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Determination of Emulsion Stability Index in W/O Emulsion

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유중수적형 유화계내에서의 유화안정지수 산출법의 확립

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Abstracts : To evaluate the emulsion stability indices of W/O emulsion system, we developed the simple and sensitive "VOLUMETRIC METHOD". This technique involved the first step of homogenizing the milk fat-water systems with Ultra-turrax T25, then the volume of the added water phase was measured immediately. After quiescent incubation in test tubes at room temperature for a desired storage time, the bottom volume of the separated water layer was measured. And then "emulsion stability index(ESI)" was calculated by the following equation : $ESI=(1-V_s/V_a)\times100$, where V_a means the volume of the added water in the W/O emulsion and V_s represents the volume of the separated water in the W/O emulsion for a desired storage time. The emulsion stability indices of W/O emulsion system at sorbitan trioleate, span 60, and tween 20 were 95.4±1.8, 56.1±2.8, and 41.6±2.2, respectively. Furthermore, the differences between "VOLUMETRIC METHOD" and "Titus et al method" were less than 5.0 of ESI Value.

요약 : 유중수적형 유화계의 유화안정지수를 산출하는 방법을 확립하기 위하여 간편하면서도 예민한 "부피 직접 측정법"을 개발하였는 바, 먼저 강력한 혼합기의 일종인 Ultra-turrax T25를 이용하여 유지방, 유화제 및 물로 구성되면서 유지방이 물보다 많이 함유되는 유중수적형의 유화계를 형성시킨 후 상온에서 시간이 경과함에 따라 분리되어지는 수분층의 부피를 측정하였으며, 유화안정지수는 $(1-V_s/V_a)\times100$(단, V_a는 유화계내에 함유되어 있는 총 수분의 부피이며, V_s는 시간이 경과함에 따라 유화계로부터 분리되어지는 수분층의 부피를 의미한다)의 계산식에 대입하여 산출하였다. 한편, sorbitan trioleate, span 60 및 tween 20을 첨가하여 유중수적형 유화계를 형성시킨 후 90분이 경과한 다음 "부피 직접 측정법"에 의하여 유화안정지수를 산출하였는 바, 각각 95.4±1.8, 56.1±2.8 및 41.6±2.2였으며, 이러한 값은 유화안정지수의 산출방법으로서 일반적으로 사용하고 있는 "Titus의 방법"과 비교해 볼 때 5.0 이내의 차이— 유화안정지수가 평형에 도달하였을 때의 차이 값— 를 나타냄을 알 수 있었다.

Key words : emulsion stability index, analytical method, W/O emulsion

1. INTRODUCTION

Many natural and formulated dairy products are composed of emulsion system. The emulsion stability in the food emulsion system is a very important criteria to improve the quality of emulsion product or to develope new product. Emulsion stability may be influenced by the numerous experimental variables including type of emulsion system and emulsifier, amount of emulsifier, and operation conditions of emulsification. To obtain the stable emulsion systems, simple and rapid method for evaluating emulsion stability in emulsion system is needed.

Methods for determining the stability of emulsion system have been reviewed by several workers.[1,3,4] Titus et al. (1968) used the extraction procedure characterized by fat extraction with ether in the bottom half of the emulsion and calculation dividing the percentage of fat in the final sample by the percentage of fat in the inital sample.[2] Petrowski has also suggested the method for determination of emulsion stability by microwave irradiation technique in 1974.[4] These procedures have the merits of high sensitivity and reliability. On the other hand, they have also the troublesomes of complexity in the way of processing sample and the occurrence of experimental error caused by high sensitivity. Therefore, food researchers have generally relied on observation of the extent of creaming or on measurement of droplet size owing to its simplicity. But these simple techniques have dismerits of low sensitivity and reliability.

Therefore, the purpose of this study was to develop a simple and sensitive procedure for the determination of emulsion stability index in W/O emulsion

2. MATERIALS AND METHODS

2.1. Materials and General Procedures

The anhydrous milk fat having a melting temperature of 37℃ was obtained from a single source to ensure a uniform supply. The mixing ratio of milk fat, emulsifier, and distilled water was 80 :1.5 :20(w/w/w). Emulsions were formed with Ultra-turrax T25 (JANKE & KUNKEL Co.) at 5,000 rpm and 37℃.

The apparatus of emulsion test tube was designed to hold a series of 80 mL glass tubes(250 mm long × 25 mm outside diameter). A masked light source and colorized milk fat were used for sharp separation of the cream line which was measured by means of a ruled glass plate at the front.

2.2. Determination of ESI by "VOLUMETRIC METHOD"

The emulsion stability indices of W/O emulsion system were evaluated by "VOLUMETRIC METHOD"(*Figure* 1). This technique involved the first step of homogenizing the milk fat-water systems with Ultra-turrax T25(refer to *Figure* 2). And after quiescent incubation in test tubes at room temprature for a desired storage time, the bottom volume of the separated water layer measured. And then "emulsion stability index(ESI)" was calculated by the following equation ; $ESI=(1-V_s/V_a)\times100$, where V_a means the volume of the added water in the W/O emulsion and V_s represents the volume of the

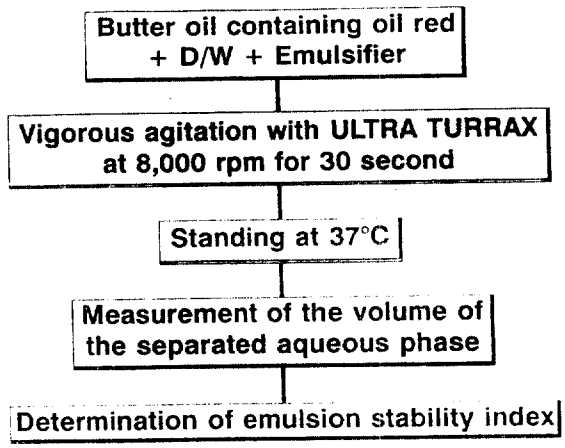

Figure 1. Procedure for determination of "Emulsion Stability Index(ESI)".

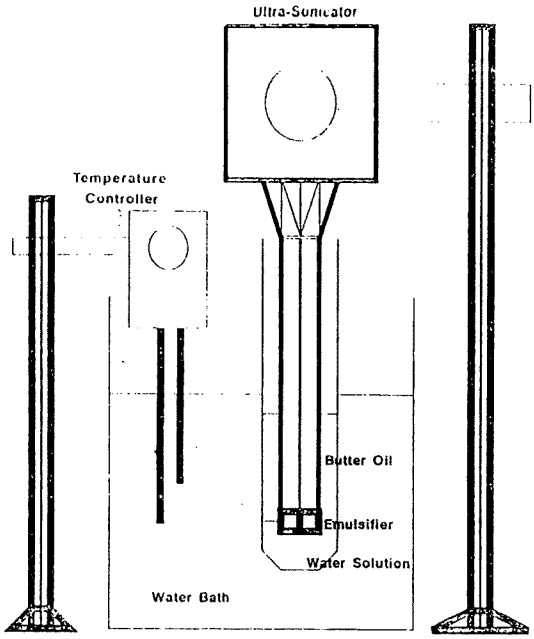

Figure 2. Schematic diagram of emulsification tool

separated water in the W/O emulsion for a desired storage time.

2.3. Determination of ESI by "Titus et al method"

This technique also involved the first step of homogenizing the milk fat-water systems with Ultra-turrax T25, then sampling the emulsion immediately. After quiescent incubation in test tubes at room temperature for a desired storage time, a second sample representing the bottom 20%(w/w) of the emulsion was obtained. The fat was extracted with ether and weighed, then the two fat percentage measurements were used to calculate a stability index. This "emulsion stability index(ESI)" was calculated by the following equation : $ESI=(F_s/F_i)\times100$, where F_i means the fat content of the W/O emulsion in the initial time and F_s represents the fat content of the separated phase in the bottom 20% (w/w) of the W/O emulsion for a desired storage time.

3. RESULTS AND DISCUSSION

To evaluate the "VOLUMETRIC METHOD", it was compared with "Titus et al. method" for the determination of the emulsion stability indices of sorbitan trioleate(HLB value of 1.8), span 60(HLB value of 5.3), and tween 20 (HLB value of 16.7), respectively.

Figure 3(a) showed that sorbitan trioleate had a high emulsifying activity in this W/O emulsion system and emulsion stability indices at final equilibrium state were the values of 95.4±1.8 evaluated by "VOLUMETRIC METHOD" and 95.0±2.0 by "Titus et al. method", respectively. The difference in the values of ESI between "VOLUMETRIC METHOD" and "Titus et al. method" was less than 5.0 of ESI value.

Figure 3(b) also showed that the W/O emulsion with span 60 (HLB value of 5.3) was roughly stable

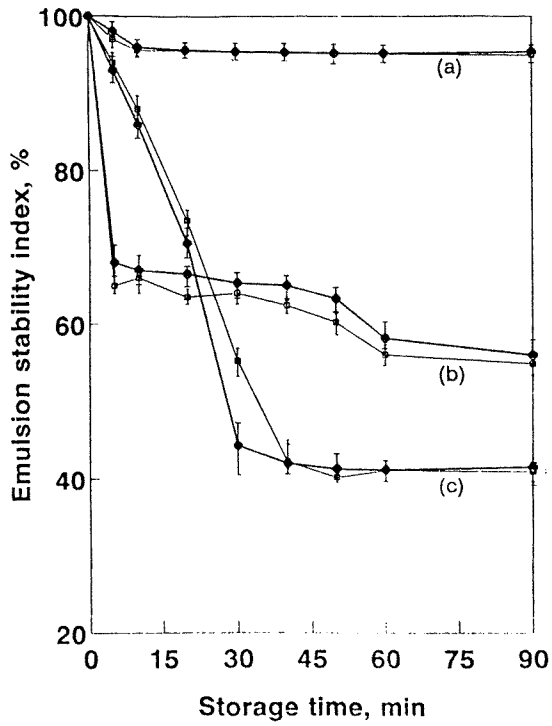

Figure 3. Comparision of ESI obtained from "VOLUMETRIC METHOD(-●-)" with "Titus et al method(-□-)" in the case of 2.0%(w/w) of sorbitan trioleate (a), span 60 (b), and tween 20 (c).

and emulsion stability indices at stable equilibrium state were the values of 56.1±2.6 described by our "VOLUMETRIC METHOD" and 55.2±2.6 by "Titus et al. method", respectively. The difference between the two methods was less than 5.0 of ESI value.

Finally, the rationality of "VOLUMETRIC METHOD" was examined in the range of low stability of emulsion (less than the ESI value of 50) using tween 20. As shown in *Figure* 3(c), milk fat-tween 20-water emulsion system had low value of ESI and their emulsion stability indices at final equilibrium state were 14.6±2.2 expressed by our "VOLUMETRIC METHOD" and 41.2±2.6 by "Titus et al. method", respectively. The result also showed that the difference between the two methods was less than 5.0 of ESI value.

Based on the above-mentioned results, emulsion stability index of W/O emulsion can be determined sensitively by the "VOLUMETRIC METHOD". Furthermore, "VOLUMETRIC METHOD" is a simple technique for determining emulsion stability index of W/O emulsion.

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