



Optimization of Manufacturing Conditions for Improving Storage Stability of Coffee-Supplemented Milk Beverage Using Response Surface Methodology

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Abstract

This study aimed at optimizing the manufacturing conditions of a milk beverage supplemented with coffee, and monitoring its physicochemical and sensory properties during storage. Raw milk, skim milk powder, coffee extract, and emulsifiers were used to manufacture the beverage. Two sucrose fatty acid esters, F110 and F160, were identified as suitable emulsifiers. The optimum conditions for the beverage manufacture, which can satisfy two conditions at the same time, determined by response surface methodology (RSM), were 5,000 rpm primary homogenization speed and 0.207% sucrose fatty acid emulsifier addition. The particle size and zeta-potential of the beverage under the optimum condition were 190.1 nm and -25.94±0.06 mV, respectively. In comparison study between F110 added group (GF110) and F160 added group (GF160) during storage, all samples maintained its pH around 6.6 to 6.7, and there was no significant difference ($p<0.05$). In addition, GF110 showed significantly higher zeta-potential than GF160 ($p<0.05$). The particle size of GF110 and GF160 were approximately 190.1 and 223.1 nm, respectively at initial. However, size distribution of the GF160 tended to increase during storage. Moreover, increase of the particle size in GF160 was observed in microphotographs of it during storage. The L^* values gradually decreased within all groups, whereas the a^* and b^* values did not show significant variations ($p<0.05$). Compared with GF160, bitterness, floating cream, and rancid flavor were more pronounced in the GF110. Based on the result obtained from the present study, it appears that the sucrose fatty acid ester F110 is more suitable emulsifier when it comes to manufacturing this beverage than the F160, and also contributes to extending product shelf-life.

Keywords coffee, emulsification, shelf-life, RSM

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Introduction

Milk has been considered as an essential element of the diet for human since the agricultural revolution (Huth *et al.*, 2006). As civilizations evolved, many types of dairy products, including cheese, yogurt, ice cream, and processed milk have been developed to satisfy consumers' needs. Nowadays, milk beverages supplemented with coffee have become favored milk products, with a global consumption constantly increasing.

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Coffee is one of the most popular beverages and principal commodities in the world (Feria-Morales, 2002; Geel *et al.*, 2005). World consumption of coffee annually approximately 1.9% increased over the last 50 years (Dmowski and Dabrowska, 2014). In addition, recently, the consumption of ready-to-drink (RTD) type milk beverages supplemented with coffee has increased markedly in certain countries, such as Korea, Japan, Taiwan, among others, since there are no traditional and specific images of coffee as a freshly brewed beverage. RTD type milk beverages supplemented with coffee are cheaper, more convenient to transport, and easier to store at refrigeration temperatures than fresh brewed coffee beverages, which also explains their popularity (Hsu and Hung, 2005; Petracco, 2001).

Lately, Muslim regions, including Arab and Southeast Asia have emerged as new big markets, and many companies are showing interest toward exporting their products to these parts of the world. However, most of these Muslim regions are located in subtropical or tropical zones, and have poor food distribution infrastructures. Milk products are easily deteriorated under hot conditions and should be distributed refrigerated to prevent quality degradation events, such as lipid oxidation and precipitation, among others. The shelf-life of food is the maximum length of the time between the dates of manufacture and sale ensuring the preservation of a satisfactory quality (Gyesley, 1991; Kilcast and Subramaniam, 2000). Therefore, studying the shelf-life extension allowing an effective and secure exportation of this type of products to these regions is necessary. More particularly, lipid oxidation and precipitation become serious issues during the storage or distribution of RTD type beverages due to the presence of various ingredients, and therefore, establishing suitable emulsification conditions is paramount toward solving this problem. This study was conducted to determine the optimum emulsification and homogenization conditions during the manufacture of milk beverages supplemented with coffee, in order to extend their shelf-life.

Materials and Methods

Materials

The materials used for the milk beverage were provided by Seoul F&B Co., Ltd. (Korea). Emulsifiers such as Almax 9280, Almax 9080, and sucrose fatty acid esters F160 and F110 were contributed by Ilshin Wells Co., Ltd. (Korea).

Manufacture of coffee beverage

The description of the beverage components provided by Seoul F&B Co., Ltd. (Korea) are listed in Table 1. The materials were primary homogenized with a high speed homogenizer (Ultra-turrex T25, IKA Werke GmbH, Germany) and secondary homogenized with a high pressure homogenizer (Picomax MN250A, Micronox Co., Korea).

Experimental design

Response surface methodology (RSM) was used to optimize the operation parameters during the manufacturing process of the coffee beverage, such as speed of primary homogenization (X_1) and amount of emulsifier (X_2) to be added, which ranged from 5,000 to 15,000 rpm and 0.1-0.3%, respectively (Table 2). The testing of both variables was designed by face central composite design (FCCD) approach. Thirteen experimental settings were generated and analyzed with MINITAB release 16 (Minitab Inc., State college, USA) and Design expert release 7.0 (Stat-Ease Inc., USA). The regression model equation for particle size (Y_1) and zeta-potential (Y_2) could be speculated as follows:

$$Y_n = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_{12}x_1x_2 + \beta_{11}x_1^2 + \beta_{22}x_2^2$$

Where, a dependent variable Y expresses response (particle size or zeta-potential), β_0 , β_1 and β_2 express constant coefficients of the intercept, linear, quadratic and interaction terms, respectively. This response surface model was also used to estimate the result by isoresponse 3-D surface plots. 3-D surface plot is the reflection of the response sur-

Table 1. Ingredients of the milk beverage supplemented with coffee

Ingredient	(%)
Raw milk	30.000
Coffee extract	30.000
Sucrose	5.500
NaHCO ₃	0.040
Etc.	34.46
Total	100.000

Table 2. Coded level for independent variables used in the experimental design for milk beverages supplemented coffee

Variables	Coded X_i	Coded level		
		-1	0	+1
Speed of primary homogenization (rpm)	X_1	5,000	10,000	15,000
Concentration of emulsifier (%)	X_2	0.1	0.2	0.3

face in a three dimensional plane.

Analysis of particle size and zeta-potential

Particle sizes ($D_{4,3}$) and zeta-potentials of coffee beverages with various emulsifiers were determined using a Zetasizer (Zen 3600, Malvern Instruments Ltd., UK). To measure particle sizes and zeta-potentials, the samples were diluted 1,000-fold with deionized water. All samples were triplicated at a constant temperature of 25°C.

Microscopic observation

The microstructures of 100-fold diluted samples were magnified 1,000-fold under a light microscope (DM 2500, Leica microsystems GmbH, Germany).

pH

The pH values of each beverage sample were measured at 4°C using a pH meter (PP-15, Sartorius AG, Germany).

Determination of peroxide value (PV)

In the present study, PV was measured by the method of AOAC (1990) with slight modifications. It was measured as follows: 1-5 g samples mixed with 25 mL chloroform/acetic acid mixture (3:2), and then 1 mL potassium iodide added into the mixture and reacted in darkness. After 10 min, 30 mL distilled water and 1 mL starch solution were poured in the sample. Free iodine was titrated with 0.01 N sodium thiosulfate. The PV was indicated in milli-equivalents of active oxygen per one kilogram (mEq active O_2 /Kg) of processed shrimp sample, as determined using Eq. (1):

$$PV = \frac{(a-b) \times f}{M} \times 10$$

Where, a : volume of 0.01 N sodium thiosulfate (mL), b : volume of 0.01 N sodium thiosulfate in blank (mL), M : mass of sample and f : factor of sodium thiosulfate solution.

Color measurement

Color values of each beverage sample were measured using a colorimeter (CT-340, Minolta, Japan), which was calibrated using original standard plate with its original value ($X=97.83$, $Y=81.58$, $Z=91.51$). Analyzed L^* , a^* , and b^* values denote lightness, redness, and yellowness, respectively, in artificial light. These were measured in triplicates.

Sensory evaluation

Ten panelists (5 males and 5 females) were selected from graduate school students at Kangwon National University in Korea. They were all non-smokers, healthy, and their average age was 26 years old (range 23-39 years old). The samples were stored at 4°C for 12 d, dispensed into 50 mL clear plastic cups with lids, labeled with random 4-digit codes, and served with a glass of water for cleansing of the mouth between each sample. Brownness, coffee flavor, milk flavor, rancid flavor, sweetness, bitterness, and overall acceptability were evaluated on a 5-point scale (1: none, 3: moderate, 5: very strong).

Statistical analysis

Analysis of variance (ANOVA) was carried out to analyze the differences between groups. Data was indicated as mean±SD and statistical significant differences were determined at $p<0.05$. All statistical analyses were performed with SAS 9.4 (SAS Institute Inc., USA).

Results and Discussion

Selection of emulsifier

To select a suitable emulsifier, various milk beverages were manufactured with the same amount of 4 types of emulsifier, and their particle sizes as well as zeta-potentials were measured. As shown in Fig. 1, the particle sizes of most samples were below 200 nm except for the sucrose fatty acid F160 added group, and there was no statistical significance among the samples ($p<0.05$). The zeta-potential results (Fig. 2) showed no statistical significance among all samples ($p<0.05$). Sucrose fatty acid esters F110 and F160 were in powder form, and Almax 9280 and 9080 were in gel form with high viscosities. Gel-type emulsifiers with high viscosity are often considered as being inconvenient for manufacturing procedures and therefore, the powder-type emulsifiers were selected. It is known that the hydrophilic lipophilic balance (HLB) of sucrose fatty acid ester F110 is 11, while that of F160 is 15-16. Emulsifiers with high HLB are good for particle size reduction (Sjöström *et al.*, 1993) and possess good emulsifying properties in O/W emulsion (Sjöström *et al.*, 1993; Yin and Walker, 1994). However, the F110 showed better results than the F160. Potential interactions with proteins and carbohydrates, among others, could be occurring in the case of this beverage, and therefore, further studies need to be carried out to investigate these phenomena.

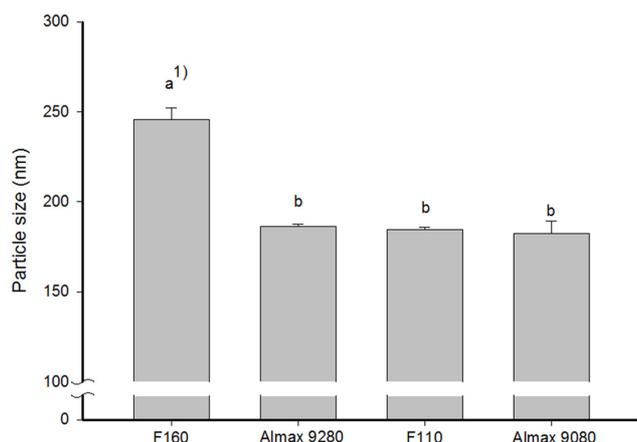


Fig. 1. Measurement of particle size to determine the optimum type of emulsifier for manufacturing of milk beverages supplemented with coffee. ¹⁾Values with different letters are significant at $p < 0.05$ by Duncan's multiple range test.

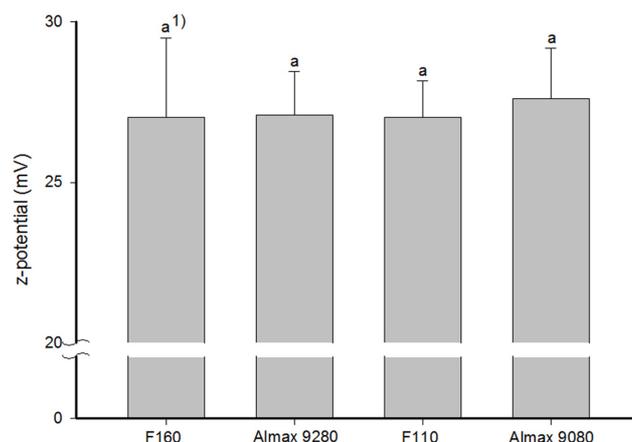


Fig. 2. Measurement of zeta-potential to determine the optimum type of emulsifier for manufacturing of milk beverages supplemented with coffee. ¹⁾Values with different letters are significant at $p < 0.05$ by Duncan's multiple range test.

Optimization of manufacturing conditions

A total of 13 experiments were conducted (Table 3) using primary homogenization speed (X_1) and amount of emulsifier (X_2) as independent variables and particle size (Y_1) as well as zeta-potential (Y_2) as responses by FCCD, and the result are indicated in Table 4. The mathematical expression of the relationship between particle size and variables X_1 (primary homogenization speed) and X_2 (amount of emulsifier) are given as coded factors.

$$Y_1 = 197.295 + 0.272X_1 + 5.906X_2 - 9.801X_2^2 + 10.467X_1X_2 \quad (1)$$

The regression of zeta-potential from variables X_1 and

X_2 are also given below as coded factors.

$$Y_2 = 31.223 - 1.406X_1 + 1.639X_2 - 2.618X_2^2 + 1.450X_1X_2 \quad (2)$$

A positive coefficient denotes a synergistic effect, on the other hand, a negative one denotes an antithetical effect (Simmons, 1978). In Eq. (1), X_1 (primary homogenization speed) and X_2 (amount of emulsifier) showed a positive relationship with particle size, indicating that an increase in the particle size would occur with an increase of these factors. In Eq. (2), X_1 showed a negative relationship, whereas X_2 displayed a positive relationship with the zeta-potential.

Table 3. Central composite design and response variables for milk beverage supplemented with coffee

Run number	Coded variable ¹⁾		Process variable		Response variables ²⁾	
	X_1	X_2	X_1	X_2	Y_1	Y_2
1	-1	0	5,000	0.2	217.867	36.1000
2	0	0	10,000	0.2	260.500	35.6000
3	0	0	10,000	0.2	186.433	30.8300
4	+1	+1	15,000	0.3	219.767	32.5666
5	0	0	10,000	0.2	181.933	29.0667
6	-1	+1	5,000	0.3	178.267	29.9667
7	-1	-1	5,000	0.1	179.900	27.5000
8	0	0	10,000	0.2	175.633	29.6000
9	+1	-1	15,000	0.1	179.533	24.3000
10	+1	0	15,000	0.2	178.367	28.2667
11	0	+1	10,000	0.3	182.167	28.2000
12	0	0	10,000	0.2	180.333	29.1000
13	0	-1	10,000	0.1	185.333	29.1000

¹⁾ X_1 and X_2 are speed of primary homogenization and the concentration of emulsifier (%), respectively.

²⁾ Y_1 and Y_2 are the particle sizes and zeta-potentials of milk beverages, respectively.

Table 4. Values of the regression coefficients calculated for milk beverage supplemented with coffee

Response variables ¹⁾	Independent variable	Regression coefficient	Standard error coefficient	t-value	Significance (p)
Y_1	Constant	201.539	17.29	1.1658	0.000
	Linear				
	X_1	-12.267	17.00	-0.722	0.494
	X_2	5.906	17.00	0.347	0.738
	Quadratic				
	X_1^2	22.763	25.05	0.909	0.394
	X_2^2	-29.220	25.05	-1.166	0.282
Interaction					
	$X_1 X_2$	10.467	20.82	0.503	0.631
Y_2	Constant	31.0339	1.253	24.767	0.000
	Linear				
	X_1	-1.4056	1.232	-1.141	0.291
	X_2	1.6389	1.232	1.330	0.225
	Quadratic				
	X_1^2	0.6630	1.816	0.365	0.726
	X_2^2	-2.8704	1.816	-1.581	0.158
Interaction					
	$X_1 X_2$	1.4500	1.509	0.961	0.369

¹⁾ Y_1 and Y_2 are the particle sizes and zeta-potentials of milk beverages, respectively.

The 3-D response surfaces and contour plots for particle sizes and zeta-potentials in the coffee beverages are shown in Figs. 3(A) and 3(B), respectively. As both the

homogenization speed and the amount of emulsifier increased, the droplet size and zeta-potential decreased. This result is similar to that obtained in the report by Qian and

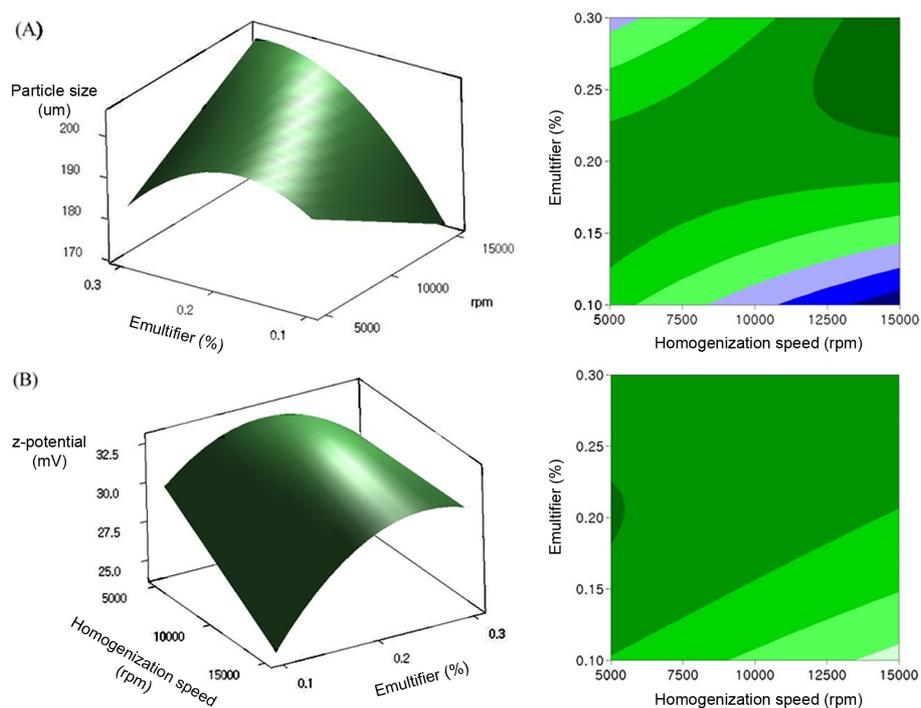


Fig. 3. The 3-D surface and contour plots for milk beverages supplemented with coffee. (A) Surface plot of particle size vs. emulsifier concentration (%) and primary homogenization speed (rpm); (B) surface plot of zeta-potential vs. primary homogenization speed (rpm) and emulsifier concentration (%).

McClements (2011). They manufactured nanoemulsions with corn oil using various concentrations of emulsifier, and analyzed their average droplet diameter. As a result, a decrease in the average droplet diameter of the nanoemulsions was observed with increasing amounts of emulsifier. It is thought that higher amounts of emulsifier should allow a faster coverage of the surfaces of any new droplet formed during homogenization (Qian and McClements, 2011), which also correlates with the results obtained by Nakauma *et al.* (2008). They analyzed changes in zeta-potentials and average diameters of O/W emulsions elaborated with triglyceride oil and various concentrations of emulsifier. In their study, it was revealed that as the amount of emulsifier increased, both the zeta-potential and the average diameter decreased.

Optimization curves help to establish the factor setting that allows optimizing a single or set of responses. It is useful tool for determining the operating conditions that will lead to a desired response (Ahn *et al.*, 2013). In the present study, the objective was to investigate the optimum conditions to produce a milk beverage supplemented with coffee, using two responses. As shown in Fig. 4, as primary homogenization speed increased, particle size increased, and zeta-potential decreased. In addition, as the amount of emulsifier increase by 0.2071%, the zeta-potential increased. Therefore, the best combination of factors to achieve the desired responses was found to be the primary homogenization speed (5,000 rpm) and the amount of emulsifier (0.2071%). Combining those factors yielded the minimum particle size and maximum zeta-potential at the same time when manufacturing the beverage. Finally, the particle size and zeta-potential of the beverage under the optimum condition were 190.1 nm and -25.94 ± 0.06 mV, respectively (Fig. 5). In this result, particle size was nearly agreed with the estimation by RSM. However, the zeta-potential value was slightly smaller than the estimation by RSM.

Storage stability of coffee beverages

pH

The changes in pH of the coffee beverage stored at 4°C for 12 d are depicted in Fig. 6. The pH values of all samples were almost consistent during 12 d of storage. The pH of the sucrose fatty acid F160 added group (GF160) and the sucrose fatty acid F110 added group (GF110) ranged from 6.59 to 6.63 and 6.56 to 6.65, respectively. In the present study, the pH of these beverages were higher

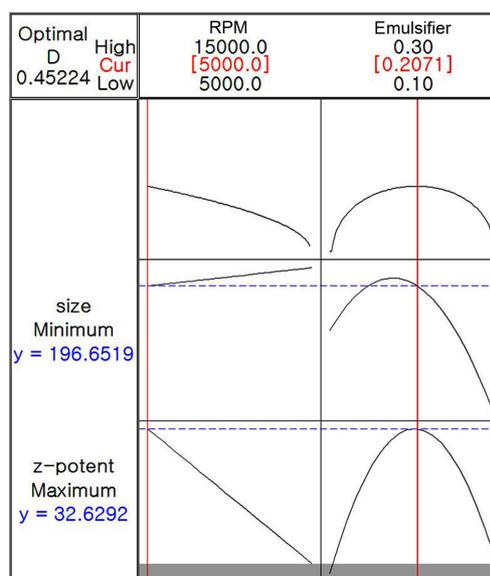


Fig. 4. Optimization curve process for milk beverages supplemented with coffee.

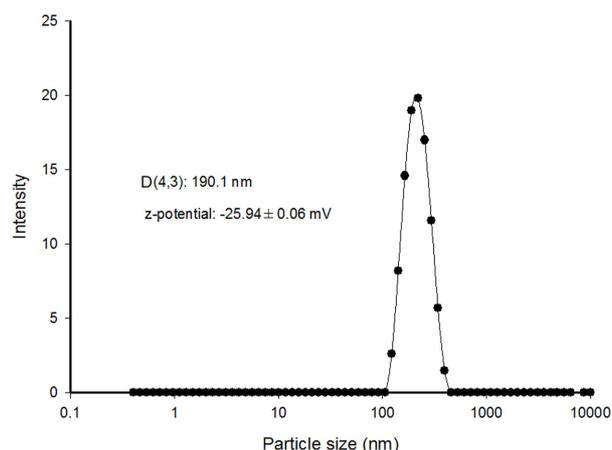


Fig. 5. Particle size distribution of milk beverage supplemented with coffee under the optimum condition by RSM.

than those reported by Moon *et al.* (2009), where the pH of coffee beverages ranged from 4.90 to 6.18, due to the presence of total chlorogenic acid in coffee beans, directly related to the roasting conditions of the green coffee beans. It is considered that sodium bicarbonate functioned as a pH controller in this beverage.

Peroxide values

Fig. 7 shows the changes in PV of coffee beverages stored during 30 d at 4°C. The PV of all samples increased during storage. The PV of the GF160 was higher than that

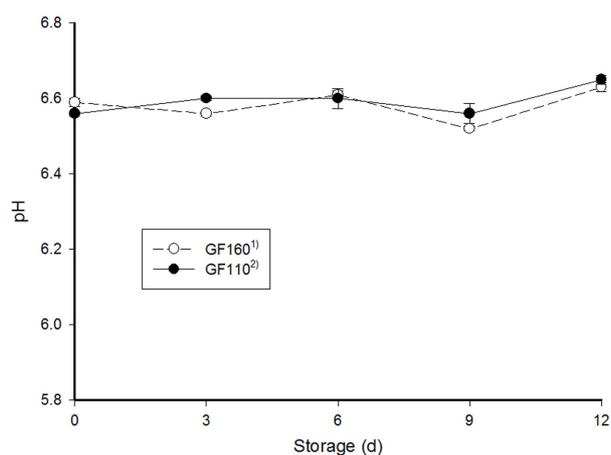


Fig. 6. Changes in pH of milk beverages supplemented with coffee in the presence of different emulsifiers during storage at 4°C for 12 d. ¹⁾GF160, sucrose fatty acid ester F160 added group; ²⁾GF110, sucrose fatty acid ester F110 added group.

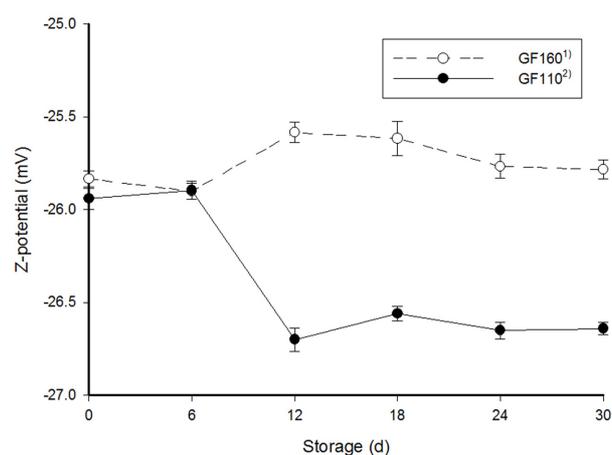


Fig. 8. Changes in zeta-potentials of milk beverages supplemented with coffee in the presence of different emulsifiers during storage at 4°C for 12 d. ¹⁾GF160, sucrose fatty acid ester F160 added group; ²⁾GF110, sucrose fatty acid ester F110 added group.

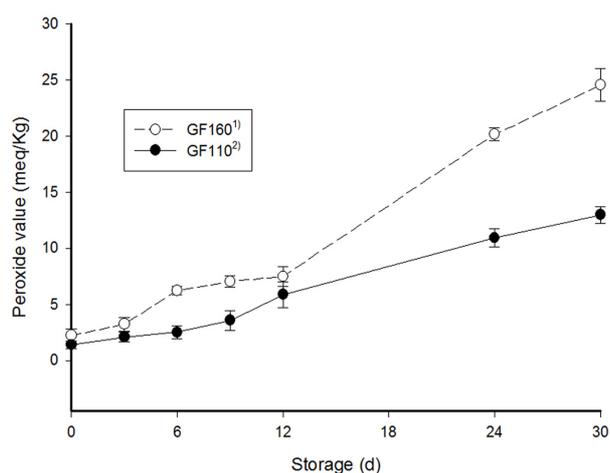


Fig. 7. Changes in peroxide values of milk beverages supplemented with coffee in the presence of different emulsifiers during storage at 4°C for 12 d. ¹⁾GF160, sucrose fatty acid ester F160 added group; ²⁾GF110, sucrose fatty acid ester F110 added group.

of the GF110. Furthermore, its PV rapidly increased after 12 d, reaching a value of 20 meq/Kg or more after 24 d. In contrast, the PV of the GF110 only increased from 1.41 to 13.0 meq/Kg during storage. In general, PV above 10-15 meq/Kg reflect oxidized fat (Joint FAO/WHO, 1999). It is thought that the milk fat globules of the GF110 were better dissolved by the selected emulsifier, sucrose fatty acid ester F110, than those of the GF160, potentially minimizing their floating properties and consequently, their chance of entering in contact with headspace oxygen.

Therefore, using sucrose fatty acid ester F110 was an effective way to prevent lipid oxidation in this beverage.

Particle size and zeta-potential

To determine the stability of the beverages during storage, their zeta-potentials and particle size distributions were measured and are presented in Figs. 8 and 9, respectively. The zeta-potential of the GF110 was higher than that of the GF160 after 6 d. It is known that a higher zeta-potential corresponds to greater particle stability (Freitas and Müllerä, 1998). Regarding size distribution of the beverages (Fig. 9), there were not changes within the GF110. In contrast, the particle size distribution of the GF160 increased during storage. Initially, the size distribution curve was sharp as well as monomodal, and remained stable. However, it gradually moved toward the right-hand side after the 12th storage day, indicating an increased droplet size. Moreover, particles above 3,000 nm were observed. This result accords with the report by Nasrabadi *et al.* (2016) showing that the droplet size of conjugated linoleic acid (CLA) beverage emulsions manufactured with 3.5% (w/w) CLA, 10% (w/w) gum Arabic, and 0.3% (w/w) xanthan gum gradually increased with time. Finally, the droplet size distribution curve of the control also became broader after 30 d of storage. A broad size distribution is suspected to be denotative of a population of irreversibly aggregated droplets formed with the passage of time after homogenization (Soleimanpour *et al.*, 2012).

The microstructures of the particles are indicated in Fig.

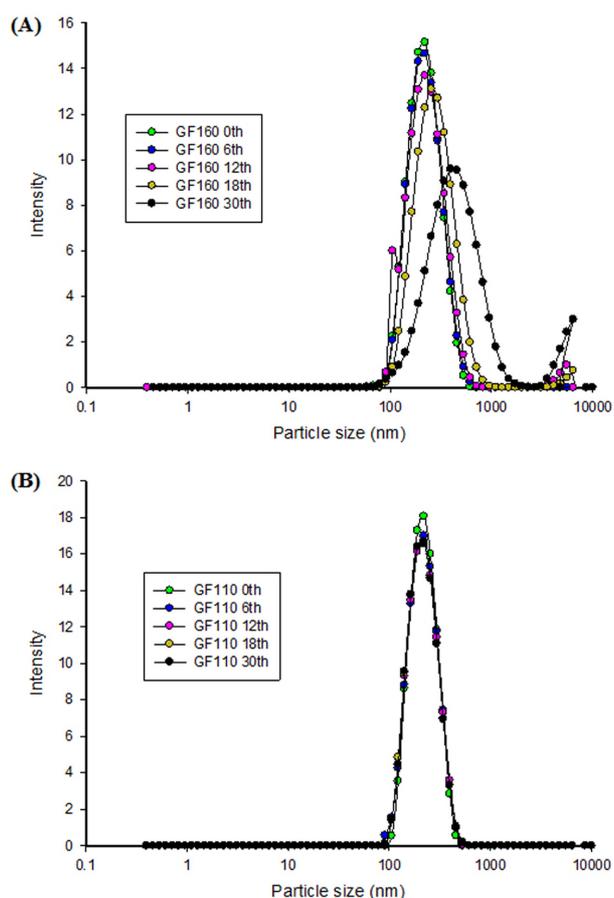


Fig. 9. Changes in size distributions of milk beverages supplemented with coffee in the presence of different emulsifiers during storage at 4°C for 12 d. (A) GF160 (sucrose fatty acid ester F160 added group); (B) GF110 (sucrose fatty acid ester F110 added group).

10. Initially, the particles of the GF160 and GF110 displayed rounded surfaces, and were evenly distributed. The particle size in the GF160 tended to increase during storage, in relation with the increase in average droplet size of the GF160 mentioned above. However, in the GF110, no changes in droplet size were observed. Sucrose fatty acid ester F110 may provide a higher fat globules emulsion level in the beverage than F160, and as a result, prevent particle aggregation more efficiently. Therefore, it was established that sucrose fatty acid ester F110 was more suitable than F160 for the manufacture of this beverage.

Color

Table 5 presents the changes in color of coffee beverages during 12 d of storage at 4°C. The L* values of either GF110 or GF160 significantly decreased during storage

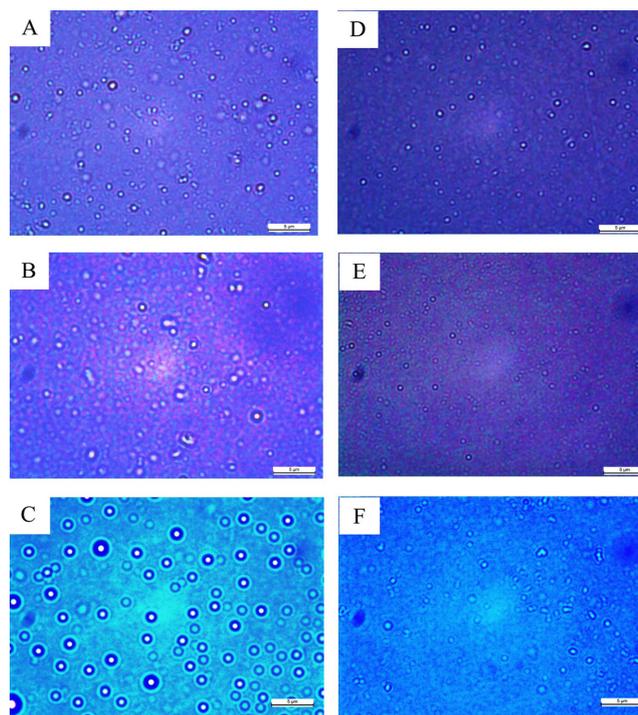


Fig. 10. Morphological changes of particles in milk beverages supplemented with coffee during storage. The photographs were taken at 1,000x magnification. Scale bar = 5 µm. GF160 at 0 d (A), GF160 at 7 d (B), GF160 at 30 d (C), GF110 at 0 d (D), GF110 at 7 d (E), and GF110 at 30 d (F).

($p < 0.05$). However, for a given day, different group types did not show significant differences in L* values during storage. According to McClements (2002), L* values increase as the droplet semidiameter decreases from 10 to 0.1 µm. Lightness depends on the direction of light scattering. As the droplet radius increases, the light gets progressively dispersed toward the forward direction, while a smaller fraction of light gets dispersed backwards and is consequently detected as diffusely reflected light. In this study, the average droplet size of the GF110 was around 190 µm, which was smaller than that of the GF160, and therefore, the L* value of the GF110 was higher than that of the GF160. The a* values of the GF110 were significantly higher than those of the GF160, except at the initial time point and after 6 d. The a* value of the GF160 was significantly higher than that of the GF110 initially, but there was no significant difference between the GF160 and GF110 after 6 d ($p < 0.05$). The b* values of the GF160 did not show significant variations during storage ($p < 0.05$), and there were only slight changes within those of the GF110. However, there was no significant differences between the GF160 and GF110 after 9 and 12 d ($p < 0.05$).

Table 5. Changes in color of milk beverages supplemented with coffee, using different emulsifiers, stored at 4°C for 12 d¹⁾

Color value	Sample	Storage period (d)				
		0	3	6	9	12
L*	GF160 ²⁾	52.07 ^{bb}	48.71 ^{bcB}	55.97 ^{aA}	44.53 ^{dB}	45.35 ^{cdA}
	GF110 ³⁾	60.36 ^{aA}	61.78 ^{aA}	56.37 ^{bA}	54.75 ^{bA}	49.61 ^{cA}
a*	GF160	6.28 ^{aA}	6.53 ^{aB}	4.93 ^{cA}	5.47 ^{bb}	5.34 ^{bb}
	GF110	5.45 ^{cB}	7.10 ^{aA}	4.69 ^{dA}	6.38 ^{bA}	6.59 ^{bA}
b*	GF160	21.26 ^{abA}	21.12 ^{abA}	22.50 ^{aA}	22.10 ^{abA}	20.90 ^{bA}
	GF110	20.02 ^{cB}	21.35 ^{bA}	21.45 ^{bb}	23.78 ^{aA}	21.14 ^{bA}

¹⁾Values with different superscript in a row (a-b) and column (A-B) are significant at $p < 0.05$ by Duncan's multiple range test;

²⁾GF160, sucrose fatty acid ester F160 added group; ³⁾GF110, sucrose fatty acid ester F110 added group.

Table 6. Sensory evaluation of milk beverages supplemented with coffee, using different emulsifiers, stored at 4°C for 12 d¹⁾

Sensory description	Sample	Storage period (d)				
		0	3	6	9	12
Brownness	GF160 ²⁾	3.20±0.45 ^{aA}	3.30±0.48 ^{aA}	3.40±0.52 ^{aA}	3.10±0.57 ^{aA}	3.10±0.32 ^{aA}
	GF110 ³⁾	3.20±0.89 ^{bA}	3.40±0.52 ^{aA}	3.30±0.48 ^{aA}	3.20±0.63 ^{aA}	3.20±0.42 ^{aA}
Floating cream	GF160	1.00±0.00 ^{bA}	1.00±0.00 ^{bA}	1.20±0.67 ^{bA}	1.30±0.63 ^{abA}	1.40±0.48 ^{aA}
	GF110	1.00±0.00 ^{aA}	1.00±0.00 ^{aA}	1.10±0.95 ^{aA}	1.10±0.63 ^{aA}	1.10±0.42 ^{aB}
Coffee flavor	GF160	3.40±1.14 ^{aA}	3.40±0.70 ^{aA}	3.60±0.97 ^{aA}	3.30±0.67 ^{aA}	2.90±0.57 ^{aA}
	GF110	3.80±1.10 ^{aA}	3.50±1.16 ^{aA}	3.30±1.20 ^{aA}	3.40±0.97 ^{aA}	3.10±0.74 ^{aA}
Milk flavor	GF160	3.30±1.79 ^{aA}	2.70±1.16 ^{aA}	2.50±0.85 ^{aA}	2.60±0.70 ^{aA}	2.40±0.97 ^{aA}
	GF110	3.20±0.84 ^{aA}	2.80±0.74 ^{aA}	2.40±0.67 ^{aA}	2.50±0.79 ^{aB}	2.40±1.07 ^{aA}
Rancid flavor	GF160	1.00±0.00 ^{aA}	1.00±0.00 ^{aA}	1.20±0.63 ^{aA}	1.30±0.67 ^{aA}	1.20±0.42 ^{aA}
	GF110	1.00±0.00 ^{aA}	1.00±0.00 ^{aA}	1.10±0.95 ^{aA}	1.10±0.70 ^{aA}	1.10±0.32 ^{aA}
Sweetness	GF160	3.60±1.34 ^{aA}	3.90±0.88 ^{aA}	3.10±0.99 ^{aA}	3.20±0.63 ^{aA}	3.50±0.85 ^{aA}
	GF110	2.80±0.45 ^{aB}	2.80±0.32 ^{aB}	3.20±1.23 ^{aA}	2.90±1.20 ^{aA}	3.60±0.52 ^{aA}
Bitterness	GF160	2.00±1.00 ^{aB}	1.80±0.79 ^{aA}	2.40±1.58 ^{aA}	1.80±1.03 ^{aA}	1.60±1.07 ^{aA}
	GF110	2.60±1.14 ^{aA}	2.10±1.29 ^{aA}	2.60±1.51 ^{aA}	2.30±1.34 ^{aA}	1.60±0.84 ^{aA}
Overall acceptability	GF160	4.20±0.84 ^{aA}	4.00±0.82 ^{aA}	3.30±1.16 ^{aA}	3.50±1.18 ^{aA}	3.40±1.07 ^{aA}
	GF110	2.80±0.45 ^{aB}	2.80±1.32 ^{aB}	3.00±1.15 ^{aA}	2.70±0.95 ^{aA}	3.00±0.82 ^{aA}

¹⁾Values with different superscript in a row (a-b) and column (A-B) are significant at $p < 0.05$ by Duncan's multiple range test;

²⁾GF160, sucrose fatty acid ester F160 added group; ³⁾GF110, sucrose fatty acid ester F110 added group.

Sensory evaluation

The sensory properties of the milk beverages with coffee during storage at 4°C for 12 d are shown in Table 6. Brownness of either the GF160 or GF110 showed no significant differences during storage ($p < 0.05$). Additionally, there was no significant difference between groups ($p < 0.05$). Floating cream levels of the GF160 gradually increased after 3 d of storage, and finally, after 12 d, significantly differed from the initial value ($p < 0.05$). Regarding the GF110, these levels increased slightly and showed no significant difference during storage. This behavior may be related to the dispersibility of fat globules in the beverages. In the same way, the size distribution, PV, and zeta-potential values of the GF110 were superior to those of the GF160, as mentioned above. Therefore, it is believed that the usage of sucrose fatty acid ester F110 as the emulsifier effectively dispersed the particles of the milk

beverage and prevented floating of cream in the GF110.

The coffee flavor of GF110 was relatively higher than that of the GF160. However, this difference was not statistically significant ($p < 0.05$). The sweetness of the control was relatively higher than that of the GF110 until day 3. However, there were no significant differences after this time point. In contrast, the bitterness of the GF110 was generally higher than that of the GF160, showing a significant difference initially ($p < 0.05$). Sweetness is known to reduce bitterness in beverages. This result correlates with a report by Li *et al.* (2014), who showed that there was a negative correlation between coffee flavor and sweetness in coffee-flavored milk. Li *et al.* (2014) investigated the correlations between various sensory properties in coffee-flavored milk. In their study, coffee flavor was negatively influenced by sweetness. Similarly, Pangborn (1982) and Anzueto *et al.* (1995) also mentioned that the addition of

sucrose decreases the sensory perception of bitterness in coffee beverages. Calvino *et al.* (1990) found that the restraint of bitterness and coffee flavor increased with enhanced sweetness. Moreover, Li *et al.* (2014) reported that sweetness is positively correlated with milk flavor. The milk flavor of the GF160 was relatively higher than that of the GF110 in the present study, even though it did not show a statistically significant difference ($p>0.05$). Rancid flavor gradually increased within all groups. However, there was no significant difference during storage ($p<0.05$). The rancid flavor of the GF110 was lower than that of the GF160, but this difference was not statistically significant ($p<0.05$). This particularity may also be due to dispersibility properties and floating cream levels, and well dispersed milk beverages, containing sucrose fatty acid ester F110, would not have floating fat globules and consequently, not form cream lines, reducing any potential contacts with headspace oxygen which therefore, would prevent rancid flavor from occurring. The other sensory properties, except for the overall acceptability, did not show significant differences between the GF160 and GF110 during storage ($p<0.05$). Overall acceptability of the GF110 was significantly lower than that of the GF160 between the initial time point and day 3 of the storage period ($p<0.05$). However, this difference was not significant anymore beyond 3 d of storage ($p<0.05$).

Conclusions

The present study indicated that the optimum conditions to manufacture of the beverage supplemented with coffee by RSM were 5,000 rpm of primary homogenization speed and approximately 0.2% emulsifier addition. Also, the qualitative properties of milk beverages supplemented with coffee emulsified with sucrose fatty acid ester F110 was superior to that obtained with F160 in terms of peroxide value, particle size distribution, zeta-potential, dispersibility, and rancid flavor. Especially, improvement of dispersibility by the F110 affected PV. It means that using the F110 into the beverage could effectively extend its shelf-life. Additionally, other properties of the beverages such as pH, brownness, coffee flavor, and tastes showed no significant difference between the GF160 and GF110. Based on the results obtained from the present study, it is considered that the application of sucrose fatty acid F110 into milk beverage supplemented with coffee can effectively prolong their shelf-life, even though further studies performing accelerated shelf-life tests are needed to provide

a deeper understanding of the phenomena involved.

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