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Characterization of the stearin obtained by thermal fractionation of anhydrous milk fat

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Abstract

Thermal fractionation of anhydrous milk fat with high initial content of low molecular weight TAGs was performed based on a 2² factorial experimental design with three central and two axial points. The independent variables were the crystallization temperature and the cooling rate. A 2.0 L agitated glass reactor was used and the rotation was kept at 20 rpm. The filtration was performed under vacuum (500 mmHg), using a 14 µm average pore diameter filter paper. Curves of isothermal crystallization of the stearin fraction showed that increasing the crystallization temperature, increases the induction period and decreases the maximum solid content of stearin. An increase of the crystallization temperature resulted in an increase in solid fat content. All runs achieved an efficient fractionation using a single separation step and stearin with defined characteristics can be obtained adapting the process conditions.

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1. Introduction

Anhydrous milk fat (butter oil) is widely used as additive in high-quality foods in order to provide smooth taste and pleasant odor on the human palate. Butter oil is considered the most complex fat mixture found in nature, containing hundreds of lipidic components resulting in a large range of melting point and cross-crystallizations [1]. Milk fat contains about 65% saturated fatty acids (mainly C16:0, C18:0 and

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C14:0) and about 35% unsaturated fatty acids (mainly C18:1). Samples of butter oil can vary in chemical composition and consequently in their physicochemical properties due to race and nutrition of the dairy cows as well as by seasonal and geographic factors [2]. Some of the functional characteristics of milk fat, like plasticity, hardness, and spreadability, can restrict its use in industrial scale.

Thus, many studies have been conducted in order to modify the milk fat chemical composition and therefore changing its chemical and physical properties. Thermal fractionation is the most recommended physical treatment, since it is a low cost process, not requiring solvents, uses simple equipments and retains most of the milk fat flavour [3]. In thermal fractionation the separation of the triacylglycerols (TAGs) is based on their melting points differences. The process consists of two main steps: crystallization and separation. Crystallization considers nuclei formation and crystal growth, and the separation is intended to remove the olein fraction (liquid part) from the stearin (crystallized fraction). A single stage of separation is usually adopted in order to obtain these fractions. The fractions have different physical and chemical properties and can be applied to different products depending on the purpose.

This study evaluates the characteristics of stearin obtained from brazilian samples of butter oil using an experimental design. The independent variables were the crystallization temperature (21°C to 27 °C) and the cooling rate (5 to 20 °C/hour). The stearin fractions were characterized by the TAGs composition and the solid fat content (SFC), measured by NMR. The difference between the solid fat content at 25 °C and at 35°C ($\Delta S_{25^{\circ}\text{C}-35^{\circ}\text{C}}$) and the value of SFC at 25°C ($S_{25^{\circ}\text{C}}$) were used as responses variables.

2. Materials and methods

2.1. Raw materials

The anhydrous milk fat used in the fractionation experiments was donated by Fonterra Brasil Ltda (Goiânia, GO, Brazil). At the time of use, the fat pails of 2 kg under refrigeration, was heated to 65°C.

2.2. Analytical Methodology

Triacylglycerol composition: determined using a capillary gas chromatograph Agilent 6850 (US), with a DB-17HT (50% methyl phenyl polysiloxan) 15 m column and internal diameter of 0.25 mm. Duplicate sample injections were performed, and the TAGs composition were calculated according to [4].

Solid fat content: SFC was determined by NMR, using a Bruker Minispec PC120 (Germany) and the direct method AOCS Cd 16b-93 [4].

Crystallization isotherms: samples were melted (100°C/15 min) and the increase in solid fat content as a function of the crystallization time was monitored by NMR Spectrometry, with the compartment stabilized at the pre-defined crystallization temperature [5].

2.3. Fractionation process

The fractionation was based on an experimental design with three central points and two axial points. The independent variables were the crystallization temperature (21, 22, 24, 26 and 27°C) and the cooling rate (5, 7.2, 12.5, 17 and 20°C/h). The conditions of each fractionation test are listed in Table 1.

The fractionation was performed in a system compose by two vessels, the crystallizer and the filtration device, both connected to a thermostatic bath. The crystallizer was a 2L jacketed glass reactor with a 20 cm anchor-paddle mechanical stirrer. Milk fat (2kg) was heated to 60°C for 15 minutes and then cooled at the previously defined rate, down to the crystallization temperature and maintained under agitation (20 rpm) for the pre-determined time. At the end of this step, a stopcock was opened and the product was transferred by gravity to the jacketed stainless steel receiving vessel of the filtration system. The mass

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