Effect of sucrose ester and carboxymethyl cellulose on physical properties of coconut milk

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Abstracts The influence of sucrose ester (SE) and carboxymethyl cellulose (CMC) on the physical properties of coconut milk was determined using response surface methodology based on central composite design. The R^2 of all response variables was more than 0.80 which indicated a high proportion of variability was explained by the model and showed that increasing the amount of SE decreased the droplet size of coconut milk. The viscosity and creaming index were dependent on the SE and CMC concentration. Increasing the SE and CMC concentration increased viscosity but creaming index was decreased. The results suggested that suitable amount of SE and CMC should be specified in order to obtain a high quality of coconut milk products.

Keywords Carboxymethyl cellulose · Central composite design · Coconut milk · Physical properties · Sucrose ester

Introduction

Coconut milk is an oil-in-water emulsion obtained from the aqueous extract of coconut meat either with or without additional water. The composition of coconut milk without the addition of water is 55.14–55.38 wt% water, 32.00–35.00 wt% lipids, 4.00–4.03 wt% protein and 1.00–1.02 wt% ash (Tansakul and Chaisawang 2006). Coconut milk products are available in the market using either sterilized coconut milk or pasteurized coconut milk.

They are convenient for household use as an important food ingredient in Asian cuisine such as curries and desserts (Seow and Gwee 1997). However, the coconut milk product is unstable and is prone to phase separation because the protein content in coconut milk is not sufficient to stabilize the product (Monera and Rosario 1982; Tansakul and Chaisawang 2006).

In order to improve the coconut milk stability, stabilizers are added during processing. Stabilizers are any ingredients (e.g. emulsifier, thickening agent) that can be used to enhance the emulsion stability. Emulsifiers are surface-active molecules that adsorb to the surface of freshly formed droplets during homogenization, forming a protective layer that prevents aggregation of the droplets. Thickening agents are ingredients that are used to increase the viscosity of the continuous phase of emulsions (McClements 2015). Several researchers have studied the effect of stabilizers on coconut milk stability. On the one hand, different emulsifiers such as Tween 20, Tween 60, sodium dodecyl sulfate and sucrose ester (SE) have been used (Simaung et al. 2004; Tangsuphoom and Coupland 2008; Ariyaprakai et al. 2013). On the other hand, stabilizing polysaccharides such as carboxymethyl cellulose (CMC), xanthan gum and acacia gum have been added alone or added with emulsifiers (Phungamngoen et al. 2004; Tipvarakarnkoon et al. 2010). Emulsifiers are also used in combinations with other types of emulsifiers and stabilizers because they can improve functional properties (Phungamngoen et al. 2004; McClements 2015) such as Tween 60 in combination with Span 80 and Tween 60 incorporated with CMC (Peamprasart and Chiewchan 2006; Tansakul and Chaisawang 2006).

The percentage of fat in coconut milk product is adjusted between 15 and 40 wt% depending upon local requirements. Many studies have investigated coconut milk

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with a final concentration ranging between 5 and 35 wt% fat (Jirapeangtong et al. 2008; Tangsuphoom and Coupland 2008; Tipvarakarnkoon et al. 2010; Ariyaprakai et al. 2013). Our preliminary study showed that the fat content of coconut milk in Thai dessert ranged from 9 to 13 wt%; therefore in this study, we prepared coconut milk with a final fat concentration of 10 wt% and investigated the effect of SE incorporated with CMC in stabilizing coconut milk. SE is a sugar-based surfactant, which has hydrophilic sucrose groups and a hydrophobic fatty acid group. It is non-toxic, odorless, tasteless, non-irritating to the skin and easily digested. It is also approved as a food additive under the food regulations of many countries such as Japan, the USA and the European Community and is widely used in food formulations, cosmetics and chemistry (Garti 2001; Neta et al. 2015). CMC, a sort of derivative cellulose, is a linear anionic polymer which is manufactured by chemically attaching carboxymethyl groups to the backbone of native cellulose. It is soluble in both hot and cold water to give clear and colorless solution with a neutral flavor and can form a viscous solution. It is commonly used in foods and beverages to prevent gravitational separation of suspended particles and also to create desirable textural attributes and mouthfeel (Imeson 1997; Murray 2000; Saha and Bhattacharya 2010; McClements 2015). The aim of this study was to investigate the effect of SE (0–1 wt%) and CMC (0.1–0.5 wt%) on physical properties and stability of coconut milk by using response surface methodology (RSM) based on central composite design (CCD).

Materials and methods

Materials

Fresh coconut milk extracted from coconut white meat without the addition of water was purchased from a local market in Bangkok. Sucrose esters (S1170, containing mainly 55% monoester with 45% di-poly-ester and moisture content 3.72 wt%) was purchased from Mitsubishi Kagaku Foods (Tokyo, Japan). Carboxymethyl cellulose (Blanose® 7HF, moisture content 11.38 wt%) was donated by Bronson and Jacobs International (Bangkok, Thailand). All other chemicals were of analytical grade. Distilled and deionized water (DI water) was used for the preparation of all solutions.

Solution preparation

Carboxymethyl cellulose (CMC) solution (2.5 wt%) was prepared by dispersing CMC powder in DI water and stirring for 2 h at room temperature until it had dissolved completely. The solution of CMC was kept overnight at

4 ± 2 °C in order to obtain complete dissolution before use (Tipvarakarnkoon et al. 2010).

Coconut milk preparation

The initial fat content of fresh coconut milk was determined using the Babcock method for determination of the fat content in dairy products (AOAC Official Method 989.04, AOAC 2006). A weighed amount of SE was added into coconut milk and mixed together with CMC solution into aliquots of coconut milk to obtain a final fat content of 10 wt%, 0.0–1.0 wt% SE and 0.1–0.5 wt% CMC. The concentration of SE used in this study was accorded to the CODEX Alimentarius (CODEX STAN 240-2003) (Codex Alimentarius 2003) and the concentration of CMC was obtained from our preliminary test. Each coconut milk sample was pre-homogenized at 15,000 rpm for 3 min using a high speed homogenizer (Ultra-Turrax T25, IKA, Staufen, Germany). Coconut milk samples were heated at 70 °C for 1 min to prevent deterioration due to microorganism spoilage and chemical change caused by lipase (Seow and Gwee 1997; Jirapeangtong et al. 2008). Coconut milk samples were then homogenized using a high pressure valve homogenizer at 200/20 bar for three passes (APV 2000, SPX Flow Technology, Unna, Germany). Sodium azide (0.02 wt%) was added to prevent microbial spoilage. All coconut milk samples were stored at room temperature for 24 h before being analyzed and the experiment was repeated three times with freshly prepared samples.

Droplet size

The droplet size of coconut milk samples was determined using static light scattering (Mastersizer 2000; Malvern Instruments Ltd., Worcestershire, UK), with a dual wavelength detection system. Each coconut milk sample was dropped and diluted in the test chamber that had been filled with distilled water in order to prevent multiple light scattering effects. The refractive indices of 1.33 for water and 1.45 for coconut oil were employed as optical properties of coconut milk samples. Droplet sizes were reported as the surface volume mean diameter, d_{32} ($= \sum n_i d_i^3 / \sum n_i d_i^2$) and volume-weighted average diameters, d_{43} ($= \sum n_i d_i^4 / \sum n_i d_i^3$), where d_i is the midpoint of the size interval and n_i is the number of droplets in that interval (Tipvarakarnkoon et al. 2010).

Optical microscopy

Coconut milk samples were diluted using distilled water at the ratio of 1:25 in glass test tubes and gently agitated to ensure a homogenized condition. A drop of sample was placed on a microscope slide under a cover slip. The

microstructure of each coconut milk sample was then observed using a conventional optical microscope (Carl Zeiss J/902165, Oberkochen, Germany) (Tipvarakarnkoon et al. 2010).

Rheological properties

Rheological measurements were carried out using a rheometer (MCR 300, Physica, Stuttgart, Germany) operating with a DG26.7 double gap rotational cylinder. Each coconut milk sample was gently mixed and then portions were transferred to the instrument. The instrument had previously been equilibrated at 25 °C and the test was run after pre-shearing for 2 min. The shear rate was increased from 10 to 1000 s⁻¹ (Tangsuphoom and Coupland 2005). The apparent viscosity at a shear rate of 50 s⁻¹ was selected to present the effect of SE and CMC on the coconut milk viscosity. The presented data were based on the average of three replicates. The shear stress (τ) data were also used to analyze the consistency index (K) and the flow behavior index (n) according to the power law shown in Eq. (1):

$$\tau = K\sigma^n \quad (1)$$

For an ideal Newtonian liquid, $n = 1$, for an emulsion which exhibits shear thinning, $n < 1$ and for an emulsion which exhibits shear thickening, $n > 1$ (Dickinson 1992).

Creaming index

Coconut milk samples (10 g) were transferred into a glass test tube (internal diameter 15 mm, height 150 mm), covered and stored for 7 days at room temperature. The total height of the emulsions (H_E) and the height of the serum layer (H_S) were measured. The extent of the phase separation was characterized using the % creaming index = $100 \times (H_S/H_E)$ for which, the lower the creaming index, the higher the emulsion stability (Tangsuphoom and Coupland 2008).

Statistical analysis

Data were presented as the mean $\pm$ SD of the triplicates. Significant differences ($P \leq 0.05$) between means were determined using one-way analysis of variance (ANOVA) and Duncan's multiple range tests (SPSS 12, SPSS, Chicago, IL, USA). Response surface methodology (RSM) based on central composite design (CCD) was employed to study the effect of SE (X_1) and CMC (X_2) concentration on the responses; droplet size (d_{32} ; Y_1), droplet size (d_{43} ; Y_2), viscosity (Y_3) and creaming index (Y_4) of coconut milk samples. Thirteen coconut milk samples were established based on the CCD with two independent variables at five

levels on each variable (Table 1). Data was used to fit with a polynomial model as followed:

$$Y_i = B_0 + B_1X_1 + B_{11}X_1^2 + B_2X_2 + B_{22}X_2^2 + B_{12}X_1X_2 \quad (2)$$

where Y_i = dependent variable; B_0 = constant intercept; B_1 , B_2 , B_{11} , B_{22} and B_{12} = constant coefficients; X_1 = SE concentration and X_2 = CMC concentration. Contour plots were generated by STATISTICA 7.0, a trial version (StatSoft, Inc., Tulsa, OK, USA).

Results and discussion

This study was conducted to investigate the effect of SE (0–1.0 wt%) and CMC (0.1–0.5 wt%) on the droplet size (d_{32} and d_{43}), viscosity and creaming index of coconut milk samples (10 wt% fat) and the results were analyzed using RSM based on CCD in order to find significant factors (SE and CMC concentrations) by applying ANOVA.

Change in droplet size and microstructure

One of the most effective indices that reflect the stability of an emulsion is the droplet size; the smaller the droplet size, the more stable the emulsion is Zhao et al. (2014). In this study, all coconut milk samples appeared as white and creamy emulsions. The droplet size of coconut milk samples was determined using a static light scattering instrument and the results are shown in Table 2. In the absence

Table 1 Central composite design for the independent variables (coded and actual levels)

Run	Coded value		Actual value	
	SE ¹ (X_1)	CMC ² (X_2)	SE ¹ (wt%)	CMC ² (wt%)
1	1	1	0.75	0.4
2	1	– 1	0.75	0.2
3	– 1	1	0.25	0.4
4	– 1	– 1	0.25	0.2
5	2	0	1.0	0.3
6	0	2	0.5	0.5
7	– 2	0	0.0	0.3
8	0	– 2	0.5	0.1
9	0	0	0.5	0.3
10	0	0	0.5	0.3
11	0	0	0.5	0.3
12	0	0	0.5	0.3
13	0	0	0.5	0.3

¹Sucrose ester

²Carboxymethyl cellulose

Table 2 Influence of sucrose ester (SE) and carboxymethyl cellulose (CMC) on physical properties of coconut milk

Run	SE (wt%)	CMC (wt%)	Droplet size (d_{32} ; μm) ¹	Droplet size (d_{43} ; μm) ¹	Creaming index (%) ¹	Apparent viscosity (mPa s) ¹	Consistency index (Pa s) ¹	Flow behavior index ¹
1	0.75	0.4	0.31 $\pm$ 0.00	0.64 $\pm$ 0.02	34.69 $\pm$ 0.82	35.03 $\pm$ 1.80	0.102 $\pm$ 0.025	0.712 $\pm$ 0.040
2	0.75	0.2	0.33 $\pm$ 0.01	0.74 $\pm$ 0.00	39.83 $\pm$ 0.36	12.15 $\pm$ 1.70	0.027 $\pm$ 0.007	0.790 $\pm$ 0.038
3	0.25	0.4	0.39 $\pm$ 0.00	1.08 $\pm$ 0.01	38.57 $\pm$ 0.34	22.38 $\pm$ 0.60	0.050 $\pm$ 0.002	0.791 $\pm$ 0.012
4	0.25	0.2	0.41 $\pm$ 0.01	1.12 $\pm$ 0.00	63.44 $\pm$ 0.49	8.49 $\pm$ 0.55	0.018 $\pm$ 0.001	0.800 $\pm$ 0.014
5	1.0	0.3	0.29 $\pm$ 0.00	0.65 $\pm$ 0.02	37.04 $\pm$ 0.36	37.28 $\pm$ 2.01	0.194 $\pm$ 0.077	0.582 $\pm$ 0.052
6	0.5	0.5	0.30 $\pm$ 0.01	0.66 $\pm$ 0.03	35.71 $\pm$ 0.34	38.15 $\pm$ 2.04	0.085 $\pm$ 0.004	0.801 $\pm$ 0.018
7	0.0	0.3	19.51 $\pm$ 1.50	22.52 $\pm$ 0.18	75.89 $\pm$ 0.06	13.18 $\pm$ 1.03	0.013 $\pm$ 0.002	0.961 $\pm$ 0.006
8	0.5	0.1	0.39 $\pm$ 0.00	0.77 $\pm$ 0.02	75.71 $\pm$ 0.50	4.34 $\pm$ 0.16	0.008 $\pm$ 0.001	0.845 $\pm$ 0.031
9	0.5	0.3	0.31 $\pm$ 0.00	0.67 $\pm$ 0.02	40.35 $\pm$ 1.18	14.68 $\pm$ 2.58	0.030 $\pm$ 0.002	0.803 $\pm$ 0.020
10	0.5	0.3	0.30 $\pm$ 0.01	0.67 $\pm$ 0.01	39.91 $\pm$ 1.01	14.63 $\pm$ 1.66	0.030 $\pm$ 0.005	0.807 $\pm$ 0.010
11	0.5	0.3	0.31 $\pm$ 0.00	0.67 $\pm$ 0.01	39.84 $\pm$ 0.22	15.30 $\pm$ 0.14	0.037 $\pm$ 0.002	0.791 $\pm$ 0.017
12	0.5	0.3	0.31 $\pm$ 0.00	0.66 $\pm$ 0.00	38.82 $\pm$ 0.83	15.30 $\pm$ 0.49	0.035 $\pm$ 0.006	0.791 $\pm$ 0.010
13	0.5	0.3	0.31 $\pm$ 0.00	0.66 $\pm$ 0.00	39.05 $\pm$ 0.37	14.33 $\pm$ 1.38	0.036 $\pm$ 0.007	0.790 $\pm$ 0.008

¹Mean $\pm$ standard deviation of three replicates

of SE, droplet sizes of coconut milk were very large (19.51 μm for d_{32} and 22.52 μm for d_{43}). The addition of SE resulted in a decrease in the average droplet size of coconut milk (0.33 μm for d_{32} and 0.75 μm for d_{43}). Figure 1 shows the droplet size distribution of coconut milk with the absence of SE which has a large population on the right of the size distribution, indicating the sample contained a larger droplet size than a sample containing SE. This was due to SE being an effective emulsifier that adsorbs onto the surface of the droplet and reduces the oil–water interfacial tension (Tangsuphoom and Coupland 2008; Tipvarakarnkoon et al. 2010; Ariyaprakai et al. 2013, Neta et al. 2015). The results shown in Table 3 confirmed

that the coefficient values of SE were higher than those of CMC, showing the strong effect on droplet size of coconut milk. Ariyaprakai et al. (2013) also reported that SE was an effective emulsifier. It is found that coconut milk stabilized with SE showed the good stability due to SE having good ability to reduce the oil–water interfacial tension. The result was confirmed by the optical micrographs (Fig. 1) which showed that the droplet flocculation occurred in coconut milk in the absence of SE, while coconut milk stabilized with SE had smaller droplet size and non-flocculated droplets.

Rheological properties of coconut milk emulsion

The rheological behavior of a particular food emulsion depends on the type and concentration of the ingredients that it contains (McClements 2015). All samples exhibited shear thinning behavior (a decrease in the apparent viscosity of a fluid as the shear rate is increased) which is typically found in food emulsions. A power law model was applied to describe the rheological behavior of the coconut milk samples as shown in Table 2. This model suitably explained the experimental data with correlation coefficients (R^2) ranging from 0.997 to 1.000 (Maskan and Göğüş 2000). The flow behavior index (n) of coconut milk was consistently below unity ($n < 1$) indicating shear thinning behavior of the coconut milk samples at all SE and CMC concentrations. The consistency index (K) is an indication of the viscous nature of food (Maskan and Göğüş 2000); the K values of the coconut milk samples had values ranging from 0.008 to 0.194 Pa.s. The results suggested that the SE and CMC concentrations had a

Fig. 1 Dependence of droplet size distributions and optical micrographs of coconut milk samples stabilized by sucrose ester (SE) incorporated with 0.3 wt% carboxymethyl cellulose (CMC) and 0 wt% SE (.....), 0.5 wt% SE (---) and 1.0 wt% SE (—)

Table 3 Predicted regression models and their R^2 values

Response variables	Model ¹	R^2
Droplet size (d_{32} ; μm); Y_1	$15.29 - 50.59X_1 + 1.69X_2 + 37.728X_1^2 - 3.14X_2^2 - 0.0X_1X_2$	0.78
Droplet size (d_{43} ; μm); Y_2	$17.87 - 57.66X_1 + 1.87X_2 + 42.98X_1^2 - 3.11X_2^2 - 0.60X_1X_2$	0.80
Viscosity (mPa s); Y_3	$15.99 - 46.29X_1 - 51.29X_2 + 40.83X_1^2 - 155.56X_2^2 + 89.90X_1X_2$	0.99
Creaming index (%); Y_4	$165.50 - 160.71X_1 - 428.23X_2 + 66.46X_1^2 + 396.51X_2^2 + 197.30X_1X_2$	0.97

¹ X_1 SE concentration; X_2 CMC concentration

significant ($P \leq 0.05$) effect on the viscosity, as increasing the concentration of SE and CMC resulted in an increase in the coconut milk viscosity. As shown in Table 3, the coefficient value of CMC was higher than SE, that mean CMC had an effect on the viscosity of the coconut milk samples higher than SE. It showed that increasing the CMC concentration from 0.1 to 0.5 wt% had a considerably positive influence on the viscosity of coconut milk. Specifically, an increase in the CMC concentration had greater effect on increasing the viscosity, compared with SE because the addition of CMC caused an increase in the viscosity of the continuous phase and therefore it was able to retard the gravitational separation of the droplets (Phungamngoen et al. 2004; Jirapeangtong et al. 2008). This result was in an agreement with Jirapeangtong et al. (2008) who found that an increase in the CMC content had a greater effect compared with Montanox 60, on an increase in the consistency index (K) and a decrease in the flow behavior index (n).

Stability in coconut milk emulsion

Generally, creaming is usually regarded as having an adverse effect on the stability of an emulsion, which is caused by gravity (McClements 2015). A lower creaming index shows greater stability of an emulsion (Tangsuphoom and Coupland 2008; Ariyaprakai et al. 2013). The creaming index of the coconut milk samples with different SE and CMC concentrations are shown in Table 2. In the absence of SE, the creaming index of the coconut milk samples after storage for 7 days was the highest (75.89%), while the addition of SE led to a decrease in the creaming index. It has also been observed that adding a small amount of CMC to coconut milk samples led to faster creaming separation due to bridging flocculation (Arancibia et al. 2013). Figure 2 shows the creaming index of coconut milk samples with added SE incorporated with CMC, where increasing the SE and CMC concentrations resulted in a decrease in the creaming index. These results agreed with previous studies which reported that increasing the concentration of emulsifiers and stabilizers resulted in a decrease in the creaming index, due to the emulsifier being

able to adsorb onto surface of droplets during homogenization, forming a protective layer, while the stabilizer is able to increase the viscosity of the continuous phase of emulsions which retards the droplets from coming close enough together to aggregate (Jirapeangtong et al. 2008; Tangsuphoom and Coupland 2008; Tipvarakarnkoon et al. 2010; Zhao et al. 2014). As shown in Table 3, the creaming index was directly related to the SE and CMC concentration. Moreover, the coefficient values of CMC were higher than those of SE. The results indicated that an increase in CMC had a greater effect on decreasing the creaming index, compared with SE because the addition of CMC caused an increase in the viscosity of the continuous phase and then the upward movement of the flocs could be retarded (Phungamngoen et al. 2004; Jirapeangtong et al. 2008).

Response surface analysis

RSM was applied to explain the effect of the SE and CMC concentration on the droplet size (d_{43}), viscosity (shear rate 50 s^{-1}) and creaming index (after storage for 7 days) of coconut milk. In order to show the relationship between the two variables, contour plots were also employed (Fig. 3). The ANOVA results showed that the selected quadratic model adequately represented the data obtained for the physical properties of the coconut milk samples. As shown in Table 3, all fitted models were significant ($P \leq 0.05$) with high R^2 values. The R^2 values should be at least 0.80 for a good fit of a model (Mirhosseini et al. 2008; Lima et al. 2010; Yaakob et al. 2012; Caporaso et al. 2016). The R^2 values for a droplet size of d_{32} were around 0.78 indicating that only 0.22 of the data could not be interpreted by the model. According to the droplet size model, the coefficient values of SE were significant ($P \leq 0.05$), indicating that the SE had an effect on the droplet size, while the viscosity and creaming index models had coefficient values of SE and CMC that were significant ($P \leq 0.05$), indicated that the SE and CMC concentrations had an effect on the viscosity and creaming index of coconut milk samples, but the coefficient value of CMC was higher than for SE, showing its strong effect on the viscosity and creaming

Fig. 2 Dependence of creaming index of coconut milk samples stabilized by sucrose ester (SE) and carboxymethyl cellulose (CMC): **a** effect of SE (incorporated with 0.3 wt% CMC) and **b** effect of CMC (incorporated with 0.5 wt% SE)

Fig. 3 Contour plots for the effect of sucrose ester (SE) and carboxymethyl cellulose (CMC) concentrations on droplet size (a); viscosity (b) and creaming index (c)

index. The regression model was confirmed by contour plots (Fig. 3) which show that SE had an effect on droplet size (Fig. 3a) while the viscosity and creaming index were dependent on both SE and CMC (Fig. 3b, c). Comparing to commercial coconut milk products, the results suggested

that 0.5 wt% SE and 0.3 wt% CMC were required to produce the high stability of coconut milk (10 wt% fat) which has small droplet size, low viscosity and creaming index.

Conclusion

RSM based on central composite design (CCD) experimental plan enabled the determination of the effect of sucrose ester (SE) and carboxymethyl cellulose (CMC) on the physical properties of coconut milk. The results showed that the SE and CMC concentration had an effect on physical properties of coconut milk (10 wt% fat). SE had an effect on the droplet size of coconut milk due to the ability of SE to reduce the surface tension of a liquid medium to prevent the flocculation of oil droplets. The SE and CMC concentration had an effect on the viscosity and creaming index of coconut milk; increasing the concentrations of both resulted in an increase in coconut milk viscosity, while the creaming index decreased. This might have been due to CMC acting as a thickening agent, which increased the viscosity of the continuous phase of the emulsion, thus preventing droplet aggregation. While SE acted as an emulsifier which was adsorbed onto the interfacial layer and prevented droplet aggregation by forming a protective layer. From this study, it can conclude that 0.5 wt% SE and 0.3 wt% CMC are sufficient at stabilizing coconut milk, which has small droplet size, low viscosity and low creaming index. The results of this research clearly showed that SE and CMC can be used to improve the stability of coconut milk product.

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