

# Contribution of Particle Sizes and Particle Size Distributions in Crystalline Fractionation of Lard

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Particle sizes and particle size distributions of lard in the process of multiple-step crystalline fractionation were studied by natural sedimentation spectrophotometry according to Stokes's law and Beers's law. The average particle size diameter and their distributions of lard fractions varied with the crystallization temperatures. Average particle size decreased significantly ( $p < 0.05$ ) with the crystallization vessel cooling rates (0.5, 1.0, and 2.0 °C min<sup>-1</sup>) and agitator speeds (50, 100, and 150 rpm). Furthermore, the firmness of lard crystal fractions decreased with the vessel cooling rate and agitator speed. Decreases in average particle sizes in addition to the firmness of lard crystal fractions were related to the increase in unsaturated fatty acid and to their triglyceride compositions.

**Keywords:** Lard; crystalline fractionation; particle size; particle size distribution; firmness

## INTRODUCTION

In the context of diet and nutrition, fats serve as a source of energy, provide essential nutrients, and facilitate the absorption of fat-soluble vitamins. Fats are also necessary for aspects of texture, flavor, color, and overall acceptability of most forms of consumed food (Kinsella, 1988). The trend to reduce intake of cholesterol and saturated fat derived from edible animal fat has significantly decreased the consumption of related animal fat products. The functional properties of fat can be improved by physical or chemical treatments and allow this product to meet the requirement of food processing and of nutritional value. Fractionation separates fats into fractions with various physicochemical properties and the potential to serve as a shortening, a hardening agent for margarine, or cooking oil. Crystalline fractionation is one of the convenient physical methods to separate original fats into plastic fats and liquid oil by cooling (Amer *et al.*, 1985) and is becoming an important method for utilization of edible animal fat. Lard is characterized by a unique flavor and plays a major role in Chinese-style baking. Although the annual consumption of edible lard is 12 million kilograms in Taiwan, the performance of various lard products for specific consumption has been little studied. Extension of lard versatility for food processing has been strongly encouraged and has become the major solution for utilizing this abundant edible porcine byproduct.

Plastic fats are involved in different types of aggregation with formation of a three-dimensional network structure of crystals in which liquid oil is trapped (Haighton, 1976). The plasticity of a fat is determined by the shape (deMan *et al.*, 1992), graininess (Vaisey-Genser *et al.*, 1989), polymorphism of triglyceride (Hernqvist, 1990), and triglyceride compositions (Bornaz *et al.*, 1993) of the crystals. However, little information about the contribution of particle sizes and their distributions and the changes in composition in crystalline fractionation to the texture of lard fractions has been published.

Dynamic light scattering technology is a quick method for determining the size of fat globules in milk model emulsion (Robin and Paquin, 1991), but this requires that the concentration of scatter be sufficiently diluted that only single scattering takes place. To avoid multiple scattering occurring, which interferes with the result observed, the sample must be diluted to the point where it is optically almost clear (Hallette, 1994); therefore, the two major problems to overcome in the use of light scattering are dilution of the sample to reduce the particle in a given volume to a level where measurement becomes possible and the low precision in observation of particle size diameter compared to several micrometers (Thomas, 1991).

Generally the extinction efficiency of particles is a function of particle size and refractive index as well as wavelength of the light beam. Natural sedimentation operates by gravitational settling. The particles settle at a rate determined by their size and density, and the particle size and relative concentration of particle can be calculated from known parameters. The particle settling rate is described by Stokes's law

$$v = (\rho - \rho_0)D^2/18\eta \quad (1)$$

where  $\rho$  and  $\rho_0$  represent a specific gravity for a particle and a solvent, respectively. The  $\eta$  and  $D$  represent the viscosity for a solvent and particle size diameter. The settling rate ( $v$ ) is also related to sedimentation depth,  $H$ , and sedimentation time,  $t$ . Therefore, the settling rate can be modified as

$$H/t = (\rho - \rho_0)D^2/18\eta \quad (2)$$

Furthermore, the particle size diameter can be given from eq 2:

$$D^2 = 18\eta H/(\rho - \rho_0)t \quad (3)$$

Turbidimetry is a generally applicable method for measurement. The turbidity,  $\tau$ , of a uniform dispersion illuminated by monochromatic light with incident intensity  $I_0$  is given by

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$$I/I_0 = \exp(-\tau l) \quad (4)$$

where  $I$  is the transmitted intensity and  $l$  is the path length. Therefore, the transmittance  $T$  at time  $t$  is given by

$$T(t) = 1/l \ln I_0/I(t) = 1/l \ln 1/\tau(t) \quad (5)$$

The turbidity is related to the particle diameter  $D$  and to the number of particles  $N$  in the light path at time  $t$

$$\ln I_0/I(t) = k l N(t) D^2 \quad (6)$$

The particle size distribution is given by

$$N(t) = 1/k l D^2 \ln I_0/I(t) \quad (7)$$

The objective of this study is to observe the effect of the operation conditions for crystalline fractionation of lard on the particle sizes and particle size distributions determined by natural sedimentation spectrophotometry.

## MATERIALS AND METHODS

**Preparation of Lard.** Porcine backfat was excised from fresh prechilled porcine carcasses and then was finely ground by using a meat grinder (Syeldemann ME-130, Germany) fitted with a 5-mm stainless steel plate. To provide consistency in fat composition, a batch of 1000 kg of raw material was rendered, accomplished by a rotary dry rendering cooker (Harrsiev Maskinfabrik A/S, Denmark) for all experiments. Crude lard oil was collected sequentially by means of filtering the dregs and horizontal centrifugation after rendering. Refined lard was made in a local fat rendering plant and followed by refrigerated storage for further studies.

**Crystalline Fractionation of Lard.** A modification of the multiple-step crystalline fractionation method proposed by Kaylegian and Lindsay (1992) was utilized. Melted lard was poured in a 2-L stainless steel jacketed vessel and water circulated by a Lauda K-20 refrigerated water bath. A marine type impeller with a 45° pitched-propeller agitator was attached to a Eyela DC-RT (Rikaikikai, Japan) drive unit, and the motor speed was maintained at 50, 100, and 150 rpm, respectively, for all processes. Lard was allowed to cool at a 0.5, 1, and 2 °C min<sup>-1</sup> to crystallization temperature, respectively, with continuous agitation after equilibrating at 50 °C for 30 min. After the crystallization temperature was approached, the vessel temperature and water bath were held at that temperature for 8 h, respectively. Changes in water and melted lard temperature were monitored with a thermocouple connected with a digital recorder (Yokogawa Electronic, Japan), respectively. Fat crystal mass was separated from the liquid fraction by vacuum filtration through Whatman No. 1 filter paper in a chamber that was maintained at that crystallization temperature after the crystallization was complete. The liquid fraction was then re-equilibrated at 50 °C and subsequently cooled to lower temperature to recrystallize. This process was repeated at consecutively lower temperatures, and fat crystal masses were produced at 35, 31, 27, 24, 21, and 15 °C, respectively.

**Determination of Particle Size and Particle Size Distribution in Crystalline Fractionation.** Preparation of the fat crystal samples for determining the particle size and particle size distribution was accomplished by a modification of the Chawla and deMan (1991) method. To avoid the fluctuation of temperature affecting the results to be observed, all samples, reagents, and apparatuses were kept isothermal at the crystallization temperature in the process of treatments or observations. After the crystallization was complete, 1 mL of samples in the crystallization vessel were suspended in 10 mL of isobutanol ( $d = 0.8060$ ;  $n = 1.3976$ ; and  $\eta(20\text{ °C}) = 1.2560$ ) in a tube and shaken gently and then kept in a water bath set at the crystallization temperature for 30 min. To reduce the interference of refractile effect from liquid fat, the upper clear layer of the liquid in samples was removed by

pipetting carefully. The fat crystal layers were resuspended in 10 mL of isobutanol. Particle sizes and particle size distributions were measured by use of a helium–neon laser light scattering spectrophotometer (LPA-3000, Otsuka Electronics Co., Japan) equipped with a thermoelectric cell holder and a temperature controller with temperature programming capability. The particle counter was connected to an LPA-3100 software correlator (Otsuka Electronics). Data were calculated by built-in software and the particle sizes and particle size distributions of lard and lard fractions were obtained.

**Measurement of Lard Fraction Firmness.** Forty grams of lard and its fractions were collected in nine vials (height, 40 mm; diameter, 20 mm), stored at 4 °C for 12 h, and then kept in a 15 °C waterbath for 1 h prior to measurement. Samples were measured by a Foudo Rheometer (NRM-2010J, Foudo Co., Japan) fitted with a flat circular stainless steel punch at a speed of 5 mm min<sup>-1</sup>. Firmness was defined as force in grams exerted by the samples instantly after 3-mm compression. The measurement was carried out with nine samples for each fraction, and the average was calculated.

**Determination of Fatty Acid and Triglyceride Compositions.** *Analysis of the Fatty Acid Composition.* Methyl esters of the fatty acids from the lard fractions were prepared using the 0.5 M sodium methanide–methanol transesterification procedure as Christie (1982) described. One microliter methyl ester samples were analyzed by a Hitachi 263-50 gas chromatograph equipped with a flame ionization detector. GC conditions were as follows: a fused silica capillary column, phase DB-Wax (30 m × 0.25 mm i.d., 0.25- $\mu$ m film thickness; J&W Scientific Inc, Rancho Cordova, CA); temperature programming from 120 °C for 4 min to 250 °C for 6 min at 2 °C/min rise; injector, 260 °C; detector, 280 °C; carrier gas, nitrogen at 25 mL min<sup>-1</sup>. The chromatograms were recorded on a Hewlett-Packard 3396 integrator. Correction factors were determined by analysis of an oil standard reference mixture of fatty acid methyl esters (AOSC No. 6, Sigma Chemical Co., St. Louis, MO).

*Analysis of the Triglyceride Composition.* The triglyceride compositions were determined by dissolving 15 mg of lard and the fractions in 6 mL of *n*-heptane and injecting 0.2  $\mu$ L into a fused silica capillary column, phase Crossbond 35% dimethyl 65% diphenyl polysiloxane (RTX-65TG, 30 m × 0.25 mm i.d., Restect Co., PA). The same gas chromatograph was used as for fatty acid analysis. The GC conditions were as follows: oven temperature programming from 340 °C for 5 min to 365 °C at 1 °C min<sup>-1</sup> rise; injector, 380 °C; detector, 400 °C; carrier gas, nitrogen 30 mL min<sup>-1</sup>. Identification of triglycerides according to carbon number was made by comparison of the retention time to the standard mixture of simple triglyceride from C<sub>42</sub> to C<sub>54</sub> (Sigma).

**Statistical Analysis.** Data were analyzed by repeated measure analysis of variance with the General Linear Model procedure of the Statistical Analysis System. Comparison of treatment means was based on Duncan's multiple-range test.

## RESULTS AND DISCUSSION

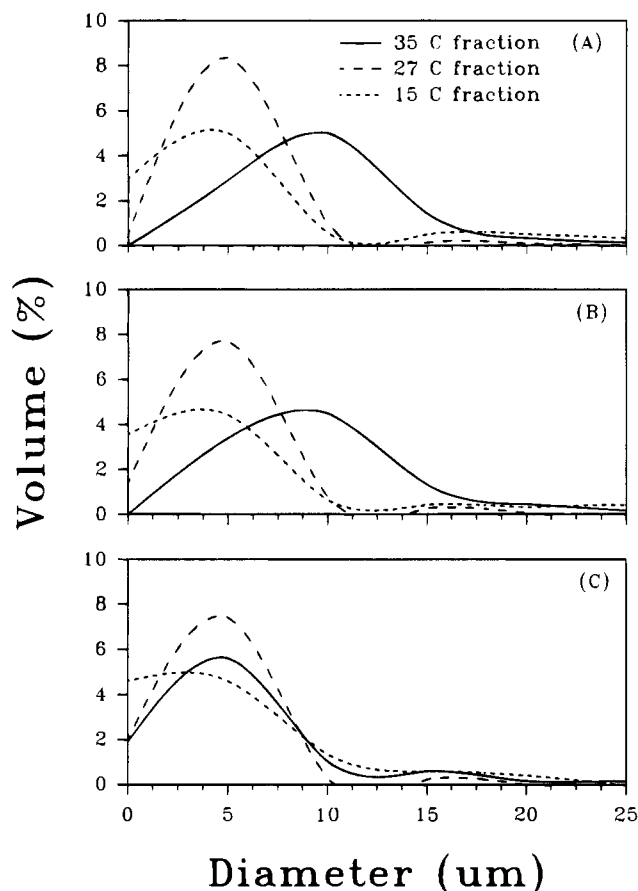
**Effect of Cooling Rate and Agitator Speed on Particle Sizes and Their Distribution of Lard Crystal Fractions.** Six lard crystal fractions are produced at a 0.5 °C min<sup>-1</sup> vessel cooling rate and a 50 rpm agitator speed. Significant differences in peak temperature in the melting curves among the 35, 27, and 15 °C crystal fractions (46.3, 30.3, and 12.6 °C, respectively) are observed by differential scanning calorimetry (DSC); hence, these three samples are selected for discussion. The appearance of 35, 27, and 15 °C lard crystal fractions is in a solid, semisolid, and meltdown state in a 25 °C incubator for 4 h, respectively.

The effect of crystallization vessel cooling rate on the average particle size diameter of lard crystals in the process of multiple-step crystalline fractionation is shown in Table 1. The average size of three lard crystal fractions decreases with the increasing cooling rate at

**Table 1. Average Particle Size<sup>a</sup> of Lard Crystals Fractionated at Various Vessel Cooling Rates and Agitator Speeds**

lard fraction	vessel cooling rate <sup>b</sup> (°C min <sup>-1</sup> )			agitator speed <sup>c</sup> (rpm)		
	0.5	1.0	2.0	50	100	150
35	16.8	14.6	10.4	16.8	10.2	7.4
27	13.6	12.8	9.8	13.6	7.4	3.2
15	9.2	6.9	4.1	9.2	4.6	1.3

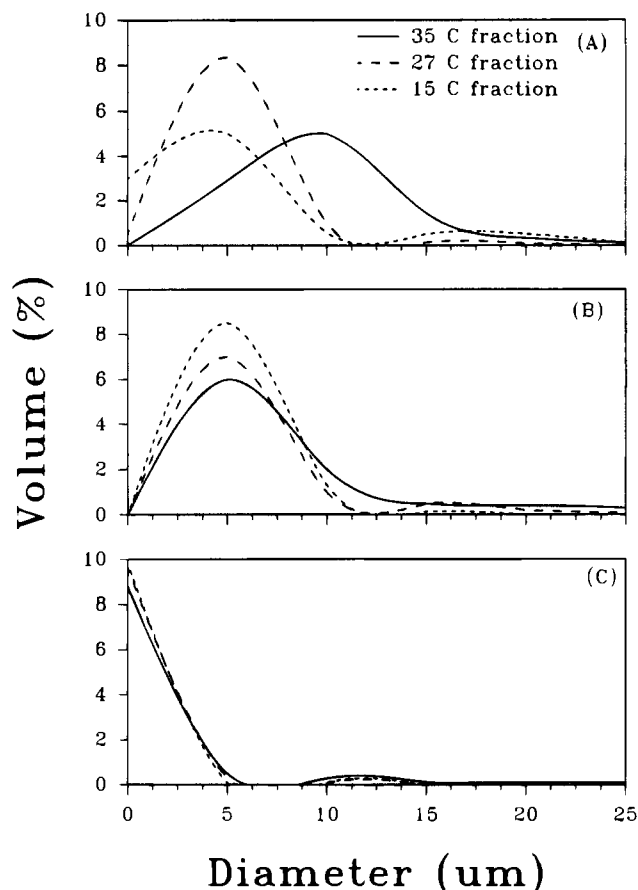
<sup>a</sup> Size of diameter (mm). <sup>b</sup> Agitator speed at 50 rpm. <sup>c</sup> Vessel cooling rate at 0.5 °C min<sup>-1</sup>.



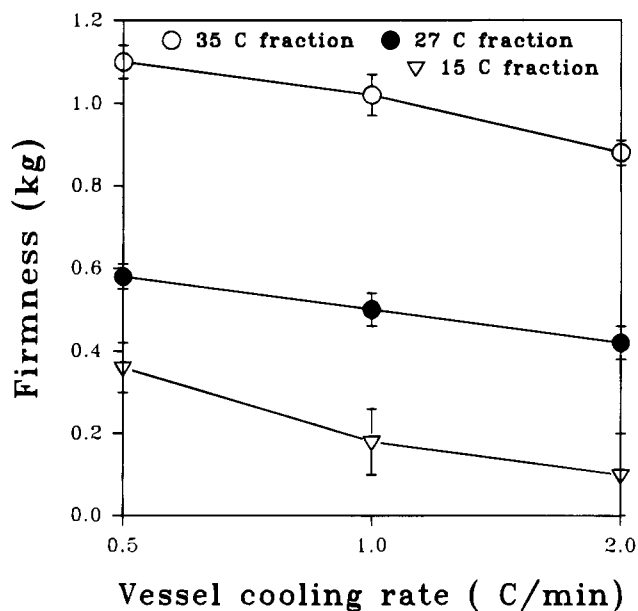
**Figure 1.** Change of the average particle size of lard crystal fraction produced at various vessel cooling rates [(A) 0.5 °C min<sup>-1</sup>; (B) 1 °C min<sup>-1</sup>; (C) 2 °C min<sup>-1</sup>] and a 50 rpm agitator speed.

50 rpm agitator speed. The effect of cooling rate on the particle size distributions of lard crystals fractionated at various crystallization temperatures is shown in Figure 1. At a 2 °C min<sup>-1</sup> vessel cooling rate (Figure 1C), the volume of particle sizes >15 μm is markedly lower than at 0.5 and 1 °C min<sup>-1</sup>. Fat crystallization involves several complicated thermodynamic and physical chemistry equilibria in the process of cooling (Sato and Garti, 1988). As Herrera *et al.* (1992) indicated, at a lower cooling rate there was sufficient time for the triglycerides to interact with each other and form a solid solution, eutectics, and pseudocompounds. Therefore, we suggest that a stable environment with the least fluctuating temperature is beneficial to the formation of large crystals in the process of crystallization at a lower cooling rate.

The effect of agitator speed on the average particle size of lard crystals crystallized at a 0.5 °C min<sup>-1</sup> vessel cooling rate is also shown in Table 1. At all crystallization temperatures, the average sizes of lard crystals significantly decreased ( $p < 0.05$ ) with agitator speed. By contrast, yield of lard fractions (data are not shown)

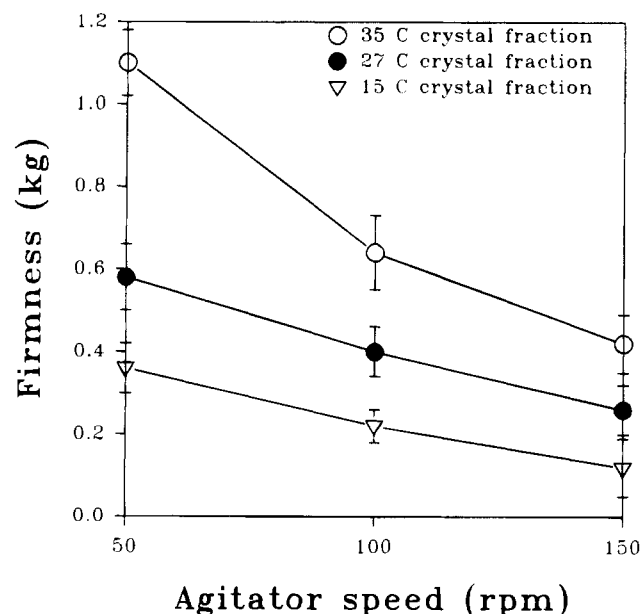


**Figure 2.** Change of the average particle size of lard crystal fraction produced at various agitator speeds [(A) 50 rpm; (B) 100 rpm; (C) 150 rpm] and a 0.5 °C min<sup>-1</sup> crystallization vessel cooling rate.

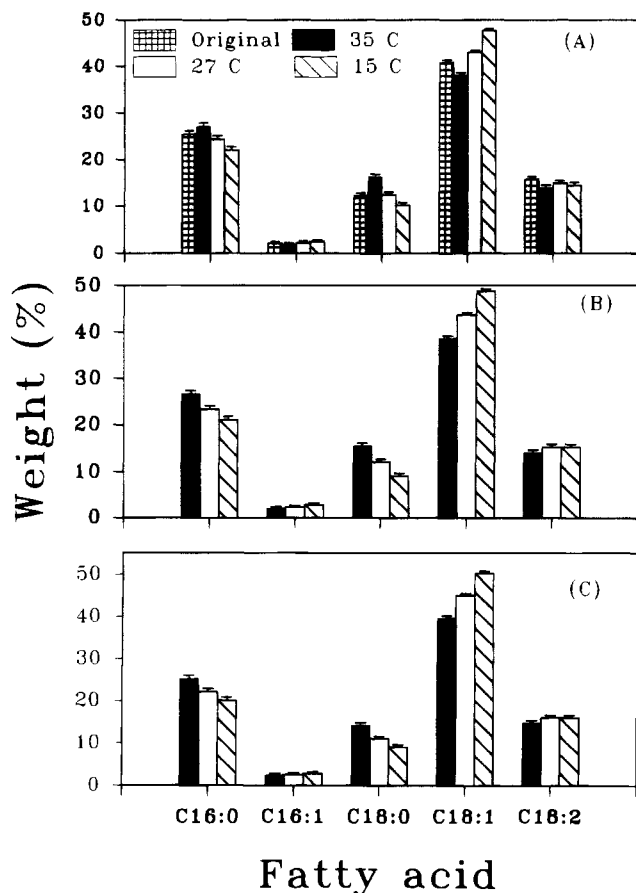


**Figure 3.** Change of the firmness of lard crystal fraction produced at various vessel cooling rates and a 50 rpm agitator speed.

increased with agitator speed. Nevertheless, at a crystallization temperature <27 °C, the higher the agitator speed, the more difficult it was to collect the fat crystals by filtration. In the 35 and 27 °C lard fractions, the extent of decrease in average crystal size is from 16.8 and 13.6 μm (50 rpm) to 7.4 and 3.2 μm (150 rpm), respectively. The volume of particle size >10 μm was less pronounced (Figure 2B,C) but increased

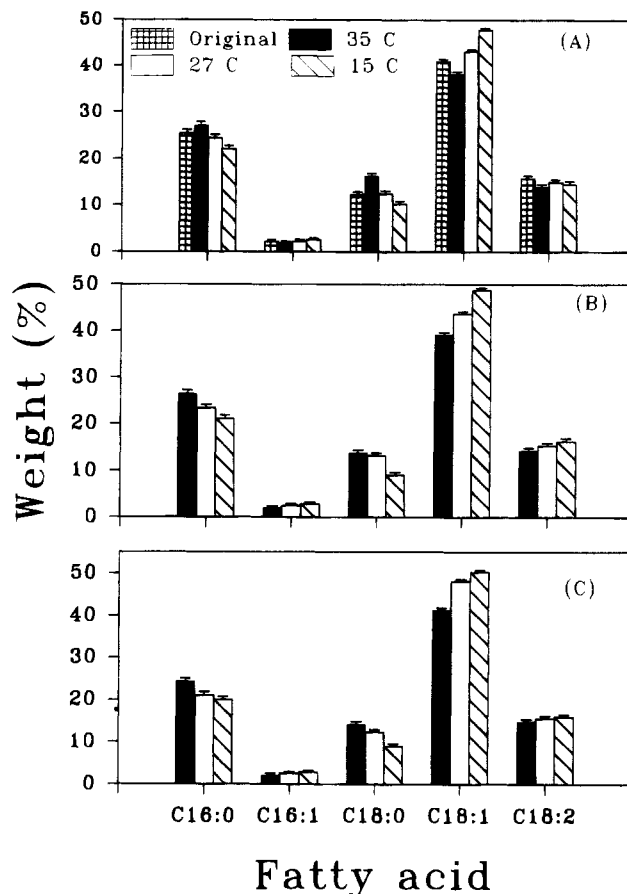


**Figure 4.** Change of the firmness of lard crystal fraction produced at various agitator speeds and a  $0.5\text{ }^{\circ}\text{C min}^{-1}$  crystallization vessel cooling rate.



**Figure 5.** Change of the fatty acid composition of lard crystal fraction produced at various vessel cooling rates [(A)  $0.5\text{ }^{\circ}\text{C min}^{-1}$ ; (B)  $1\text{ }^{\circ}\text{C min}^{-1}$ ; (C)  $2\text{ }^{\circ}\text{C min}^{-1}$ ] and a 50 rpm agitator speed.

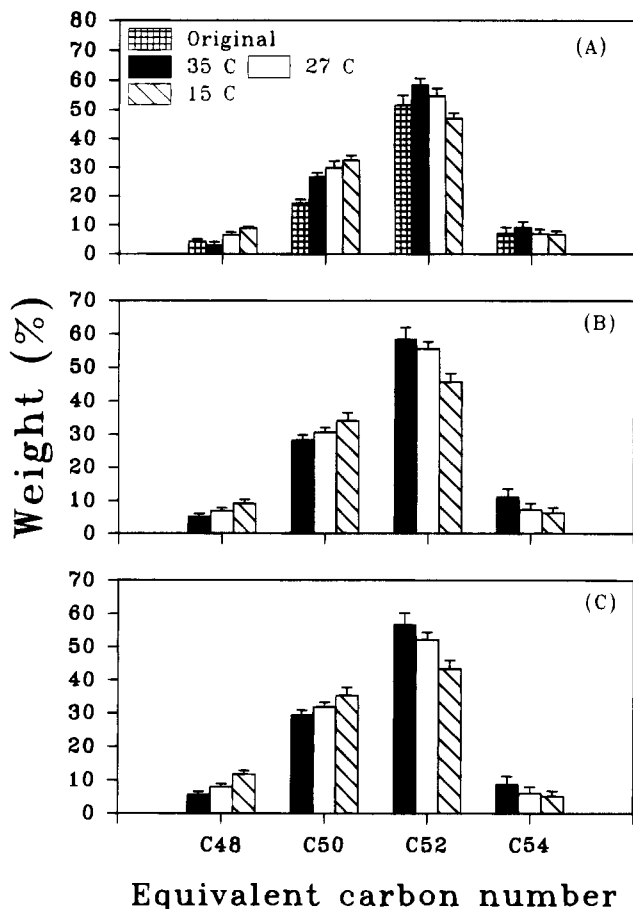
obviously in range of  $<5\text{ }\mu\text{m}$  with agitator speed. Agitation may enhance contact between crystals, nucleation, and growth of fat crystals in the process of crystallization and prevent destruction of the primary crystal structure from the thermal variation (Hartel, 1992); furthermore, fat crystals are stabilized by van der Waals–London forces (Haighton, 1963). Nevertheless, fat crystals may destabilize and partially solubilize



**Figure 6.** Change of the fatty acid composition of lard crystal fraction produced at various agitator speeds [(A) 50 rpm; (B) 100 rpm; (C) 150 rpm] and a  $0.5\text{ }^{\circ}\text{C min}^{-1}$  crystallization vessel cooling rate.

in liquid oil (Herrera and Anon, 1991) and form an unstable polymorphic crystal with vigorous agitation. As Shi *et al.* (1989) described, a high fluid shear may cause disruption of crystal surfaces in the lactose crystal growth. Particle size and particle size distributions of the lard crystal fractions decreased with agitator speed and may result from growing crystal structure destabilization.

**Effect of Cooling Rate and Agitator Speed on the Firmness of Lard Crystal Fractions.** The firmness of lard fractions varied with crystallization temperatures, and the values are lower with the lower crystallization temperatures. Changes in firmness of lard crystal fractions produced at various vessel cooling rates are shown in Figure 3. The firmness in both the 35 and 27 °C fractions significantly ( $p < 0.05$ ) decreases at a  $2\text{ }^{\circ}\text{C min}^{-1}$  vessel cooling rate, decreasing 20.0% and 26.7%, respectively. The firmness of the crystal fraction produced at 15 °C decreases with the cooling rate. The effect of agitator speed on firmness in 35, 27, and 15 °C lard crystal fraction is shown in Figure 4. All lard crystal fractions significantly ( $p < 0.05$ ) decrease with agitator speed; furthermore, a pastelike appearance of the samples produced at 15 °C and agitator speed  $>50\text{ rpm}$  is observed. Solid fat is a mixture of innumerable tiny fat crystals and liquid oil trapped in the spaces between the fat crystals. Therefore, the particle size of fat crystals plays an important role in the firmness or consistency of fat (Chawla and deMan, 1990). On the basis of the results mentioned above, the firmness of lard crystal fractions is related to their average particle size and size distribution. The decrease in firmness is also proposed to result from the

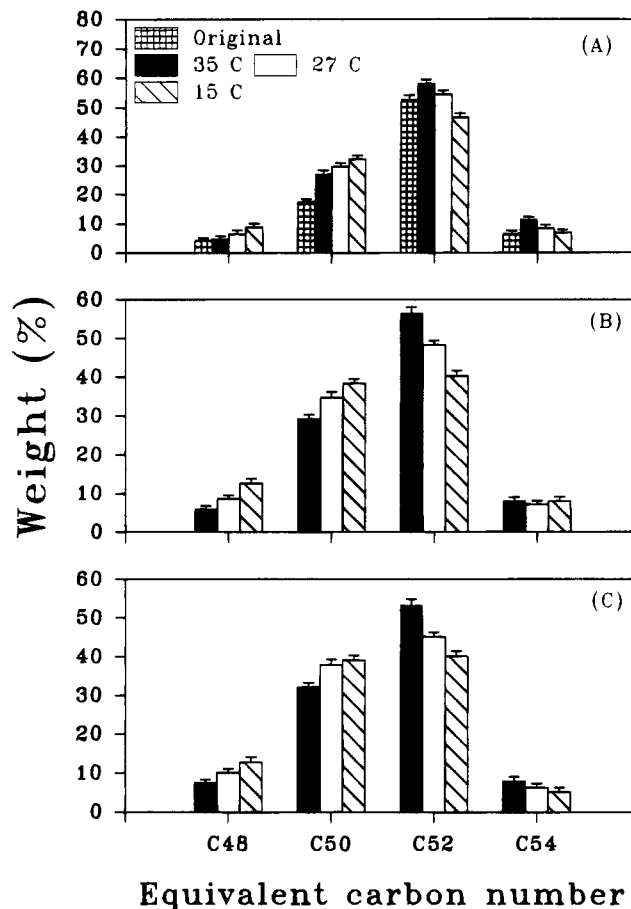


**Figure 7.** Change of the triglyceride composition of lard crystal fraction produced at various vessel cooling rates [(A)  $0.5\text{ }^{\circ}\text{C min}^{-1}$ ; (B)  $1\text{ }^{\circ}\text{C min}^{-1}$ ; (C)  $2\text{ }^{\circ}\text{C min}^{-1}$ ] and a 50 rpm agitator speed.

disruption of fat crystals as well as composition of higher content of liquid oil at a higher agitator speed.

**Changes of Fatty Acid and Triglyceride Compositions in Lard Crystal Fractions.** To comprehend the contribution of chemical characteristics to the firmness and formation of particle sizes of lard crystal fractions, fatty acid compositions and triglyceride compositions are determined. The major fatty acids in all samples, regardless of processing conditions, are palmitic acid (16:0), palmitoleic acid (16:1), stearic acid (18:0), oleic acid (18:1), and linoleic acid (18:2). A similar phenomenon in the change of fatty acid compositions in all lard crystal fractions is observed, except in the 35  $^{\circ}\text{C}$  fraction. The saturated fatty acid compositions decrease but the unsaturated fatty acids increase when compared with the original lard. The changes in fatty acid composition in the process of the lard crystalline fractionation at various cooling rates are shown in Figure 5. In the 35  $^{\circ}\text{C}$  fraction, the 16:0 and 18:0 contents are significantly ( $p < 0.05$ ) higher than in the original lard and other crystal fractions at 0.5 and 1  $^{\circ}\text{C min}^{-1}$  cooling rates, respectively. No marked change in 16:1 and 18:2 content, respectively, is observed when compared with the two higher cooling rates; however, the unsaturated fatty acid composition decreases. In contrast, the unsaturated fatty acid composition in the 27 and 15  $^{\circ}\text{C}$  fractions increases with the cooling rate, and a significant ( $p < 0.05$ ) elevation is observed at a 2  $^{\circ}\text{C min}^{-1}$  cooling rate. In addition, the ratio of unsaturated fat to saturated fat in the lard crystal fractions produced at 27 and 15  $^{\circ}\text{C}$  is significantly higher than that of the original lard and the 35  $^{\circ}\text{C}$  fraction.

The observation of fatty acid compositions in lard



**Figure 8.** Change of the triglyceride composition of lard crystal fraction produced at various agitator speeds [(A) 50 rpm; (B) 100 rpm; (C) 150 rpm] and a  $0.5\text{ }^{\circ}\text{C min}^{-1}$  crystallization vessel cooling rate.

crystal fractions produced at various agitator speeds is shown in Figure 6. The changes of the fatty acid compositions vary with the treatment. Furthermore, the unsaturated fatty acid content increases with the agitator speed for all fractions. The higher melting component in the mother liquid oil is fractionated at a higher crystallization temperature in a multiple-step crystalline fractionation (Banks *et al.*, 1985). The fatty acid composition in the fat crystals plays a significant role in the performance, *e.g.* spreadability, meltdown, and sensory characteristics, of shortening and margarine (Kaylegian and Lindsay, 1992; D'Souza *et al.*, 1991; deMan *et al.*, 1992; Rohm and Raaber, 1992). Generally, the content of unsaturated and saturated fatty acids in fat relates to the melting (Grompone, 1985, 1989) and textural properties (Danmark and Bagger, 1989). However, the mechanism of fatty acid distribution of lard crystal fractions in the process of crystallization is still uncertain. The decrease in firmness of the lard crystal fractions is parallel to the increase in unsaturated fatty acid composition as shown Figures 6 and 7. The change in content of oleic acid (18:1) is highly correlated ( $R = 0.98$ ) with the firmness of the lard crystal fractions.

The equivalent carbon numbers (ECN) of triglyceride in original lard and the lard crystal fractions produced from multiple-step crystallization at various vessel cooling rates and agitator speeds are shown in Figure 7. Four major triglycerides, C<sub>48</sub>, C<sub>50</sub>, C<sub>52</sub>, and C<sub>54</sub>, in the original lard are observed. The results are similar to a lard shortening fractions by organic solvent (deMan *et al.*, 1992). The content of C<sub>52</sub> and C<sub>54</sub> triglycerides in the 35  $^{\circ}\text{C}$  fraction is obviously higher than in the original lard, the 27  $^{\circ}\text{C}$  fraction, and the 15  $^{\circ}\text{C}$  fraction.

However, the content of C<sub>52</sub> and C<sub>54</sub> triglycerides decreases with the vessel cooling rate among the lard crystal fractions, while the content of C<sub>48</sub> and C<sub>50</sub> triglycerides increases with cooling rate. Similar evidence is also observed in the lard crystal fractions produced by higher agitator speeds as shown in Figure 8. The content of C<sub>52</sub> and C<sub>54</sub> triglycerides significantly decreases with agitator speed regardless of the lard crystal fraction. Moreover, changes in the firmness of the lard fraction are positively related to the variation of the C<sub>52</sub> ( $R = 0.94$ ) and C<sub>54</sub> ( $R = 0.88$ ) triglycerides. However, little information about the triglyceride composition distributions and their contribution to the firmness and particle size distribution in the fat crystallization is reported. Bornaz *et al.* (1993) suggest that the triglyceride composition as well as iodine number and fatty acid composition is a reliable indication of the textural properties of the butter. The crystal size and the thermal characteristics of a selected triglyceride is related to the triglyceride carbon number by polar light microscopy (deMan *et al.*, 1985); furthermore, the higher content of long-chain triglycerides in the fat fraction and the greater appearance of the sample will be inclined to solid state (Arul *et al.*, 1987; Merkle and Larick, 1993). The change of the C<sub>52</sub> and C<sub>54</sub> triglyceride content is consistent with the visible performance of these lard crystal fractions.

Therefore, we suggest that the average particle size of the lard crystal fractions varies with the crystallization temperatures and the processing conditions. Changes in the samples are related to their particle sizes and their size distributions. With an increase in the unsaturated fatty acid content, the C<sub>52</sub> and C<sub>54</sub> triglyceride content is parallel to the decrease in the firmness of the lard crystal fractions in the process of the crystallization.

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