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Non-triglyceride components modulate the fat crystal network of palm kernel oil and coconut oil

Xiuhang Chai[a], Zong Meng[a], Jiang Jiang[a], Peirang Cao[a], Xinyu Liang[b], Michael Piatko[c], Shawn Campbell[c], Seong Koon Lo[b,c], Yuanfa Liu[a,*]

[a] State Key Laboratory of Food Science and Technology, Synergetic Innovation Center of Food Safety and Nutrition, School of Food Science and Technology, Jiangnan University, 1800 Lihu Road, Wuxi 214122, Jiangsu, People's Republic of China
[b] Rich Products Corporation, 75 Suhong West Road, Suzhou 21520, Jiangsu, People's Republic of China
[c] Rich Products Corporation, One Robert Rich Way, Buffalo, NY 14213, United States

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ABSTRACT

PKO and CNO are composed of 97–98% triacylglycerols and 2–3% minor non-triglyceride components (FFA, DAG and MAG). Triglycerides were separated from minor components by chromatographic method. The lipid composition, thermal properties, polymorphism, isothermal crystallization behavior, nanostructure and microstructure of PKO, PKO-TAG, CNO and CNO-TAG were evaluated. Removal of minor components had no effect on lipid composition and equilibrium solid fat contents. However, presence of minor components did increase the slip melting point and promoted the onset of crystallization from DSC crystallization profiles. The thickness of the nanoscale crystals increased with no polymorphic transformation after removing the minor components. Crystallization kinetics revealed that minor components decreased crystal growth rate with higher $t_{1/2}$. Sharp changes in the values of the Avrami constant k and exponent n were observed for all fats around 10 °C. Increases in n around 10 °C indicated a change from one-dimensional to multi-dimensional growth. From the results of polarized light micrographs, the transformation from the coarser crystal structure to tiny crystal structure occurred in microstructure networks at the action of minor components.

1. Introduction

Palm kernel oil (PKO) and coconut oil (CNO) are rich in medium chain fatty acids (MCFA) and exhibit easy digestibility and absorbability. These outstanding properties make them useful for application in health food products. In addition, the high proportion of lauric acid in PKO and CNO gives their sharp melting properties, resulting in its application in fat-structured food materials, such as margarine, cocoa butter substitutes and ice cream (Pantzaris & Basiron, 2002).

PKO and CNO as fat-structured food materials could crystallize to form fat crystal network (Franke, Bindrich, & Heinz, 2015; Maleky, 2015; Ramel, Peyronel, & Marangoni, 2016), which impacts the mechanical and rheological properties of plastic fat. In many foods, the fat crystallization determines the consistency, plasticity sensory properties, physical stability, and appearance of fat-rich products (Marangoni et al., 2012). In addition, the presence of minor lipid components (diacylglycerols, monoacylglycerols and phospholipid) contributes significantly to the quality of various products (Maruyama et al., 2014). Therefore, it is imperative to study the influence of indigenous minor components on molecular structure, crystallization behavior, nano- and meso-scale structure of fat.

Fats and oils are mainly composed of triglycerides (> 95%) and small amounts of minor non-triglyceride components. The major minor components found in fats and oils include FFAs, MAGs, DAGs and PS (Smith, Bhaggan, Talbot, & van Malssen, 2011), which can influence the nucleation, the crystal growth, polymorphic transformation and microstructure (Patel & Dewettinck, 2015; Ribeiro et al., 2015; Smith et al., 2011). It has been reported that the crystallization kinetics was changed after removing minor compounds from milk fat (Wright, Hartel, Narine, & Marangoni, 2000) and that DAG increased the crystallization induction time of milk fat (Wright & Marangoni, 2002); while the overall kinetics of nucleation, crystal growth, and polymorphic transformation was delayed by the action of the minor components (Mazzanti, Guthrie, Sirota, Marangoni, & Idziak, 2004). In palm oil (PO), the high-melting monopalmitin (MP) initiated the crystallization process and induced a fractional crystallization of the PO triacylglycerols (TAG) (Verstringe, Danthine, Blecker, Depypere, & Dewettinck, 2013), and monoglycerides had the similar effect on the

* Corresponding author.
E-mail address: yfliu@jiangnan.edu.cn (Y. Liu).

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fully hydrogenated PO (Basso et al., 2010; Fredrick, Foubert, De Sype, & Dewettinck, 2008; Verstringe, Danthine, Blecker, & Dewettinck, 2014). Palm-based DAGs could also influence the nucleation and crystallization rate of PO (Saberi, Lai, & Toro-Vázquez, 2011). For CNO, dilauroylglycerol retarded nucleation while dioleoylglycerol had no significant effect (Gordon & Rahman, 1991). Also addition of lauric acid and low hydrophilic/lipophilic balance (low-HLB) sucrose esters also changed the isothermal crystallization kinetics of CNO (Chaleepa, Szepes, & Ulrich, 2010). Moreover, formation of smaller spherulites occurred for CNO after the emulsifier addition.

Despite the plethora of studies dealing with the influence of extraneous additives on crystallization, only few have focused on the impact of naturally existing minor components on fat crystal network. Therefore, the primary objective of this study was to investigate the influence of non-triglyceride components on the lipid composition, thermal properties, polymorphism, crystallization behavior, nanostructure and microstructure of PKO and CNO. In addition, the relationship between microstructure and crystallization kinetics were established for further analysis of the influence mechanism at the action of minor components.

2. Materials and methods

2.1. Materials

Palm kernel oil (PKO) (iodine value (IV) 17.61 g I_2/100 g and slip melting point (SMP) 27.12 °C) and coconut oil (CNO) (IV, 8.23 g I_2/100 g and SMP, 24.30 °C) were produced and donated generously by Kerry Specialty Fats Ltd. (Shanghai, China). Supelco 37 Component FAME mixture was purchased from Sigma-Aldrich China (Shanghai, China). All other chemicals and organic solvents of analytical or chromatographic grade purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China) and Fisher Scientific (Shanghai, China), respectively.

2.2. Analysis of triglycerides, diglycedides, monoglycerides and free fatty acid in PKO and CNO

The contents of triglycerides, diglycedides, monoglycerides and free fatty acid in PKO and CNO were determined using Normal Phase HPLC (NPHPLC, refractive index detector) using a phenomenex Luna column (250 mm × 4.6 mm, 5 μm particle size) according to the previous method (Zeng, Qi, Xin, Yang, & Wang, 2015). The mobile phase was n-hexane and 2-propanol (15:1, v/v). The flow rate was 1 mL/min. Peaks in HPLC were evaluated by comparison of their retention times with those of known standards.

2.3. Separation of triacylglycerols from PKO and CNO

Purification of PKO and CNO was achieved using column chromatographic method (Siew & Ng, 1999). Briefly, a chromatographic glass column (2.5 cm diameter, 40 cm length) equipped with a Teflon stopcock was packed with 30 g of silica gel. 30 g of oil dissolved in 120 mL of petroleum ether were loaded onto the column and eluted with solvents into two fractions: the TAG of samples and the minor non-triglyceride components. The PKO-TAG and CNO-TAG were eluted with 95:5 petroleum ether/ethyl ether and subsequently more polar lipid components were eluted with diethyl ether. The progress of separation was monitored by thin-layer chromatography (TLC), which was performed on silica gel GF 254 plates (Haiyang Co. Ltd., Qingdao, China) with a developing solvent of hexane/diethyl ether/acetic acid (80/20/1, v/v/v). The bands were visualized using iodine vapor. The same fractions collected from multiple runs were combined, and the solvent was evaporated under vacuum.

2.4. Sliding melting point (SMP)

The SMP was determined by use of the AOCS standards method Cc3-25 (AOCS, American Oil Chemists' Society, 2009).

2.5. Fatty acid (FA) composition

FA composition was analyzed using a gas chromatography (GC) system (GC-2010PLUS, Shimadzu, Tokyo, Japan) equipped with a flame ionization detector (FID) and a fused-silica capillary column (TR-FAME, 60 m × 0.25 mm × 0.25 μm) according to AOCS Official Method Ce 2-66 (AOCS, 2009). The temperatures of the injector and FID detector were set at 50 and 250 °C, respectively. The column was heated to 60 °C and held for 3 min, then programmed at 5 °C/min to 175 °C, held for 15 min, then increased to 220 °C at 2 °C/min, and held for 10 min. The FA acids were identified by comparing their retention time to those of the FAME standards.

2.6. TAG composition

TAG composition of different samples was measured according to AOCS Official Method Ce 5-86 (AOCS, 2009) using an Agilent 7820A Series GC system (Agilent, California, USA) equipped with FID detector and a high temperature capillary column (Rtx-65TG, 30 m × 250 μm × 0.1 μm). The column temperature was initially held at 250 °C for 1 min, and then increased to 280 °C at a rate of 20 °C/min. The column temperature was then increased to 340 °C at a rate of 10 °C/min, and finally increased to 350 °C at a rate of 1 °C/min and held at 350 °C for 20 min. The carrier gas was hydrogen, and the total gas flow rate was 40 mL/min. The temperatures of the FID and injector were 360 and 350 °C, respectively. TAG profiles were determined according to the retention times of TAG standards.

2.7. Thermal analysis

The thermal properties of different samples were performed using a differential scanning calorimeter (DSC 8500, Perkin Elmer, USA) equipped with a liquid nitrogen cooling system according to literature method (Meng et al., 2010). Samples (5–10 mg) were hermetically sealed in an aluminum pan with an empty pan serving as reference. Samples were heated to 80 °C for 10 min to ensure complete melting and erasure of all crystal memory, and the crystallization profiles were obtained by cooling to − 40 °C at 5 °C/min. After a 10 min hold at this temperature, the melting profile was obtained by heating to 80 °C at 5 °C/min. All DSC analyses were performed in triplicate.

2.8. Solid fat content (SFC)

SFC was measured by pulsed nuclear magnetic resonance (*p*NMR) with a Bruker PC120 series NMR analyzer (Bruker, Karlsruhe, Germany). Water bath was used for rapid cooling and offered accurate temperature control. The instrument was automatically calibrated by use of three standards (supplied by Bruker) with the solid content of 0, 31.3, and 74.6%. Approximately 2.5 g of samples was placed in each glass NMR tube for all *p*NMR experiments and was kept at 80 °C for 30 min to ensure complete melting and destroy any crystal memory, and then placed in a thermostated water bath set at 0, 5, 10, 15, 20, 25, 30, 35 and 40 °C. All measurements were performed in triplicate.

For crystallization kinetics, *p*NMR was used to monitor changes in SFC as a function of time at different crystallization temperatures (5, 10, 15, 20, 25, 30 and 35 °C) according to literature method (Wright, Hartel, Narine, & Marangoni, 2000). Samples were heated at 80 °C for 30 min before analysis to destroy any crystal history. Three replicates of each preheated sample in glass NMR tubes were determined at each crystallization temperature and the SFC readings as a function of isothermal crystallization time were recorded by the equipment software

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