Determination of Emulsion Stability by Microwave Irradiation[1]

ABSTRACT

The stability of emulsions has been determined by a microwave irradiation method. After this treatment, the surface temperature of the emulsion tends to be greatest and the temperature difference between the surface and the bottom of the emulsion smallest for the more stable emulsions. These results have been explained in terms of the nature of microwave heating and a partially destabilized emulsion.

INTRODUCTION

The determination of emulsion stability has evolved via several approaches: observation of the extent of creaming or coalescence by measuring the phase volumes either directly with time or following accelerated stresses of centrifugation; heating or freeze thaw cycles; and measurement of droplet size either directly via optical microscopy or indirectly by reflectance measurements (the smaller the droplets, the greater the reflectance and the more stable the emulsion) (1,2).

If emulsion destabilization is visualized as shown in Figure 1, examination of the partially destabilized emulsions 1c and 1d may provide stability information before actual coalescence occurs. The upper portions of 1c and 1d are relatively oil rich compared to the lower part (assuming an oil in water emulsion with the density of water greater than the density of oil). Analytical methods which could

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[1]Presented at the AOCS Meeting, September 1973, Chicago.

quantitatively measure the proportion of oil in this upper region of the emulsion would provide a guide to its stability.

Measurement of the dielectric constant of the upper part of emulsions has been correlated with stability and droplet size since the rate of creaming follows Stokes law $V = 2/9[r^2(d_1 - d_2)g/\eta]$ (3-5), where V = rate of creaming, r = droplet radius, d_1, d_2 = phase densities, g = gravity, and η = viscosity.

The present method involves microwave irradiation. In its simplest form, microwaves create heat by passing through a body and causing the molecules within to align with the field. This alignment causes intermolecular friction, thus heat. The amount of heat created is proportional to the dielectric constant of the medium (6). The dielectric constant of water is 80, while that of oil is $\sim$3 (7). When partially destabilized emulsions are heated by microwaves, the surface temperature of the more stable emulsions (less oil rich) should be higher than that of the more destabilized emulsions (more oil rich). Also, the temperature difference between the surface and the bottom should be less for the more stable emulsions than for the less stable emulsions, since distribution is more homogeneous in the former.

EXPERIMENTAL PROCEDURES

Emulsions generally were prepared by taking 30 g (41%) oil, 43.3 g water, and 0.33 g (0.45% based upon total emulsion) emulsifier, mixing them hot, and then homogenizing with a laboratory size piston homogenizer using

TABLE I

Emulsifier Composition vs. Microwave and Normal Stability
Measurements for a Series of Corn Oil-in-Water Emulsions

Emulsifier HLB value[a]	Coalesced oil (g/10g emulsion)	Surface temperature (F)[b]	Temperature difference (F)[c]
4.7	2.50	95	−28.4
6	2.40	110	− 8.1
7	1.37	115.5	
8	0.27	121	− 2.2
9	0.06	125.5	
10	0.06	129	+ 3.4
11	0.20	129	
12	0.13	123.5	+ 0.5
13	0.57	124	
14.9	0.61		+ 1.9

[a]Integral emulsifier hydrophile-lipophile balance (HLB) values were obtained using sorbitan monostearate HLB 4.7 (A) and polyoxyethylene (20) sorbitan monostearate HLB 14.9 (B) mixtures according to the relationship HLB mixture AB = 4.7·% A/100 + 14.9·% B/100.

[b]Measured with a thermocouple after 15 sec microwave heating. For calibration, pure water 130 F, pure corn oil 95 F.

[c]Surface temperature minus bottom temperature after 15 sec microwave heating. For calibration purposes, value for pure water is +28, thus the +3.4 value is the smallest difference, since it comes closest to the water value.
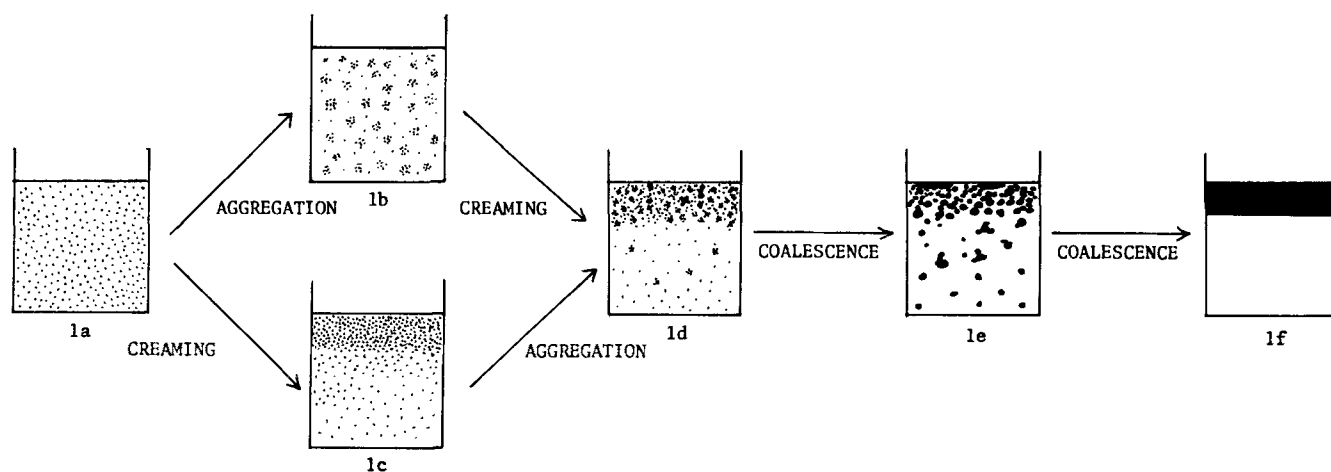
FIG. 1. Simplified emulsion destabilization scheme.

~3000 psi. Resultant average droplet size varied with emulsifier but was 0.5μ-10μ. The hydrophile-lipophile balance (HLB) values of emulsifier mixtures used were determined from the equation $HLB_{(mix)} = \%\,A\cdot HLB_A/100 + \%\,B\cdot HLB_B/100$, where A and B were sorbitan monostearate HLB 4.7 and polyoxyethylene (20) sorbitan monostearate HLB 14.9 (8). Emulsion stability was determined by measurement of coalesced oil after centrifugation at 37,000 x G for 10 min expressed in terms of g coalesced oil/10 g emulsion. Centrifugation of stable emulsions was preceded by heating the emulsion at 160 F for 1-3 hr or subjecting it to one or more freeze-thaw cycles.

Microwave heating of the emulsions was carried out using a Litton model 900 microwave oven of 1 kw rf power. Emulsion samples (10 g) in test tubes were supported in a styrofoam base on the floor of the oven. A 15 sec irradiation resulted in elevated temperatures without causing boiling out. Corrections for nonuniformity of field were obtained by heating pure water or oil in each of the tube positions and noting final temperature variations due to positioning. Surface and bottom emulsion temperatures were determined with a copper constantin thermocouple attached to a 5 mv strip chart recorder for rapid and permanent readings.

RESULTS AND DISCUSSION

Qualitatively, it was found that, after microwave heating of 15 sec duration, the surface temperature of emulsions, which visibly were destabilized (creamed or coalesced, see Fig. 1, 1d, e, or f), was lower than for stable emulsions, as expected. The technique was extended or refined to evaluate quantitatively the present or future stability of emulsions which appeared to be stable. This was accomplished by preparing a series of fairly similar emulsions using the same oil but varying the ratio of two emulsifiers, sorbitan monostearate HLB 4.7 and polyoxyethylene (20) sorbitan monostearate HLB 14.9, in the emulsion according to the resultant HLB value of the mixture. Table I shows the results for corn oil, one of several oils used.

After microwave heating, the emulsions exhibiting the highest surface temperatures yielded the lowest amount of coalesced oil after centrifugation. The emulsifier mixture HLB value in this emulsion agrees closely with the required HLB value of 9 for corn oil (8). Similar trends also were observed for a series of different oils provided that the emulsions were destabilized sufficiently. This would imply, referring to Figure 1, that assessing the extent of formation

of 1d, which may not be destabilized visibly, allows the prediction of the relative rate of formation of 1e and 1f. Obviously differences in aggregation as in 1b will not be observed since no creaming occurs, thus no oil rich surface region; some destabilization of the emulsion must have occurred for the present method to differentiate between emulsions.

The effect of irradiation time variations may be lessened by observing the temperature difference between the surface and the bottom of the irradiated emulsions. The use of a thin wire thermocouple allowed measurements without disturbing the gradient layering. The smallest temperature differences considering the water calibration were observed for the most stable emulsions as judged by coalesced oil measurements as shown in Table I.

The results obtained for simple model emulsions containing water-oil-emulsifier demonstrate that emulsion stability may be determined using microwave heating methods. Ordinary heating would be ineffective, since it occurs slowly from outside in allowing for equilibration of the layers. The effect of other ingredients present in practical emulsions, such as gums, proteins, solids, as well as emulsion type, i.e. oil-in-water or water-in-oil, warrant further investigation. A correlation of microwave heating characteristics vs. long term storage stability would be especially useful.

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