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Pilot-Plant Production, Tempering, and Evaluation of Global Edible Spreads from Vegetable Oils[1]

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A RECENT paper describes the discovery of an edible-spread composition of wide plastic range to meet the needs of Armed Forces stationed under widely differing climatic conditions (5). The composition contains vegetable oil, distilled glycerol monostearate, and other ingredients. It may be spread on bread or crackers at low temperatures and still does not melt or separate at high temperatures encountered in the tropics.

After a basic formula had been found for preparing a spread with desirable plasticity characteristics, several problems arose in the further development of the product. For example, when prepared in the laboratory, the spread usually developed small crystals which imparted an undesirable grainy "mouth feel." It was also difficult to prepare, in the laboratory, uniform material in sufficient quantities for organoleptic evaluation. Preliminary studies in the pilot plant in which a small Votator[3] unit was used for rapidly solidifying the spread demonstrated that an improved and more uniform product could be made.

Previous conceptions of a spread with suitable mouth "getaway" prescribed that it must melt readily and almost completely in the mouth (1). Undoubtedly the most difficult problem to be solved in preparing a suitable high-melting edible spread was to discover a formulation which would be emulsified readily when eaten with other food without leaving a pasty or waxy residue in the mouth. A description of pilot-plant techniques and of recent advances made toward improving getaway and physical stability of the global-spread composition is the subject of this paper.

Pilot-Plant Production

The first paper describing a global spread contained a table giving a typical formulation (4). Subsequent refinements and modifications now justify further comments regarding the composition. Since the product contains no aqueous phase, the salt is prepared by grinding sodium chloride (C.P.) in a nickel-plated hammer mill. Approximately 99% of the ground salt will pass through a 325-mesh screen.

[1] Presented at the American Oil Chemists' Society meeting, Chicago, Ill., Nov. 2-4, 1953.

[2] One of the Branches of the Agricultural Research Service, U. S. Department of Agriculture.

[3] The mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

Originally, the addition of 0.2 part of butter-flavor concentrate per hundred parts of total glycerides was recommended. However subsequent evaluations indicate that a spread containing 0.04 part of this concentrate is preferred by the taste panel at the Northern Utilization Research Branch. In place of 0.2 part of butter-color concentrate, 0.0035 part of dry carotene has been used satisfactorily. Composition of the spread currently being produced is given in Table I.

TABLE I

Composition of Global Spread

Ingredients	Parts
Vegetable salad oil	83
Distilled monostearate	17
Soybean phosphatides (oil-free)	0.2
Salt	2.5
Carotene	0.0035
Butter-flavor concentrate	0.04
Propyl gallate	0.01
Citric acid	0.005
Vitamin A	1,650,000 units/100 lbs.
Vitamin D	330,000 units/100 lbs.

The equipment used for preparing global spread in the pilot plant, as shown in Figure 1, consists of a jacketed mixing kettle, a means of introducing nitrogen into the material as it is drawn to a gear pump, and a Votator cooling unit. The cylinder of the Votator is constructed of nickel, and other equipment is stainless steel.

The constituent materials for preparing a spread are added to the mixing kettle, heated to about 170°F. (77°C.) and then pumped to the Votator from which the spread issues in a fluid stream. Fifteen to 20 volume-percentage nitrogen based on the oil volume is introduced ahead of the pump. There is no pressure valve on the discharge side of the Votator, and the material flows readily and evenly into containers. The mass rather quickly "sets up" to a firm consistency.

A mixing kettle with a capacity of 2 gal. is chosen since only about 10 lbs. of material are required for various analyses and taste-panel evaluations. The Votator chamber has a diameter of 3 in. and 90 sq. in. of heat-transfer area. A gear pump with a capacity of 12 gal. per hour is used. Cooling water at 60°F. (16°C.) is adequate for chilling the spread.

Difficulties were encountered in incorporating the salt in the spread. Attempts were made to mix salt

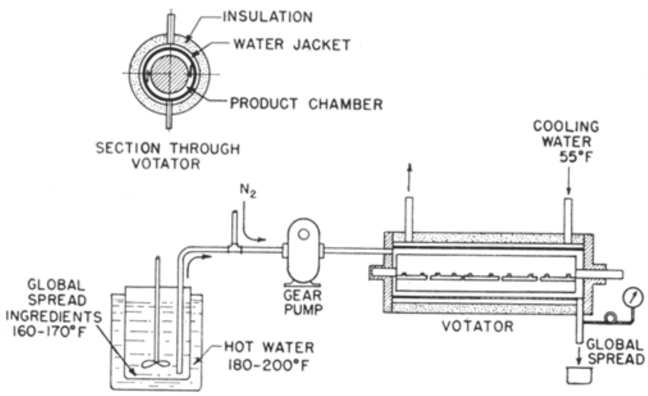

FIG. 1. Diagram of equipment for producing global spread in the pilot plant.

in the plasticized spread after it had passed through the Votator. However this resulted in the inclusion of more air than was consistent with good appearance and texture. An improved product was obtained if the salt was kept dispersed in the molten mixture until crystallization occurred in the Votator. This was accomplished by providing sufficient agitation of the molten mixture and by using small pipe. The gear pump has handled this material satisfactorily for over a year although some abrasion of the pump has occurred.

After the first trials by this method the product had a gray-green color. When plastic blades were substituted for the stainless-steel scraper blades, the discoloration was eliminated.

Table II gives the operating conditions for a typical pilot-plant run. It may be calculated from these

TABLE II

Operation Conditions for Preparing Global Spread in a Pilot-Plant Unit

Temperature of cooling water into Votator	64.7°F. (18°C.)
Temperature of cooling water out of Votator	66.5°F. (19°C.)
Temperature of global spread into Votator	161°F. (72°C.)
Temperature of global spread out of Votator	75°F. (24°C.)
Flow rate of cooling water	2,540 lbs./hr.
Flow of global spread	70 lbs./hr.
Speed of Votator rotor	670 r.p.m.
Heat transfer area	89.2 sq. in.

data that the heat-transfer coefficient in the Votator is nearly 200 b.t.u./hr./sq. ft./deg. F. Because of the small-scale operation the Votator was operated at only about 20% of its maximum capacity. The change in enthalpy for a typical global spread between 161° and 75°F. (72° and 24°C.) is 65.3 b.t.u./lb. under conditions of rapid chilling. From a consideration of specific and latent heats this indicates that less than 15% of the oil component crystallizes.

Effect of Tempering and Composition on Plasticity and Organoleptic Characteristics

Spreads of the composition reported previously (5) had a satisfactory plastic range and consistency as indicated by penetration data and initially were acceptable organoleptically. On storage at room temperature however they developed an undesirable waxy mouth-feel and unsatisfactory getaway characteristics.

The lowering of the monoglyceride content produced a softer spread, and a significant improvement in general acceptability resulted. However the necessary reduction in monoglyceride content was so great that the spread lacked sufficient firmness and tended to separate or "bleed" oil at a temperature of 112°F. (44°C.). Accordingly attempts have been made a) to improve the getaway, b) to stabilize the spread against bleeding of free oil at elevated temperatures, c) to insure freedom from graininess, and d) to prevent changes in the physical characteristics. It was found that the addition of phosphatides along with tempering had a pronounced beneficial effect on the plastic characteristics of the spread. Marked improvement was found in the getaway of the spread containing phosphatides after a relatively short tempering at an elevated temperature. Of equal importance was the improvement in softness at relatively high monoglyceride levels and the retention of plasticity when stored at room temperature for long periods of time. A study of the physical changes which occur during tempering is reported in a separate publication (3) where crystal habit, microscopic, X-ray, and micropenetration data are discussed.

Changes in characteristics of spreads of various formulations with time and tempering conditions were evaluated by penetration measurements, organoleptic tests, and other suitable chemical and physical measurements. Samples for organoleptic study were collected in No. 2, flat, 10-oz. cans which were sealed under air, nitrogen, or carbon dioxide immediately after preparation. Those for macropenetration study were collected in metal ointment cans 1⅛ in. deep and 3 in. in diameter and subsequently leveled with a straight edge and weighed for density determinations. Other jar samples were taken for determination of salt content by chemical analysis and gas content by a physical method. Initial penetration measurements were made 20 to 30 min. after production of the spread, with an A.S.T.M. Precision Universal Penetrometer equipped with a standard-size and standard-shape grease-penetration cone constructed from aluminum so that the total weight was 26 g. Samples having a penetration in excess of 6 mm. were found to spread easily. However the desirable range was between 15 mm. to 20 mm.

Preliminary tempering tests were conducted in air-circulating ovens adjusted at 90°, 112°, and 130°F. (32°, 44°, and 54°C.), respectively. Penetration measurements were made after 1½ hrs., 3 hrs., and 24 hrs. at these temperatures. After completion of the tempering period, samples were removed from the oven and periodic determinations of penetration were continued at 75°F. (24°C.) storage until the samples apparently assumed constant firmness, usually in about 100 hrs. An untempered or control sample was held at 75°F. (24°C.), and penetration values were determined for a similar length of time. Final penetration values reported in the tables were obtained as described above.

Organoleptic and penetration data led to the following preliminary conclusions:

1. Tempering was beneficial to all phosphatide-containing spreads and for nonphosphatide-containing spreads at low monoglyceride levels of 13 to 15%. However, at a level of 30% in the absence of phosphatides, no improvement of the tempered over the untempered samples with respect to penetration was noted.
2. Tempering at 112°F. (44°C.) and at 130°F. (54°C.) was more rapid than at 90°F. (32°C.).
3. Tempering at 112°F. (44°C.) for 24 hrs. was preferable to 3 hrs.

The results indicated that tempering phenomena were occurring under the three experimental temperatures selected but at different rates. Since global spreads contain more than 80% liquid vegetable oil, tempering should be carried out under mild conditions so as to minimize the development of undesirable flavors. Consideration of factors such as the manufacture and the subsequent storage conditions before use led to the selection of 112°F.(44°C.) as the temperature for studying the effect of tempering and formulation on general acceptance.

Tempering Pattern of Spreads Not Containing Phosphatides

Table III lists pertinent penetration data and getaway scores for spreads of increasing monoglyceride content. In view of the satisfactory getaway of the 13% monoglyceride spread it would appear that it was of a satisfactory composition. As pointed out previously, this spread lacked firmness and tended to bleed oil at 112°F.(44°C.). Moreover, when the product was stored for approximately 10 days at 112°F. (44°C.), the penetration decreased and the getaway score was adversely affected. The purpose of the storage test was to determine if the canned spread was stable when stored for extended periods at elevated temperatures such as might be encountered in use.

TABLE III

The Effect of Monoglyceride Content on Penetration and "Getaway" of Tempered Spreads

Monoglyceride, %	Untempered penetration at 75°F.(24°C.)		Penetration at 112°F. (44°C.) after			Tempered 24 hr. at 112°F.(44°C.)	
	Initial	Final[a]	1 hr.	3 hr.	24 hr.	Final[a] penetration at 75°F. (24°C.)	"Getaway" score[b]
Without phosphatide							
13	29.7	16.3	21.1	22.8	25.3	18.5	1.0
15	24.7	14.2	21.5	22.0	21.0	16.2	1.8
18	22.6	10.3	19.5	18.4	17.9	13.3	2.6
30	18.1	4.5	11.2	7.4	6.8	4.5	3.5
With 0.2% phosphatide							
17	24.5	8.5	23.5	23.0	24.0	17.5	1.2
18	15.1	8.9	18.9	20.7	22.1	18.1	1.4
22	16.1	6.9	15.9	17.1	17.1	14.1	2.6

[a] Value when penetration became constant.
[b] A score of 0.0 indicates "getaway" characteristics comparable to butter; a score of 4.0 indicates a very poor "getaway."

The influence of monoglyceride content is shown by the increase in initial and final firmness of the untempered control as well as the final firmness of the tempered sample. Samples containing over 15% monoglyceride became progressively firmer during their retention in the tempering oven.

The difference in penetration values between the final untempered and the tempered product has been shown to be a good measure of the extent of tempering, which indicated that spreads made with 13, 15, and 18% monoglycerides were benefited by tempering. These findings are shown graphically in Figures 2 and 3 and lead to the conclusion that nonphosphatide spreads are best prepared with the minimum amount of monoglyceride consistent with sufficient firmness at room temperature. This amount is between 13 and 15%. Tempering of nonphosphatide spreads appeared to alter the crystal habit or structure of the spread, and at suitable monoglyceride levels tempering resulted in a softer product of improved getaway characteristics.

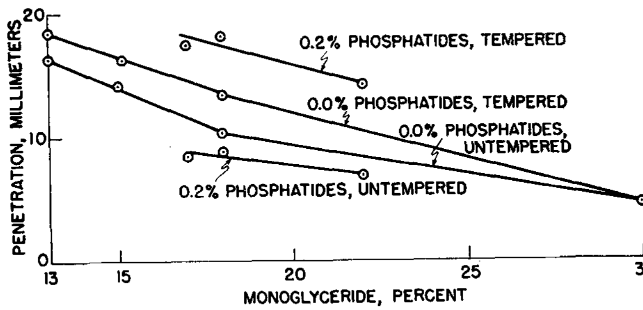

FIG. 2. Effect of monoglyceride content on penetration of spreads at 75°F. (24°C.).

Tempering Characteristics of Spreads Containing Phosphatides

Tempering studies at 112°F.(44°C.) have been conducted on spreads containing 0.2% of oil-free phosphatides, 17% monoglycerides, and other ingredients so as to establish the optimum treatment time. Samples from several typical preparations were placed in the tempering oven and subsequently removed at intervals and held at 75°F.(24°C.) until penetration measurements were seemingly constant.

The data in Table IV show that minimum getaway score and maximum penetration fall within the 12- to 48-hr. tempering period and that the undesirable increase in firmness caused an increase in getaway score after about two months of storage at 112°F. (44°C.). This finding strongly indicates that whereas optimum tempering was beneficial, prolonged storage at 112°F.(44°C.) subsequently reduced the general acceptance of the product. In contrast, nonphosphatide-containing spreads develop these undesirable characteristics in about one-fourth the time.

Having chosen a suitable tempering time and temperature, it was deemed necessary to determine the optimum amount of phosphatide for incorporation in an 18% monoglyceride spread. Table V lists the pertinent data, which show that the initial penetration measurements as well as final values for the untempered products decreased with increasing quantity of phosphatide. Values obtained at 112°F.(44°C.) exhibit a small progressive decrease at low phosphatide levels and an increase at the higher levels. Table III showed that increasing mongolycerides promoted

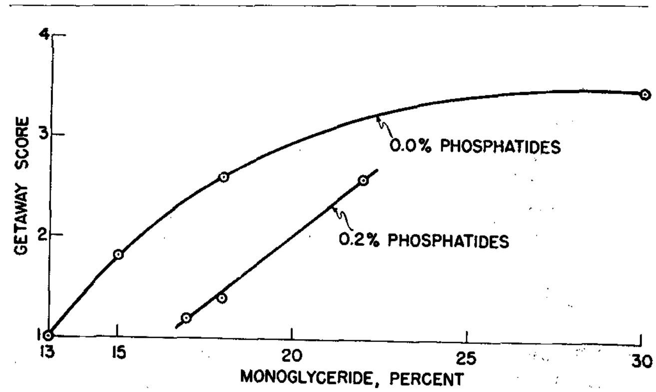

FIG. 3. Effect of monoglyceride content on getaway of tempered spreads.

TABLE IV

Tempering Spreads Containing 17% Monoglyceride and 0.2% Phosphatide

Tempering time at 112°F. (44°C.)	"Getaway" score	Penetration
Days		*mm.*
0	2.7	15
½	1.4	17
1	1.3	17
1½	1.6	16
2	1.6	17
28	1.6	14
56	2.1	11

hardness, indicating that phosphatides and monoglycerides act in opposition in determining the final penetration.

Getaway scores improved and softness increased with increasing phosphatide percentage and leveled off at about 0.2% for tempered spreads as shown in Figure 4.

TABLE V

The Effect of Phosphatide Concentration on Penetration and "Getaway" of Spreads Containing 18% Monoglyceride

Phosphatide, %	Untempered penetration at 75°F. (24°C).		Penetration at 112°F. (44°C.) after			Tempered 24 hrs. at 112°F. (44°C.)	
	Initial	Final[a]	1 hr.	3 hr.	24 hr.	Final[a] penetration at 75°F. (24°C.)	"Getaway" score
0	22.6	10.3	19.5	18.0	17.9	13.3	2.6
0.01	19.8	10.1	20.3	19.3	18.1	13.1	2.4
0.1	17.9	10.1	20.0	20.0	20.1	15.9	2.0
0.2	15.1	8.9	18.9	20.7	22.1	17.4	1.2
0.4	9.3	8.7	20.7	22.5	23.4	16.5	1.5

[a] Value after penetration became constant.

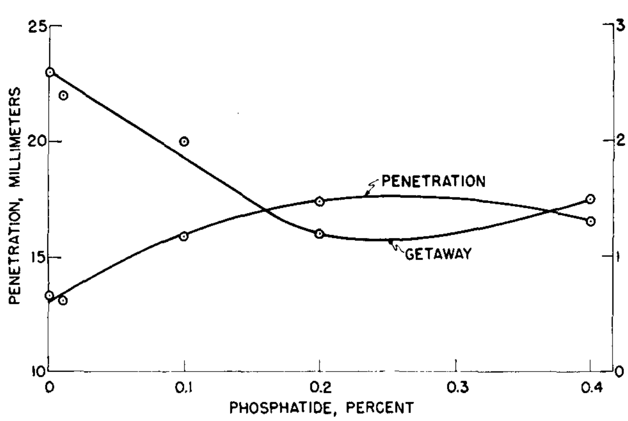

FIG. 4. Effect of phosphatide content on penetration at 75° F. (24°C.) and getaway of tempered spreads containing 18% monoglycerides.

In view of the superiority of those spreads containing 0.2% phosphatides at the 18% monoglyceride level, it was logical to investigate their effect at other monoglyceride levels. The results in Table III show that phosphatides were effective in softening a 22% monoglyceride spread. They should also be effective at lower monoglyceride levels, but this point has not as yet been established.

Phosphatide-containing spreads usually were made with various commercial vegetable oils. Others have been made in which hydrogenated vegetable oils, margarine oil, and butter oil replaced a part of either the soybean or cottonseed oil. It has been established that amounts of the hydrogenated vegetable, margarine, and butter oils, approximating about 10%, may be included without detracting from the general acceptability of the product. However their incorporation decreased the spreadibility at temperatures below 32°F. (0°C.).

Acceptance and Organoleptic Evaluation

Changes in composition and processing techniques were followed closely by organoleptic evaluation. A 9-point balanced scoring system (4) was adopted with 0 as the ideal score. Thus four positive units and four negative units were used to indicate the superiority or inferiority of the product over that of the ideal. Scores inferior to the ideal are considered negative, and those superior are positive. To avoid the confusion of carrying a negative sign, all scores are reported as positive figures since all scores to date have been negative. A few panel evaluations have been conducted for general over-all acceptance of the product, using both a simple ranking test and the above-scoring system.

The taste panel was initially trained on high-scoring butter and margarine for both getaway and flavor. Getaway scores for various spreads used throughout this work are shown in Table VI. The improvement in getaway of the present products over the early global-spread compositions which rated scores of 3 to 4 is apparent. The values shown in the table indicate that the product is clearly in the range of margarine and superior to that of the conventional Army spread. Penetration values are also included, and a study on 86 spreads of varying composition indicated a correlation coefficient of 0.5 between getaway and penetration. Although a higher value might be expected, it was observed that, in comparisons between spreads with a definite difference in penetration values, the softest spread usually received the better getaway score. Factors besides tempering which affect the getaway scores are the monoglyceride content and the level of phosphatide addition. The effect of composition and tempering time on getaway scores was discussed in their respective sections under tempering.

TABLE VI

Average "Getaway" Scores for Spreads

Spread	No. of panel evaluations	Average score	Penetration 75°F. (24°C.)	Penetration 34°F. (1°C.)
Margarine A	8	0.85	14.0	2.0
Margarine B	6	1.4	9.3	2.0
Margarine C	8	1.0	16.1	1.7
Global spread A	5	1.4	14.6	10.1
Global spread B	8	0.83	17.4	13.9
Global spread C	5	1.1	17.4	14.0
Army margarine	4	3.0	11.7	1.5

Indications by independent evaluation groups are that the general acceptability of global spread is very good. Tests performed at the Quartermaster Food and Container Institute have shown that a sample of global spread examined compared favorably with margarine, particularly the military type (6). Evaluations by the Northern Utilization Research Branch panel are shown in Table VII. The selections of the 15 tasters are significant, indicating that the panel is able to distinguish differences, by ranking, which are not significant differences when the samples are scored. Thus when scored for acceptance, a highly significant

TABLE VII
Comparable Acceptability Tests for Global Spreads

Spread	Acceptability by ranking			"Getaway" score
	1st place	2nd place	3rd place	
Army margarine	0	2	2	3.1
Global spread	1	12	13	1.4
Margarine	14	1	0	0.7
	Acceptability by scoring			
Army margarine		3.3	....	
Global spread A		1.2	1.3	
Global spread B		1.4	1.5	
Margarine		....	1.1	
Significance		high	none	

difference is shown between the Army margarine and the global spread, but a significant difference does not exist between global spreads and regular margarine. The acceptability shown by ranking and the getaway score are indicative of the quality of the product.

Extensive work on flavor and flavor stability was not undertaken until compositional studies and processing conditions had reached a favorable position in the course of developmental work. Synthetic flavors have not been entirely satisfactory, and present compositions include 0.04% of Butr-trate 2XW, a product described as a special-distilled, butter-flavor concentrate. Taste-panel studies indicate that a very minimum of flavor is desirable. This agrees with the trend that less and less flavor is desired in the staple food items which are consumed in large quantities in the daily diet. Flavor, flavor stability, and oxidative stability of the global spreads under various storage conditions are the current problems under investigation.

Summary

Global spread of improved plasticity characteristics has now been produced on a pilot-plant scale. Chilling equipment similar to that used in margarine production was slightly modified; and because of the scale of operation, throughput was reduced to one-fifth the rated capacity.

Global spread produced by the formulation previously described suffered the defect of becoming progressively firmer when stored, consequently both the "mouth feel" and general acceptability were impaired. This deficiency was largely overcome by tempering at 112°F.(44°C.) for 24 hrs. in the presence of 0.2% oil-free soybean phosphatides which softens the spread and slows the hardening process which occurs during prolonged storage at elevated temperatures. Spreads currently being produced compare favorably with commercial margarines in general acceptability when evaluated at room temperature and are superior in plasticity characteristics when compared at low and high temperatures.

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Measurement of Oxidation in Dried Milk Products With Thiobarbituric Acid[1,2]

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THE need for objective tests for measuring flavor deterioration in food products and the use of 2-thiobarbituric acid as a reagent for such methods were discussed in a previous paper (8).

The chemical test used most often for measuring oxidative changes in fat-containing food products is the determination of peroxides. The absolute peroxide value may sometimes have no significance in relation to flavor score because of the simultaneous formation and decomposition of the peroxides (2). A peroxide value of approximately 2.0 m.e./kg. of fat is normally considered to be low for most fats, and they are generally free of oxidative off-flavors at this level of peroxide value. The reverted flavor of soybean oil is detectable at a peroxide value of 2.5 (4). Milk powder also undergoes serious flavor changes of an oxidized nature by the time the fat reaches a peroxide value of 2.0 (6).

Patton and Kurtz (7) and Dunkley and Jennings (3) have ably reviewed the literature leading to the application of 2-thiobarbituric acid as an agent for the detection of oxidized fat in dairy products. Procedures have been described for the application of the thiobarbituric acid (TBA) test to fluid milk (3), butterfat (7, 8), and to cheese, butter, and milk powder (1).

The procedure to be described offers certain advantages over previously published methods. Freedom from the physical interference of proteins as well as freedom from turbidity in the test solution is accomplished by steam distillation of an acidified slurry of milk powder. Less complicated apparatus than that of the only other published procedure for milk powder (1) is required. Furthermore no organic solvent, such as isoamyl alcohol or pyridine, is neces-

[1] Presented at the 27th Fall Meeting of the American Oil Chemists' Society, Chicago, Ill., Nov. 2-5, 1953.

[2] This paper reports research undertaken by the Quartermaster Food and Container Institute for the Armed Forces and has been assigned No. 452 in the series of papers approved for application. The views and conclusions contained in this paper are those of the authors. They are not to be construed as necessarily reflecting the views or indorsement of the Department of Defense.