surfactant were determined and possible dilution of standard was examined.

Agricultural foams ("chemifoams") are generated to give frost and freeze protection to row crops, to act as carriers for herbicides, fungicides, defoliants, and the like, to serve as soil covers after soil injection with volatile fumigants, and to act as farm markers. Application of foams for these purposes *via* ground-operated equipment minimizes pollution in that chemicals are not carried through the air by wind, prevents drift, and reduces total chemicals required. For the above purposes, foams generally contain stabilizers to give them strength, form, the capability of producing skins, extended use life, self-destructiveness of foam and skin, absence of phytotoxicity, and economic feasibility.

Whey solids recovered from whey, the byproduct of cheese manufacture, have potential as a stabilizer for chemifoams. Whey has a high biological oxygen demand and has long been a prime pollution problem in areas where large cheese manufacturers are operating (Chem. Eng. News, 1971). The environmental problem of disposing of whey from cheese manufacture became sharply critical in 1970. Published statistics on whey utilization (Mathis, 1970) show that approximately 11 billion lb of whey out of a total of 21.4 billion lb produced annually are returned to farms for feed or fertilizer, or they are wasted, the last amounting to nearly half of the whey returned. Profitable new uses are urgently needed. Basically, the components present are protein (lactalbumin), lactose, and salts. It would be preferable to utilize this material in foods and feeds but apparently the current market suffers from a lack of outlets and other outlets, even industrial ones, should be considered.

Whey is dried by spraying into hot air or by coating it on the surface of steam-heated rollers. The least expensive of the products is roller dried and is suitable only for animal feed (Webb, 1970). It is this last product which has been found utilizable in chemifoams either alone or in combination with other materials. This paper presents data to show that inedible whey solids can be substituted for gelatin (Braud and Chesness, 1968) or animal glue (Lambou *et al.,* 1972) in the preparation of long-lasting chemifoams, shows that their performance can be improved greatly by combining with additional protein-containing materials, and discusses some of the parameters and their effects on the characteristics of these foams.

EXPERIMENTAL SECTION

Materials. Listed in Table I are products from seven companies recovering both edible and inedible whey solids in a range of prices and composition. Edible whey solids are spray dried and most feed-grade whey solids are roller dried. Actually all of the whey solids listed produce good foams. An inedible whey product was selected for further study because of its price differential and ease of handling in evaluating a number of variables affecting pertinent agricultural foam characteristics.

Also selected for study were: surfactant Hi-X (a modified alkylsulfate), Walter Kidde & Co., Inc.; viscosity-builder Natrosol (hydroxyethylcellulose), Hercules, Inc.; and herbicide Karmex [3-(3,4-dichlorophenyl)-1,1-dimethylurea], E. I. Dupont de Nemours & Co., Inc.

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Table I. Whey Solids Used in the Preparation of Chemifoams

Trade name	Industrial label	Price, ¢/lb	Composition: Protein, %	Lactose, %	Fat, %	Ash, %	Moisture, %	pH
Edible								
Teklac[a] Sweet Dairy Whey	KAO11 No. 375	8	12–14	67–68	1.25 max	10	5	
Placto[b] Acid Whey		16.5 to 14.5	7.5	71.5	0.4	11.7	3.0	4.6 (10% solids)
Kraflow[b]		28–24	15.0	68.5	1.25	8.0	3.0	5.9 (10% solids)
Edible Whey Solids[c]			12–13	69–73	1.08	8–9	5	6.0 (6% solids)
XL Sweet Dairy Whey[d]		10.5 to 7.5	11		1.25		5	
Modified Whey[e]	LP20	12–11	22	56	1–2	21	4	7.2 (10% solids)
Inedible								
Kraco[b]	Type X 291LO	5.5	12.9	72.0	0.9	8.0	4.5	5.9 (50% solids)
Kraco[f]	202DO	5.5	12.9	72.0	0.9	8.0	4.5	5.9 (50% solids)
Feed Grade Whole Whey[g]	PDW	4.5	16	50	1.0	17	7 max	
Animal Whey Solids[c]		6.5	12–13	69–73	1.25 max	8–9	6.2	6.0 (6% solids)

[a]Product of Foremost Foods Co., spray dried. [b]Product of Kraft Foods, spray dried. [c]Bongard's Creameries, spray dried. [d]Land O' Lakes, spray dried. [e]Purity Products Co., spray dried. [f]Kraft Foods, roller dried. [g]Foremost Foods Co., roller dried, Code MNC, No. 129.

Equipment. Foams were generated mechanically on a laboratory scale in the apparatus shown in Figure 1. This generator was built by the engineers at Louisiana State University (LSU-BR) in Baton Rouge. The apparatus is described by Braud and Chesness (1968) and depends on its capacity to incorporate large quantities of air in small bubbles in a selected liquid formulation by forcing the liquid through a series of screens at 35 psi. Field application of foam was accomplished with a tractor-drawn foam generator fitted with a cone spray nozzle and two screens. Viscosity determinations were made in a Brookfield Synchroelectric Viscometer at 75°F using a no. 1 spindle at 20 rpm. Density, volume, and persistence were measured in metal pans 8 in. in diameter, 3 in. in height, and fitted with a ⅜-in. i.d. drainage tube. Side of pan opposite drainage tube was elevated 1.25 in. to facilitate drainage.

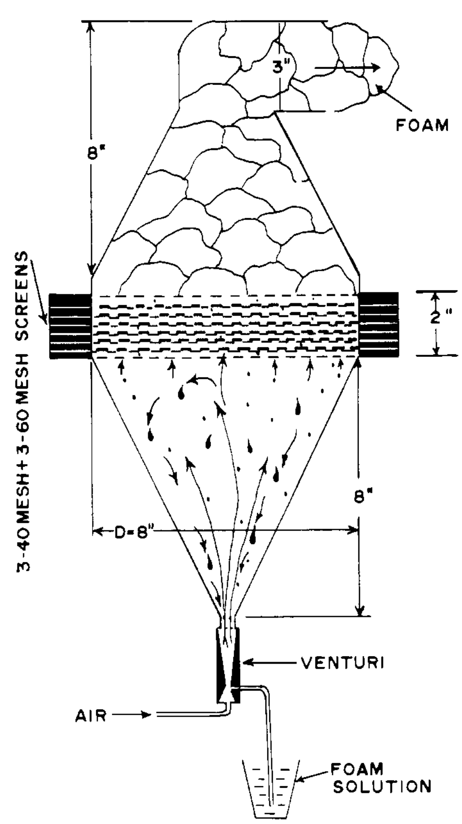

Figure 1. Laboratory-size cone generator for preparing chemifoams.

Methods. Principal characteristics of the foams measured included: density as grams per cubic centimeter; expansion as cubic centimeters per gram; viscosity in centipoises; drainage as cubic centimeters of liquid draining from the foam; and persistence as time (in hours) at which the foam subsided to 33% of its original volume.

RESULTS

Whey as a Stabilizer. Foam characteristics of at least two formulations from each category, edible and inedible, containing besides whey solids 0.2% Natrosol and 2% Hi-X, are outlined and compared with foam characteristics of an animal glue formulation (Lambou *et al.*, 1972) in Table II. Densities and expansions are of the same order of magnitude. Persistence at 36°F compared favorably with that of animal glue. Foams made from whey solids were observed not only at 36°F but also at outdoor temperatures 64–78°F and at room temperature 72–74°F. The foams seemed to be sensitive to temperature; higher temperatures significantly reduced their persistence. Foams containing feed-grade whey solids that had been roller dried were entirely satisfactory and compared favorably with those made from spray-dried edible whey solids. Hence, inedible whey solids can be substituted for animal glue in a chemifoam formulation.

Table II. Use of Edible and Animal Grades of Whey Solids in the Preparation of Chemifoams

Trade name	Concentration, %	pH	Density	Expansion	Persistence in hours at 36°F	Room temp, 72–74°F	Outdoors, 64–78°F
Teklac[a]	2	5.6–5.85	0.0178–0.0223	45–56×	15–20	3–5	2.5–3
Placto[a]	2	4.6–4.96	0.0152–0.0179	56–66×	18–23	4–5	
Animal Glue	2		0.0174–0.0230	44–58×	20–24		
Feed Grade Whole Whey	2		0.0181	55×	24	4	1.9
	3		0.0230	44×	24	5	2.1
Kraco[b]	1		0.0145–0.0196	51–69×	24–32	5–6	3–7
	2		0.0120	50×	41	6	7

[a]Edible. [b]Feed grade.

Field Trials. Field trials of whey solids foam as a herbicide carrier were successfully carried out by the Agricultural Engineers (LSU-BR). Plots used were 4 × 32 ft. They were covered by mature crabgrass (5-6 in. tall) and a few pigweeds. Crabgrass at the above stage is hard to kill. Treatments included: Karmex at the standard rate of application of 0.5 lb/acre of active ingredient plus the Southern Regional Research Laboratory (SRRL) foam containing 1.5% Kraco plus 2% Hi-X; Karmex at one-half the standard rate of application plus the SRRL foam carrier; and Karmex at the standard rate applied conventionally as a spray. Four replications of each treatment were set up. There were also four untreated plots. Three days after treatment the following results were observed: Karmex, standard rate + SRRL foam, 50% weed kill; Karmex, one-half standard rate + SRRL foam, 20% weed kill; Karmex, standard rate, no foam, 0 weed kill; and check, no treatment, 0 weed kill.

The foam had an expansion of 65-70× and a longevity or persistence of 45 min, which was considered good for the purpose. The herbicide was added to the formulation prior to generation of the foam and its application. The mixture was compatible.

Improved Foam Stability. Stability or persistence of a whey solid foam could be improved by adding small concentrations of protein such as animal glue (0.5-1% of A Reg 349 Foamy) (Lambou *et al.*, 1972) to a formulation containing 1 or 2% inedible Kraco, 0.3% Natrosol, and 2% Hi-X. An orderly increase in persistence was observed at three temperature levels (Table III). Expansion decreased with an increase in the concentration of animal glue. Because of the cool (72-62°F) cloudy weather outdoors, the exposed foams exhibited a particularly lengthy longevity. The better formulations seemed to be those made with 1% whey solids. One percent animal glue added to 1% whey solids plus 2% Hi-X and 0.3% Natrosol in the formulation improved longevity of the foams by 208% at 36°F, 386% at 68°F, and 400% at 73°F. For the purposes of this paper, the preceding formulation will be known as the "standard formulation."

Table III. Improvement of Whey Foam Characteristics with Addition of Protein as Animal Glue

Concentration: Whey solids, %[a]	Animal glue, %	Expansion	Persistence in hours at 36°F	72–74°F	64–78°F
1		51×	32	6	7
1	0.5	38×	41.5	6	23
1	0.8	31×	60	18	3
1	1	31×	66.5	24	27
2		50×	41	6	7
2	0.5	45×	56	8+	8
2	0.8	33×	56	17	8
2	1	25×	68	24	

[a]Kraco, feed-grade whey solids.

Effect of Temperature. If results outlined in Table III are averaged at the various levels of animal glue added to the formulation (0.5% curve A, 0.8% curve B, and 1% curve C) and graphed as in Figure 2, a series of almost parallel lines evolves showing that temperature has an orderly effect on persistence of these foams and that persistence increased as the temperature was lowered. Average temperatures at which the foams were observed are 73, 68, and 36°F. Each point on a curve represents six determinations. A curve representing whey solids without animal glue would fall below curve A.

Effect of pH. In total volumes of 700 cm^3 of standard formulation containing 1% whey solids, 1% animal glue, 0.3% Natrosol, and 2% Hi-X, the pH was adjusted to range between 3 to 9 by adding the required quantities of 0.1 *N* acetic acid or 0.1 *N* ammonium hydroxide. Changing of pH caused much irregularity in foam characteristics, as shown in Figure 3. Persistence at 36°F was at an optimum at pH 6 and pH 9. Expansion was lowest at pH 9 and density and viscosity were highest. However, the same persistence was obtained at pH 6, which is the standard formulation that does not require a pH change. Hence it would appear that a pH change is not desired.

Effect of Dilution. Dilution of the standard formulation with distilled water in 10% increments from 0% (undiluted) to 50% gave the results shown in Figure 4. Reducing concentration by dilution increased expansion from 47× to 72× and decreased viscosity from 62 to 15 cP. Dilution also reduced persistence at the three temperature levels

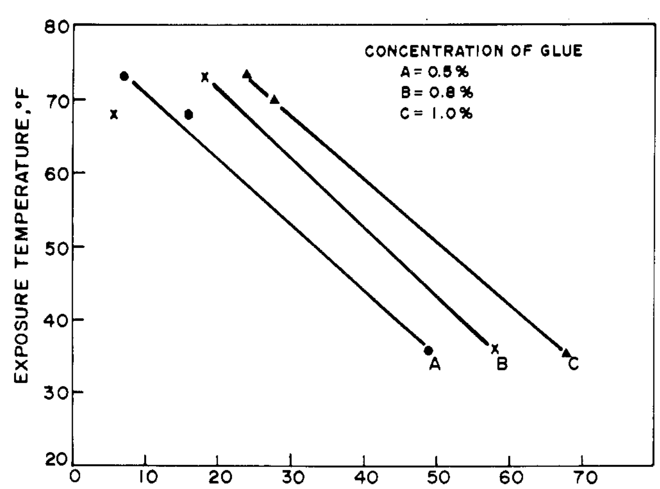

Figure 2. Effect of temperature on whey solids foams enhanced with animal glue protein.

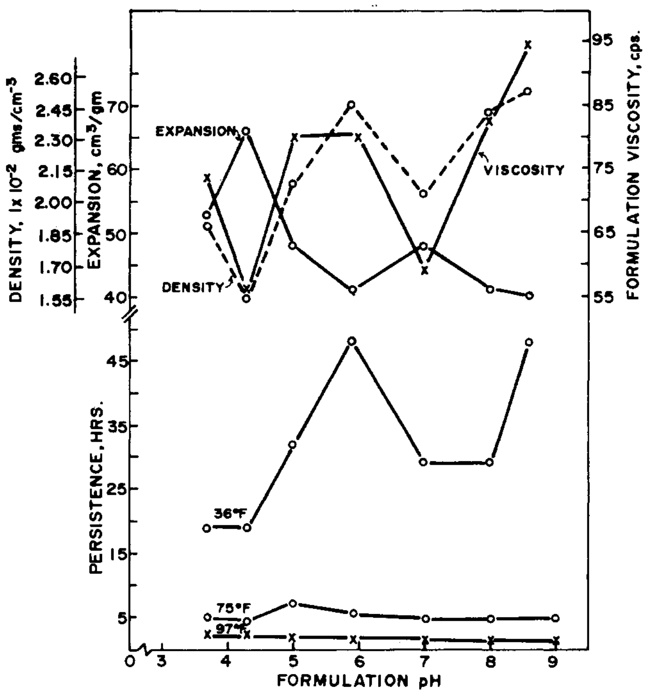

Figure 3. Effect of pH on foam characteristics.

36, 79, and 98°F. Optimum results in persistence with average expansion and viscosity are shown to correspond to the formulation diluted with 20% water. This is an important factor in cost calculations.

Effect of Aging. A series of 18 experiments was conducted to determine the effects of age of the standard formulation on such foam characteristics as expansion, density, viscosity, pH, and persistence. Age of the formulation extended from 24 hr to 5 days and was tested daily.

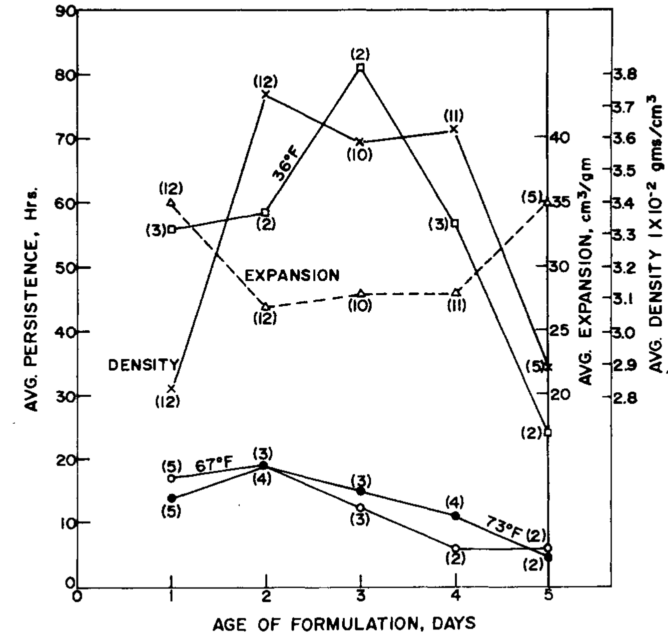

Figure 5. Effect of aging of standard formulations on foam characteristics.

Persistence, density, and expansion are shown graphically in Figure 5. Numbers in parentheses at the various points on the curves indicate the number of replicates averaged for that particular point. Density fluctuated irregularly and, for the most part, inversely to expansion. Expansion resulted in a bowl-shaped curve, higher on the first and fifth days and considerably lower on the second, third, and fourth days. For foams observed at room temperature (73°F) and those set outdoors at an average temperature of 67°F, persistence peaked when the formulation from which the foams were generated was 2 days old and thereafter decreased for older formulations. Persistence reached a peak for foams generated from 3-day-old formulations when stored at 36°F and decreased rapidly on the fourth and fifth days. It can be concluded from this set of experiments that if a foam generated from the standard whey solids–glue formulation is to be used for frost or freeze protection, the prepared liquid formulation may be up to 3 days old and still be usable. If, on the other hand, the formulation is to be used in the middle temperature range, 65–75°F, the liquid formulation should not be more than 48 hr old before generation of the foam.

Viscosity and pH measurements were also made on the standard formulation over a period of 7 days, as shown in Figure 6. Viscosity increased rapidly between the first and second day and thereafter decreased steadily. There was very little fluctuation in pH.

Effect of Concentration of Viscosity-Builder. Concentrations of Natrosol were increased from 0.1 to 0.5% in increments of 0.1% in the standard formulation. Both 24-hr and 48-hr formulations were tested. Figure 7 shows, as expected, that expansion decreases as density and the concentration of the viscosity-builder increase, and that increasing the concentration of Natrosol causes an accompanying increase in density. Of interest is the clustering of points on both expansion and density curves at the 0.3% concentration of the viscosity-builder where expansion ranges from 35 to 40 cm^3/g and density ranges from 0.025 to 0.029 g/cm^3. Higher expansions, desirable from a cost standpoint, can be obtained with lower concentrations of the viscosity-builder, such as 0.1 or 0.2%, but very little thickening of the formulation takes place at these concentrations; that is, a significant viscosity-building effect is

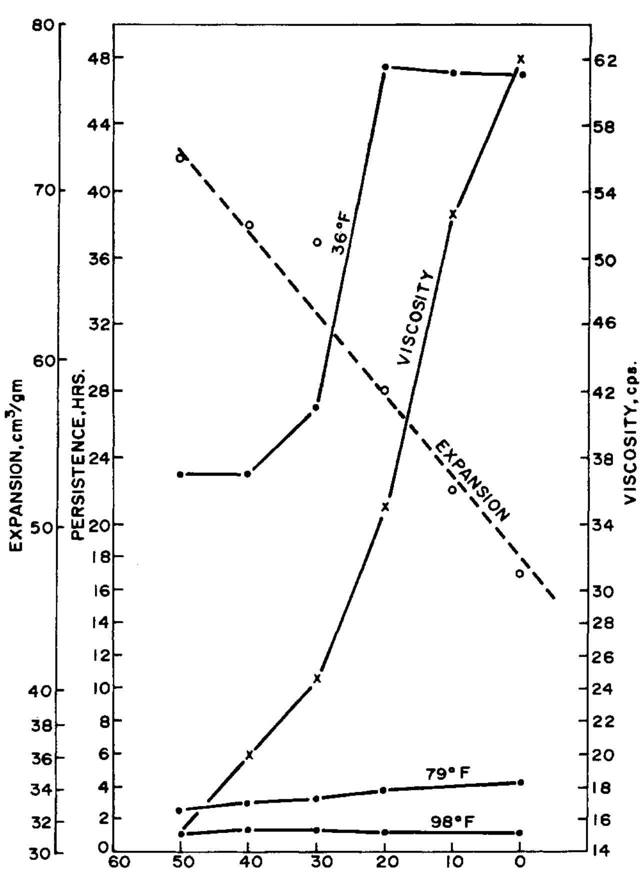

Figure 4. Effect of dilution of the formulation on foam characteristics.

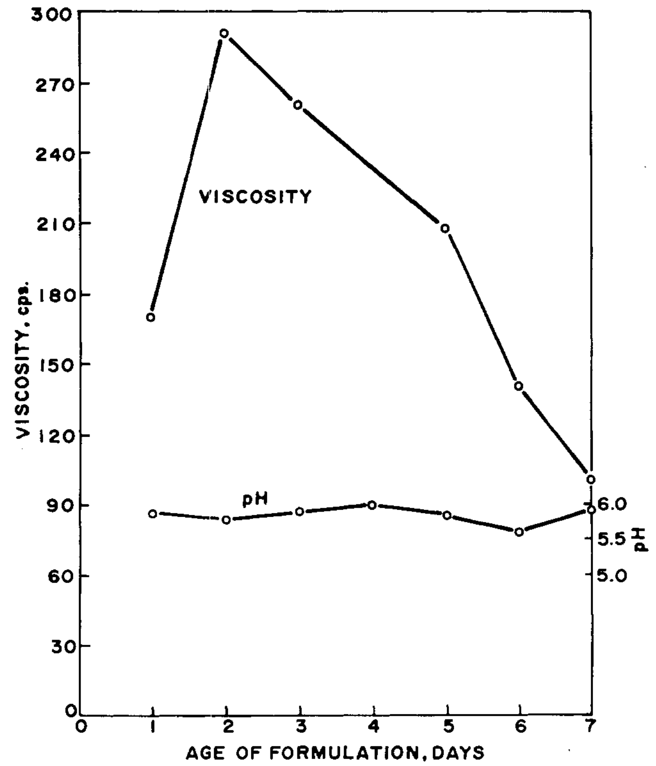

Figure 6. Effect of aging of standard formulation on pH and viscosity.

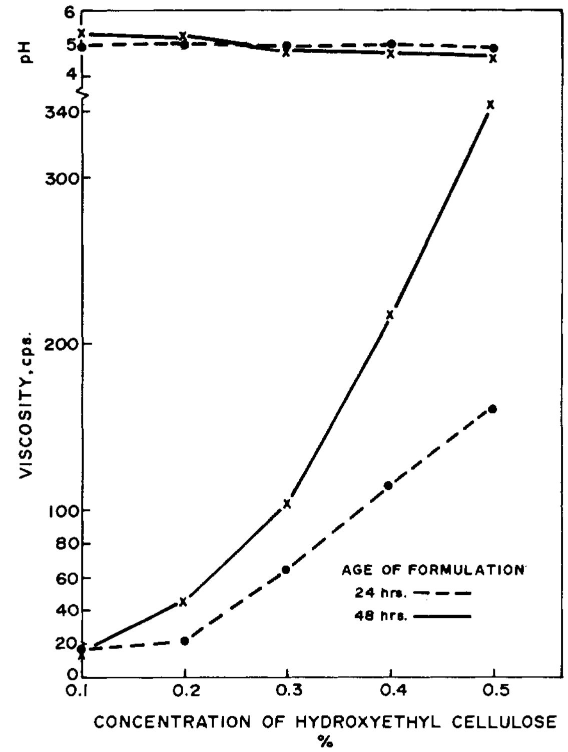

Figure 8. Effect of concentration of hydroxyethyl cellulose on pH and viscosity of foam formulation.

not obtained, as is shown in Figure 8. Viscosity increases were greater in the older formulation.

Persistence of these foams generated from 24-hr and 48-hr formulations at three temperature levels is shown in Figure 9. In foams generated from 24-hr formulations and stored at 36°F, there occurred a dip in the curve shown at the 0.2% concentration of Natrosol. This dip occurred because of the relatively high persistence obtained for the 0.1% concentration. Except for this inconsistency, an otherwise orderly increase in persistence with increase in concentration of Natrosol was obtained at 36°F for foams generated from formulations 24 and 48 hr old. This lends credence to the belief that an excessively high persistence had been obtained for the 24-hr-old formulation contain-

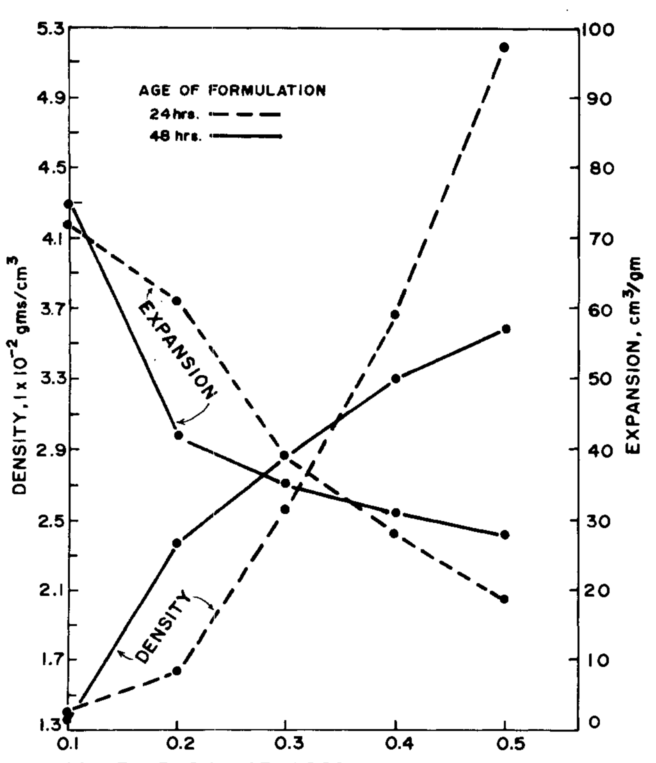

Figure 7. Effect of concentration of hydroxyethyl cellulose on density and expansion of foams.

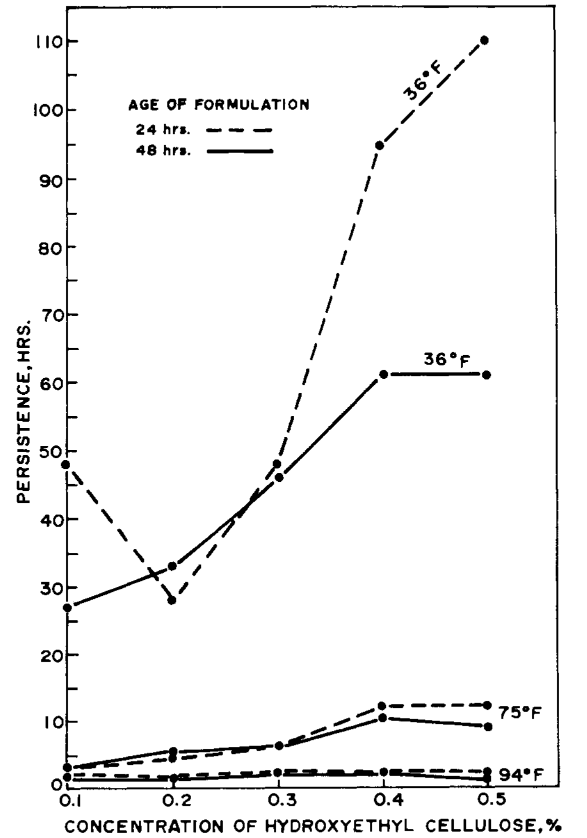

Figure 9. Effect of concentration of hydroxyethyl cellulose on foam persistence at 36, 75, and 94°F.

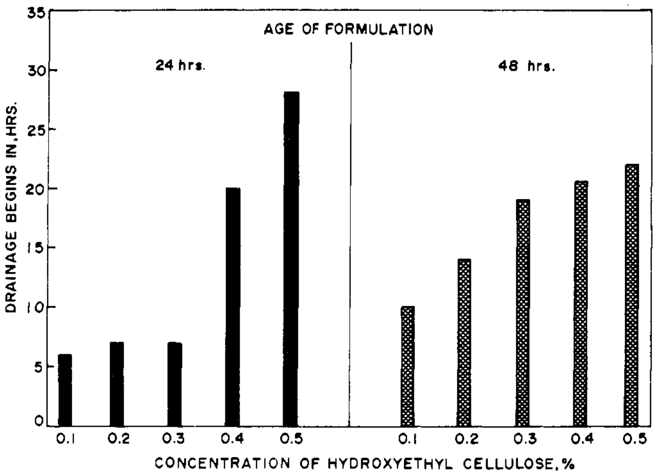

Figure 10. Relationship between concentration of hydroxyethyl cellulose and initial drainage from foams stored at 36°F.

ing 0.1% of the viscosity-builder. Foams observed at 75 and 94°F showed no great differences. It is seen that 9 to 12 hr of longevity at viscosity-builder concentrations of 0.4 and 0.5% were obtained at a temperature of 75°F. These are both interesting if one is operating under weather conditions that do not exceed 75°F. For these conditions, a foam might be tailored to yield such a persistence. In doing so, it must be remembered that expansion (volume of foam generated from a specific weight of formulation) must be sacrificed (Figure 7).

There were no essential differences in drainage rates of foams made from 24-hr and 48-hr formulations. However, differences did occur in the time elapsed before drainage began when foams were stored at 36°F (Figure 10). At 36°F an increasing significant postponement of the initial drainage occurred from 6 to 28 hr (24 hr old) and 10 to 22 hr (48 hr old), with a corresponding increase in concentration of Natrosol. A weighted combination of the favorable results shown in Figures 7 through 10 leads to the selection of a 0.3% concentration of Natrosol as the optimum concentration of the viscosity-builder for the standard formulation.

Effect of Concentration of Surfactant. Concentration of Hi-X was varied from 0.3 to 1.9% in increments of

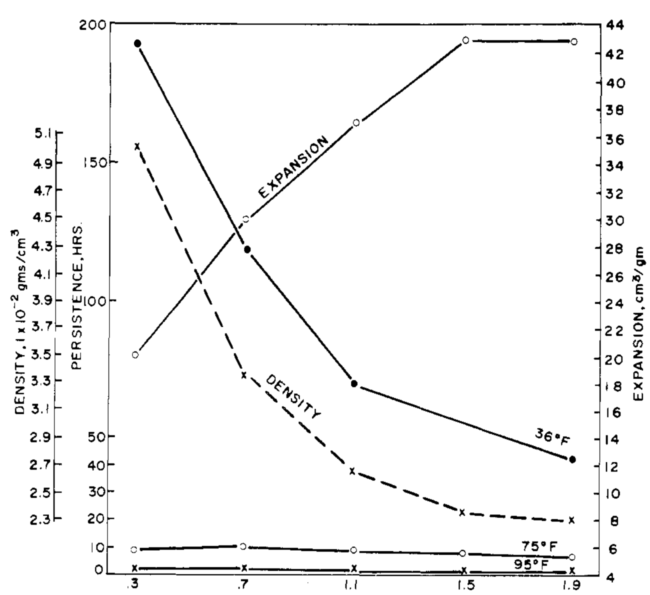

Figure 11. Effect of concentration of surfactant on foam characteristics.

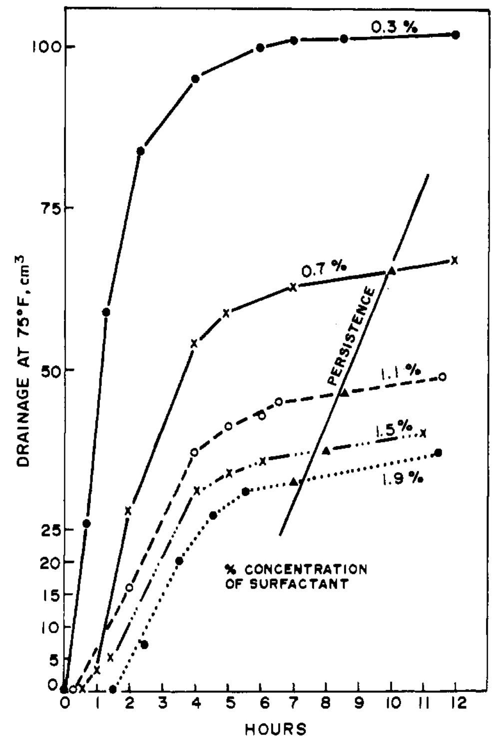

Figure 12. Drainage of foams at 75°F as affected by concentration of surfactant. Triangles represent persistence values.

0.4%. Effects of this variable are shown in Figure 11. Persistence at 36°F and density curves paralleled each other and decreased as the concentration of Hi-X increased. Greatest persistence, more than 192 hr at 36°F, was obtained at 0.3%, where density was also highest and expansion was lowest. Extended persistence at such a low concentration of surfactant was confirmed by replicating four times. Expansion increased with an increase in concentration of Hi-X up to 1.5%, where it leveled off. Persistence ranged from 7 to 10 hr at 75°F, peaking at 10 hr and 0.7% concentration of Hi-X. Persistence at 95°F was poor, remaining under 2 hr for all concentrations.

Drainage at 75°F gave an orderly family of curves, decreasing as the concentration of Hi-X increased (Figure 12). Persistence of the foams (designated by triangles and denoting the time at which 33% of the original foam volume remained) increased with a decrease in concentration of surfactant from 1.9 to 0.7%. At 36°F drainage was greatest at the 0.3% concentration and least for the longest period of time (94 hr) at 0.7% concentration (Figure 13). On a relatively short-term basis (26 hr), drainage was least at the 1.1% concentration.

The best skin or film covering the foam was obtained at a 0.3% concentration of surfactant. Quality of the skins deteriorated as the concentration increased at all temperature levels. Although all foams stored at 36°F had a complete paper-like skin, the lowest concentration, 0.3%, had the strongest or most durable skin. Skins are important because they protect the foams from strong winds and have a tendency to anchor the foam in place around a plant or on either side of a row.

Considering all of the above effects of surfactant concentration, it is believed that Hi-X should be used at a concentration of 0.7% for the best all-around results. If the foam is to be used at freezing or near-freezing temperatures, concentration of Hi-X can be reduced to 0.3%, taking advantage of its long-lasting persistence (8 days).

DISCUSSION

Feed-grade whey solids were found suitable stabilizers for the chemifoams. Persistence of these foams could be enhanced by 200 (36°F) to 400% (73°F) on addition of protein in the form of animal glue. Persistence of the foams generally increased in an orderly manner as the temperature was lowered. This is in accord with a property known as rigidity or gelation of some proteins, the animal glue in this case. Gelation of glue takes place at refrigerator temperatures, as with edible gelatin. If the foam is stored at 36°F, it can be expected to gel and drainage will be postponed for hours (Figure 13) or even days because the moisture remains entrapped in the gel. Adjustment of pH of the formulation in the range of 3 to 9 units did not increase persistence of the foams over that of the standard formulation at pH 6, where no adjustment is needed (Figure 3). Adjustment of pH of the standard formulation on the acid side produced only low to medium persistence, whereas a high persistence was experienced with the original standard formulation at pH 6 and when the pH was adjusted to 9. It is believed that adjustments in pH to 7 and 8 were not affecting the glue because of the heterogeneity of the mixture and the range of pH's of the individual components, which contributed in some way to preventing the glue from being affected until a pH of 9 had been achieved. Optimum results were obtained when the formulation was diluted with 20% water (Figure 4). This meant balancing expansion, density, viscosity, and persistence to arrive at optimum conditions. If a foam is to be used at temperatures ranging from 65 to 75°F, the liquid formulation should not be more than 2 days old (Figures 5 and 6). However, if the foam is to be used at or near freezing temperatures, the liquid formulation may be as much as 3 days old. It is believed that hydrolytic breakdown of the glue is the important factor in aging of the foam precursor. Optimum concentration of the viscosity-builder, Natrosol, was found to be 0.3% (Figures 7–10). Concentration of the surfactant, Hi-X, should range from 0.7 to 1.1% for persistent foams (Figure 11). When these convergent factors are considered and costs are calculated according to Braud and Chesness (1968), foam generation of the preferred formulation for application to row crops in bands 1-in. deep, 10 in. wide, and spaced 42 in. would cost approximately $9.32 per acre-in. This compares favorably to formulations containing edible gelatin plus Hi-X, starch phosphate plus Hi-X, and starch phosphate, Hi-X and Natrosol, costs of which were calculated at $24.30, $25.00, and $19.70, respectively, per acre-in.

Since aging has an effect on the formulation and the foam generated therefrom, there is a possibility that a reaction is occurring among the components of the mixture. No attempt has been made yet to determine its nature.

It would probably not be required in the South, but a foam having a longevity of 8 days near or at freezing temperatures is possible from the preferred formulation by reducing the concentration of surfactant to 0.3% (Figure 11). This foam is protected by a strong, complete, paper-thin skin, highly desirable under adverse weather conditions. Such a foam would give adequate insulation from wind and low temperatures.

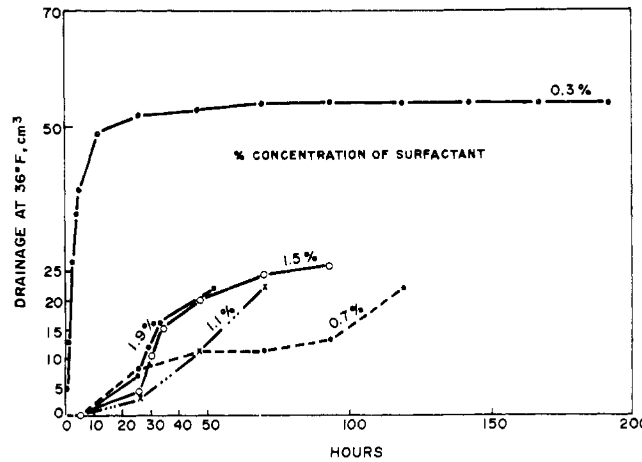

Figure 13. Drainage of foams at 36°F as affected by concentration of surfactant.

Protection from low temperatures is not the only use of a foam made from the preferred formulation. Persistence of the foams in a range of temperatures from 50 or 60°F to 80°F was of sufficient duration, 18 to 24 hr, to make them usable as carriers for herbicides, fungicides, and defoliants and as foam covers after soil fumigation with gaseous fumigants. Unless the temperature exceeds 80°F by 10–20°F, these foams may also be used as farm markers.

ACKNOWLEDGMENT

The authors gratefully acknowledge the assistance of the Eastern Regional Research Laboratory (ERRL), ARS, USDA, for their cooperation in sending samples of whey solids and animal glues and especially I. A. Wolff, who suggested the use of whey solids, and of Hercules, Inc., for furnishing the viscosity-builder, Natrosol.

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It is not the policy of the Department to recommend the products of one company over those of any others engaged in the same business.