Effect of Heating and Homogenization on the Stability of Coconut Milk Emulsions

NATTAPOL TANGSUPHOOM AND JOHN N. COUPLAND

ABSTRACT: The effects of homogenization and heat treatment on the colloidal stability of coconut milk were studied. Fresh coconut milk (15% to 17% fat, 1.5% to 2% protein) was extracted and stored at 30 °C before homogenization at 40/4 MPa (stage I/stage II). Both homogenized and non-homogenized samples were heated at 50 °C, 60 °C, 70 °C, 80 °C, and 90 °C for 1 h. Homogenization reduced the size of the primary emulsion droplets from 10.9 to 3.0 μm, but increased the degree of flocculation, presumably via a bridging mechanism. This flocculation was also responsible for increased viscosity of the homogenized samples. Heating increased the degree of flocculation in both non-homogenized and homogenized samples. A slight amount of coalescence was also observed after heating above 80 °C. All samples creamed after 24 h of storage, but the heated samples formed a larger cream layer, presumably because the flocculated droplets packed together less efficiently. Optical microscopy was used to confirm the combination of flocculation and creaming responsible for changes in coconut milk quality. The information obtained from this study provides a better understanding of the emulsion science important in controlling coconut milk functionality.

Keywords: coconut milk, emulsion, emulsion stability, heating, homogenization

Introduction

Coconut milk is the white opaque liquid obtained from shredded coconut (*Cocos nucifera* L.) meat made by comminuting or grating the flesh of the nut (with or without the addition of water) and pressing or dewatering the comminuted pulp. It is an important ingredient for Asian cuisine as well as in other parts of the world. The composition of coconut milk varies according to variety, age, growing environment of the coconut, cultural practices, method of preparation, and the process conditions used in extraction, for example, the amount of added water and the temperature used for extraction (Cancel 1979; Gonzalez 1990). Typical compositions of the coconut milk directly expelled from coconut kernel (without added water) are protein, 2.6% to 4.4%; water, 50% to 54%; lipids, 32% to 40%; and ash, 1% to 1.5% (Seow and Gwee 1997).

Coconut milk is essentially an oil-in-water emulsion, stabilized by the naturally occurring proteins (globulins and albumins) and phospholipids (for example, lecithin and cephalin) (Birosel and others 1963). As with all emulsions, coconut milk is not physically stable and is prone to phase separation. Natural coconut milk will separate into a cream and serum layer within 5 to 10 h of manufacture.

Thermal processing is an effective means of extending the shelf life of coconut milk. The processing of canned coconut milk starts with the extraction of coconut milk, which is then heated to a temperature of about 92 °C to 95 °C for 5 to 20 min (a process often referred to in the coconut industry as pasteurization) and often mixed with emulsifiers and/or stabilizers before a homogenization process. The homogenized milk is either hot-filled in cans or passed through an exhaust box before can sealing. Because the pH of coconut milk is about 6, it is considered as a low-acid food and the cans must be retorted (Timmins and Kramer 1977; Arumugan and others 1993).

MS 20050246 Submitted 4/26/05, Revised 5/10/05, Accepted 7/11/05. *The authors are with Dept. of Food Science, The Pennsylvania State Univ., 126 Borland Laboratory, Univ. Park, PA 16802. Direct inquiries to author Coupland (E-mail: coupland@psu.edu).*

The physical properties of coconut emulsions have not been well studied. It is common practical knowledge that heating and homogenization affect the stability of coconut milk emulsions. However, the mechanisms of such stability alterations and the underlying emulsion science are still unclear. In this work we determine the effects and mechanisms of homogenization and heat treatment on the colloidal stability of freshly-manufactured coconut milk.

Materials and Methods

Sample preparation

Coconuts were purchased from a local retailer, deshelled, and shredded using a traditional coconut grater. Coconut milk was produced by mixing the shredded pulp with an equal weight of warm distilled water (60 °C) in a Waring blender (Waring, 1120, Winstel, Conn., U.S.A.), filtered through a double-layered cheese cloth, and manually squeezed with a twisting motion to extract most of the milk. Thimerosal (0.01 wt%; Sigma Chemical, St. Louis, Mo., U.S.A.) was added as an antimicrobial agent. The extracted emulsion was stored at 30 °C before analysis and used within 24 h of manufacture.

The crude protein content of coconut milk was measured using the nitrogen combustion method by an automatic nitrogen analyzer (Leco, FP-528, St. Joseph, Mich., U.S.A.). The fat content was determined using solvent extraction with petroleum ether (Sigma Chemical) at a sample-solvent ratio of 1:3 in a Majonnier flask (FMC, Chicago, Ill., U.S.A.) and weighing of the fat after the solvent was evaporated in a Soxtec extraction unit (Foss Tecator, Eden Prairie, Minn., U.S.A.).

Homogenized samples were prepared by recirculating fresh coconut milk through a twin-stage valve homogenizer (GEA Niro Soavi, Panda, Hudson, Wis., U.S.A.) at a pressure of 40/4 MPa for 4 min to achieve multiple passes through the valves. Both non-homogenized and homogenized samples were heated in a temperature-controlled water bath set at 50 °C, 60 °C, 70 °C, 80 °C, and 90 °C for 1 h and then cooled to 30 °C in another water bath before analysis.

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Particle size analysis

The weight-average diameters (d_{43}) of the coconut milk emulsion droplets and their size distribution (volume fraction as a function of particle size) were measured using a laser diffraction particle analyzer (Horiba, LA-920, Irvine, Calif., U.S.A.) assuming a relative refractive index of 1.15. Coconut milk samples were diluted to approximately 0.001% fat before analysis to minimize multiple scattering effects. In some experiments, the coconut milk was diluted in 1 wt% sodium dodecyl sulfate (SDS, Sigma Chemical) solution rather than water. SDS is an anionic surfactant that effectively displaces protein from the surface of emulsion droplets and disrupts droplet flocs formed due to interdroplet protein-protein interactions. Particle size measured in water is referred to in this work as an "effective" particle size and includes the presence of flocs, whereas measurement in SDS solution is called the "primary" particle size. Emulsion coalescence will be seen as a change in both primary and effective particle diameter, whereas flocculation will increase the effective diameter but the primary diameter will remain unchanged.

Microscopy

Samples of coconut milk (about 25 μL) were placed on a microscope slide, gently covered with a cover slip, and observed at 200× magnification using an optical microscope (BX40,Olympus, Melville, N.Y., U.S.A.) equipped with a color video camera (DXC-970MD, Sony, New York, N.Y., U.S.A.).

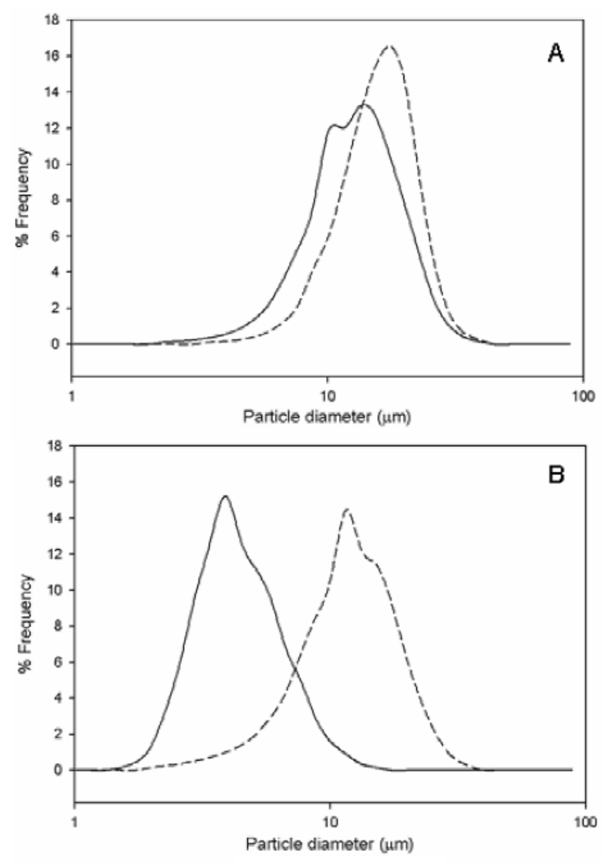

Figure 1—Representative droplet size distribution of non-homogenized (a) and homogenized (b) coconut milks dispersed in distilled water (— — —= effective particle size distribution including the presence of flocs) or sodium dodecyl sulfate (SDS) (— = primary particle size distribution) before analysis.

Rheological studies

Rheological measurements were carried out using a controlled strain rheometer (TA Instruments, Ares, New Castle, Del., U.S.A.) operating with a cone and plate geometry (cone dia, 50 mm; cone angle, 0.04 radian). Samples were equilibrated at 30 °C, gently mixed, and then portions (about 1.5 mL) were transferred to the instrument. The instrument had previously been equilibrated at 30 °C and the test was run immediately. The shear rate was increased from 0/s to 100/s over 7 min and the required stress used to calculate the apparent viscosity.

Creaming stability measurements

Portions (10 g) of coconut milk samples were transferred into glass tubes, covered, and allowed to stand for 24 h at room temperature. All samples separated into the opaque layer at the top and the transparent aqueous phase at the bottom during storage. The extent of the phase separation was assessed by creaming index, which is the percentage ratio between the height of the transparent layer (H_T) and total height of the emulsion (H_E) in the test tube.

Determination of free fat

The degree of emulsion destabilization was measured as the amount of solvent-extractable oil. Samples of coconut milk (10 g) were transferred to Majonnier flasks and extracted with petroleum ether (Sigma Chemical) at the volume ratio of 3:1. The organic extracts were evaporated to dryness in a Soxtec extraction unit (Foss Tecator) and the extractable oil weighed. The extraction was repeated 5 times, and the cumulative value of extracted fat was calculated.

Statistical analyses

Most experiments were conducted in triplicate with freshly prepared coconut milk used on each occasion. Data were analyzed using SPSS for Windows, release 11.5.0 (SPSS, Chicago, Ill., U.S.A.). One-way analysis of variance (ANOVA) and Duncan's multiple range tests were used to evaluate the significance of differences ($P < 0.05$) between the samples. Only significantly different ($P < 0.05$) results are discussed in the text. Data are presented as the mean and standard deviation.

Results and Discussion

The coconut milk prepared by the described method contained 15% to 17% fat and 1.5% to 2% protein. The droplet size distribution in the fresh milk had an approximately log-normal form (Figure 1a). The effective droplet diameter (d_{43}) of the fresh coconut milks was 13.1 μm with a standard deviation of the distributions of approximately 2 μm. Surprisingly, the homogenized milk had only slightly different effective particle size from the non-homogenized milk (Figure 1b). However, when the milk was dispersed in SDS solution rather than distilled water before laser diffraction particle sizing, the homogenized samples were much smaller than the non-homogenized samples. SDS displaces the protein from the oil-water interface, thus disrupting any flocculation caused by interdroplet protein-protein interactions and allows the instrument to measure the primary particle size rather than the apparent size of flocs present. The non-homogenized milk particle size was not markedly affected by dilution in SDS, suggesting the particles present were less significantly flocculated.

This suggests that homogenization significantly reduces the mean diameter of the droplets, but the fine droplets formed quickly flocculate to approximately the same effective size as was present before homogenization. The non-homogenized milk has large droplets but these are largely non-flocculated. Optical micrographs of the homogenized and non-homogenized samples reveal more large droplets in the former and more flocculation in the latter (Figure 2). A likely explanation for this is the amount of protein capable of stabilizing the emulsion is limited in coconut milk (del Rosario and Punzalan 1977). As the particle size is reduced, the interfacial area increases and a single protein molecules originally adsorbed to 1 droplet ends up simultaneously adsorbing to the surface of 2 droplets, leading to bridging flocculation (McClements 1999).

In other experimental work, the effect of homogenization pressure on the effective and primary particle diameter of coconut milk was measured (Figure 3). Samples of freshly extracted coconut milks were homogenized at 20/2, 40/4, and 60/6 MPa (stage I pressure/stage II pressure) for 1 to 5 passes through the homogenizer. Homogenization reduced both the effective and primary particle sizes of coconut milk by about 50% to 75%. Increasing the homogenization pressure marginally decreased the effective particle size, and after the 3rd pass through the homogenizer, subsequent passes had no further effect on particle size. This supports our hypothesis that the amount and quality of protein limits the effectiveness of homogenization on coconut milk.

Figure 4 shows the mean droplet diameter of both non-homogenized and homogenized coconut milks heated at different temperatures. The effective particle size of homogenized coconut milk increased dramatically (from about 10 μm to more than 22.7 μm) after heating at above 70 °C for 1 h, whereas the primary particle size increased only after heating at 90 °C. For non-homogenized samples, effective particle size changed from 12.2 to 30.5 μm when the heating temperature increased. Significant changes in the primary particle size are also detected at higher heating temperatures. Both the effective and primary particle size increased in non-homogenized and homogenized coconut milks heated at temperatures above 70 °C, suggesting that both flocculation and possibly a slight degree of coalescence occurred in heated coconut milk emulsions. This is supported by observations of the microstructure of the heated emulsion samples (Figure 2). Just as solutions of globular proteins sometimes gel if thermally denatured, protein-stabilized emulsions have been shown to flocculate after heating as the protein-protein associations formed bind the droplets together in a network (Sliwinski and others 2003). Coconut proteins have been shown to denature and coagulate at 80 °C and higher (Gonzalez 1990; Kwon and others 1996); it seems likely that the denaturation and aggregation of surface-bound proteins is responsible for the thermally-induced flocculation seen here. Coalescence (seen as a change in primary particle size) may then result due to the breakdown of the lamella separating the flocculated droplets as the effectiveness of the proteins as stabilizing agents is reduced.

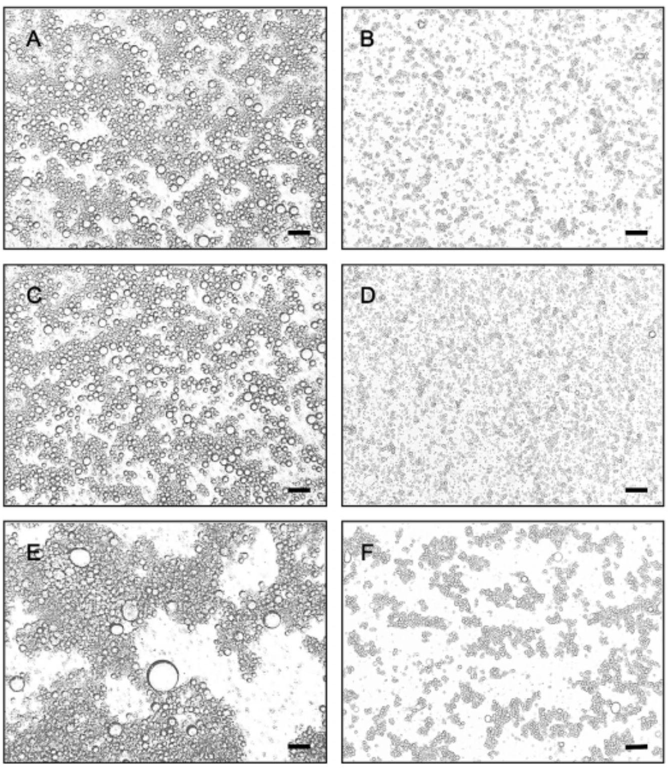

Figure 2—Micrographs taken of non-homogenized (a, c, e) and homogenized (at 40/4 MPa) (b, d, f) coconut milks either unheated (A, B) or heated to heated to 50 °C (C, D) or 90 °C (E, F). Scale bar is 50 μm.

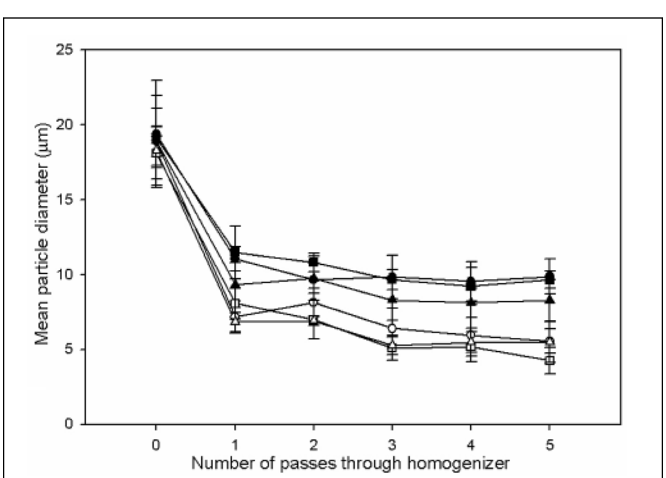

Figure 3—Effect of homogenization on the mean particle diameter of coconut milks homogenized at (●,○) 20/2, (■,□) 40/4, and (▲,△) 60/6 MPa. Filled points represent emulsions dispersed in water; open points represent emulsions dispersed in sodium dodecyl sulfate (SDS).

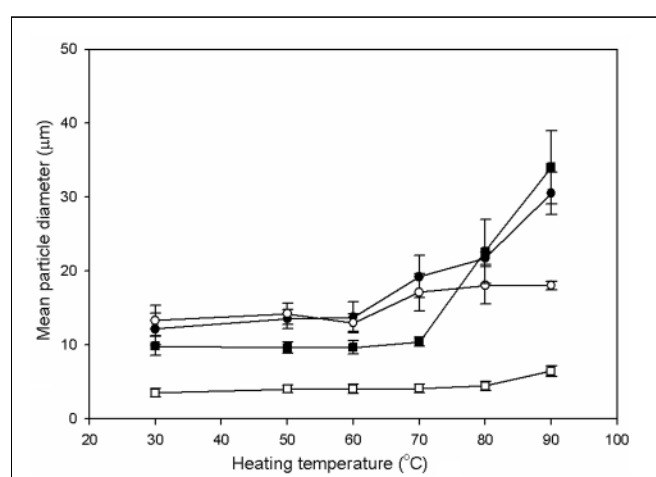

Figure 4—Effect of heating temperature on the mean particle diameter of (●,○) non-homogenized and (■,□) homogenized (at 40/4 MPa) coconut milks. Filled points represent emulsions dispersed in water; open points represent emulsion dispersed in sodium dodecyl sulfate (SDS).

Rheological measurements showed that both the non-homogenized and homogenized coconut milk samples were shear-thinning fluids whose apparent viscosity decreased with increasing shear rate. Similar flow behavior was reported in previous studies (Vitali and others 1986; Simuang and others 2004). The flow curves were modeled using a power law equation ($\tau = K.\gamma^n$, where τ is the shear stress, γ the shear rate, K is the consistency index, and n is the flow behavior index the shear stress. Power-law equations are frequently used to describe emulsion rheology, and the K and n parameters are often used to describe the inherent structure of whatever weak network is present and how readily it is disrupted by shear, respectively (McClements 1999). In all cases, the power-law equation described the data well ($r^2 > 0.98$), and values of K and n are reported in Figure 5.

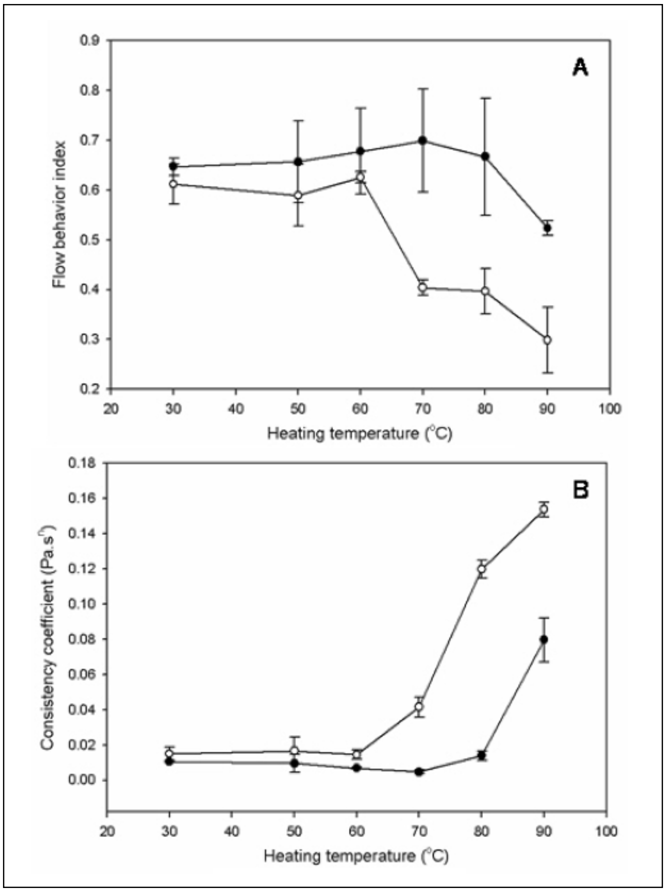

Figure 5—Effect of heating temperature on the power-law flow behavior index, n (a) and consistency index, K (b) of (●) non-homogenized and (○) homogenized (at 40/4 MPa) coconut milks

The homogenized samples were more viscous than the non-homogenized coconut milks, consistent with the presence of flocculated droplets. Emulsion flocculation leads to a higher effective particle volume fraction and thus higher viscosity (McClements 1999). The consistency index increased with temperature for both homogenized and unhomogenized samples probably due to additional thermally induced flocculation. The flow behavior index decreased with temperature as the structures formed could be readily disrupted by applied shear.

The creaming indices for the non-homogenized and homogenized coconut milk samples are shown as a function of thermal history in Figure 6. All samples creamed after 24 h storage, but the heated samples separated less as indicated by the lower serum heights and, thus, the lower creaming indices. Non-homogenized coconut milk is more prone to creaming than homogenized coconut milk because of its larger globule size (Monera and del Rosario 1982). The large but flocculated particles present in homogenized milk cream more slowly because the density contrast of a floc is smaller than that of a droplet and secondly because large flocs can be extensively interconnected and trap the droplets in a network (Parker and others 1995). The large network necessary to inhibit creaming may not be seen in light scattering measurements from a diluted emulsion as the process of sample preparation disrupts the fragile structures. Better creaming stability was found in both non-homogenized and homogenized coconut milks after heating at temperatures above 80 °C for 1 h, probably due to the higher viscosity in these samples slowing the creaming rate (McClements 1999) or the differences in the structure of the creamed layer (Chanamai and McClements 2000).

Fink and Kessler (1985) argued that oil extraction by organic solvent (in native milk fat globules) is a measure of the ability of the interfacial layer to protect the fat against the destabilization process. They also argued that degradation of this interfacial layer could be inferred from changes in the amount of extractable oil. In our experiments, more than 80% of oil in fresh coconut milk could

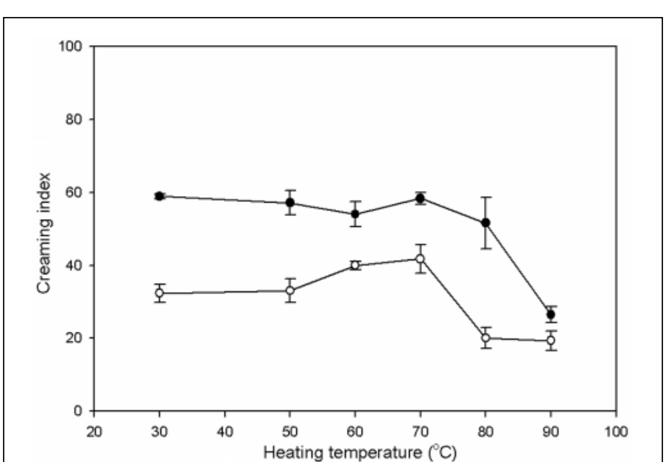

Figure 6—Effect of heating temperature on the creaming index of (●) non-homogenized and (○) homogenized (at 40/4 MPa) coconut milks

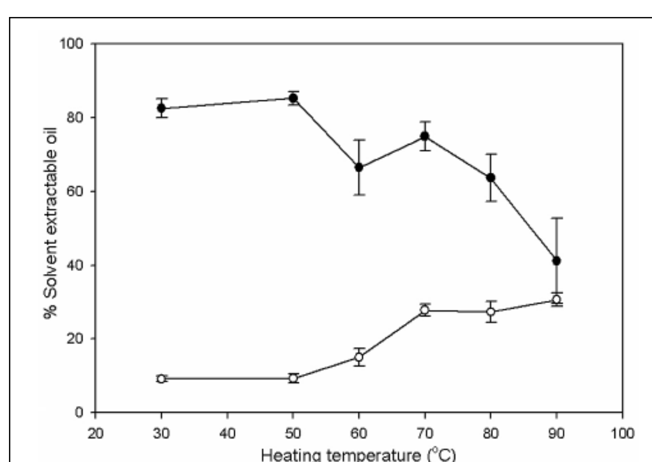

Figure 7—Effect of heating temperature on the free oil solvent-extracted from (●) non-homogenized and (○) homogenized (at 40/4 MPa) coconut milks

be extracted by petroleum ether, whereas only about 10% of the oil from similar homogenized milks could be extracted (Figure 7). Homogenization reduces the primary particle size of the coconut milk and leads to extensive flocculation (Figures 1b, 2, and 4) and it seems likely that the droplets in the core of the flocs are more protected from the extracting solvent.

Thermal processing progressively decreased the amount of solvent-extractable oil in the non-homogenized samples while increasing it in the homogenized samples. The markedly changes were found at the heating temperature between 60 °C and 70 °C. When heated at 90 °C, the amount of oil can be extracted from the coconut milks was independent of the homogenization process. Heating the emulsion leads to protein denaturation. We hypothesize that, on heating the homogenized samples, the protein involved in bridging flocculation rearranges and is pulled away from 1 of the bridged droplets to expose some lipid surface and allow more fat to be solvent extracted. In the non-homogenized samples, the protein is not involved in bridging flocculation so it is not necessarily pulled away from the droplet surface on denaturation. The resultant flocculation of the non-homogenized samples is induced by protein-protein hydrophobic attractions and may serve to decrease the amount of solvent-extractable oil.

Conclusions

Coconut milk is a natural emulsion extracted with minimal pro cessing from fresh coconuts. The emulsion particle size is naturally of the order of 13.1 μm and can only be slightly reduced on homogenization as the quality and quantity of emulsifiers (probably protein) naturally present is low. The homogenized emulsions tend to be highly flocculated, probably by bridging flocculation, and this increases the product viscosity. Thermal treatment increases the degree of flocculation, probably because of protein-protein hydrophobic attractions following denaturation, and leads to increased effective particle size and apparent viscosity. The creaming stability of the emulsion improved on heating because the larger flocs can form a network in the more viscous emulsion. Solvent-extractable oil of non-homogenized coconut milk decreased with increasing heating temperature, whereas those of homogenized samples increased because of the differences in accessibility of the extracting solvent to the fat globules. The information obtained from the study provides a better understanding of the changes in stability of coconut milk emulsion during processing important in controlling coconut milk functionality.

Acknowledgments

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