



## Influence of cocoa butter refining on the quality of milk chocolate

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### ABSTRACT

A refining step is often crucial for the removal of undesired components in fats and oils. More flexible refining technologies are required due to a global decline in cocoa butter quality and to meet industry's demand for cocoa butters with improved properties. The aim was to investigate the impact of the cocoa butter refining process on milk chocolate quality. Therefore a crude cocoa butter was subjected to a steam refining at different temperatures and this with or without a silica pretreatment. The major effect of the silica pretreatment was the complete removal of phosphorus (thus phospholipids), iron and alkaline components. During the steam refining step mainly Free Fatty Acids (FFA) were removed at increased temperatures ( $T \geq 200$  °C). The refining of the cocoa butter influenced the rheological properties of the chocolate. An increased packed column temperature, coinciding with the removal of FFA, resulted in a lower yield stress and a higher viscosity. Reduction of FFA positively influenced the crystallization kinetics and the formation of the crystal network, resulting in differences on a macroscopic scale.

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### 1. Introduction

As cocoa butter (CB) forms the continuous phase of chocolate, it has a major influence on the quality of the final product. Therefore, it is crucial that the CB has an optimal quality. Many processing steps precede the final CB pressing but it may still contain undesired components sometimes making it necessary to refine the product.

CB should contain less than 1.75% free fatty acids (FFA, based on oleic acid) to be in compliance with the [European Union Directive, \(2000\)](#) and needs to be free from off-flavors, molds and rancidity ([Calliauw et al., 2008](#)). The conventional CB refining process exists of a filtration followed by a batch or continuous deodorization. However, more flexible refining technologies have become necessary ([Vila Ayala et al., 2007](#)) because firstly, a global decrease in CB quality is noticed (high amount of FFA, more phosphorus and more alkalinity). Secondly, there is also an increased industry's demand for different types of CBs (e.g. in terms of color, degree of neutral flavors, melting profiles etc.). [De Greyt et al., 2003](#) suggested an improved three stage refining process. In the first step a pretreatment with silica is carried out to adsorb alkaline

components, impurities and phosphatides. An optional bleaching step can be applied to modify the color but this is only important for the production of white chocolate. In the final step packed column stripping or tray deodorization is performed. In a packed column, which is filled with structured packing material with a high surface area ( $250\text{--}350\text{ m}^2/\text{m}^3$ ), there is an intensive counter-current contact between the oil and the stripping steam resulting in a high stripping efficiency and an overall lower steam consumption. A so-called scrubber is attached to the packed column unit for the condensation of FFA. Another feature of the packed column is the shorter residence time which allows a higher process temperature (needed for the stripping of FFA) without unwanted side reactions (interesterification, isomerisation and formation of cyclic and trans fatty acids) ([Vila Ayala et al., 2007](#)). Therefore, this technique is very interesting for CB refining as the unique crystallization properties of CB should be preserved.

[Vila Ayala et al. \(2007\)](#) and [Calliauw et al. \(2008\)](#) did a thorough study on the effect of bleaching and/or packed column steam refining on CB properties. They concluded that there was no effect on the fatty acid profile, triacylglycerol (TAG) distribution and monoacylglycerol (MAG) and diacylglycerol (DAG) content, even up to temperatures of 260 °C. The FFA removal in relation to the oil temperature could be described by a sigmoid function. Furthermore, the bleaching step before steam refining efficiently removed the alkaloids, theobromine and caffeine. Steam refining around 200 °C also completely removed these alkaloids. It is known from industrial practice that these alkaloids tend to deposit in the vapor duct between the deodorizer and the vapor scrubber which may

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give rise to frequent cleaning of the equipment. In the study of the crystallization behavior of steam refined CB, it was concluded that the CB crystallized sooner and faster when FFA were removed.

Physical refining also has an impact on minor components in fats and oils. These may be desirable components such as tocopherols or undesirable ones such as pesticides or polycyclic aromatic hydrocarbons. The retention of the individual tocopherols decreases in the same order as their molecular weight, retention  $\alpha$ - > retention  $\gamma$ - > retention  $\delta$ -tocopherol (Cmolik et al., 2008). Timms and Stewart (1999) reported a typical reduction up to 15%, with a typical level in deodorized CB of 250 ppm.

The effect of the refining process on the physicochemical properties of CB has been described by different researchers (Calliauw et al., 2008; Timms and Stewart, 1999; Vila Ayala et al., 2007). However, information about the impact of different refining conditions of the CB on the final chocolate quality is limited.

In this research a crude cocoa butter with or without a silica pretreatment, was subjected to a steam refining in a packed column at five temperatures: 150, 175, 200, 225 and 250 °C. The impact of the refining treatments on the physicochemical properties was evaluated. In the next step the refined CBs were used to produce milk chocolate. Different quality characteristics such as particle size distribution (PSD), rheological behavior and texture were then examined to assess the influence of the refining treatment.

## 2. Materials and methods

### 2.1. Materials

The Malaysian crude CB was obtained from an industrial producer. The crude CB was thoroughly mixed and split in two parts. One part of the butter was treated with silica (0.5%) pretreatment. The refining was carried out in pilot plant equipment installed in the R&D Center of Desmet Ballestra Group N.V. (Zaventem, Belgium).

### 2.2. Physical refining

The silica pretreatment was carried out in a batch reactor according to the following procedure. The crude CB was first heated to 80 °C and 0.6% (v/w) of a 15% (w/w) citric acid solution was added and thoroughly mixed with the CB. After 15 min, 0.5% (w/w) silica powder (Trysil, Grace, Germany) was added and agitation continued for another 30 min. A vacuum was applied to remove the water. The silica was separated from the CB in a plate-and-frame filter mounted with a standard filter cloth by pressurizing the reactor with compressed air.

The crude (non pretreated) and silica pretreated CBs were stripped in a pilot packed column unit (internal column diameter: 7.6 cm, packing height: 2 m) filled with a structured packing with a specific surface area of 250 m<sup>2</sup>/m<sup>3</sup>. CB was pumped at a rate of 13 kg/h and 1% stripping steam was injected. A top pressure of 3 mbar was applied and the pressure drop over the packing was 0.5–0.75 mbar. Estimated residence time is approx. 7 min. CB temperature was varied between 150 and 250 °C with a 25 °C interval.

### 2.3. Physicochemical characterization

The fatty acid (FA) profile was analyzed by GC according to the AOCS Official Method Ce 1c-89 (Firestone, 1997). The fatty acid methyl esters of the acylglycerols and FFA were prepared as described in Official Method Ce 2-66 (Firestone, 1997). Mono, di- and triacylglycerol distribution of the butter was determined by

HPLC according to the AOCS Official Method Ce 5b-89 (Firestone, 1997), and further described in Vila Ayala et al. (2007).

The amount of FFA was determined by titration according to the AOCS Official Method Ca 5a-40 (Firestone, 1997) and expressed as oleic acid. The alkalinity was determined as sodium oleate by the titrimetric method described in the official AOCS method Cc 17-75 (Firestone, 1997). The quantification of phosphorus and iron in oil was done by using inductively coupled plasma optical emission spectroscopy (ICP-OES) as described in the AOCS Official Method Ca 20-99 (Firestone, 1997). Tocopherols were determined with normal phase HPLC as described by the AOCS official method Ce 8-89 (Firestone, 1997). Samples were analyzed on a HP series 1050 chromatograph with FLD (Hewlett Packard, Avondale, PA, USA) using a 0.5% (v/v) 2-propanol in hexane mobile phase. Theobromine and caffeine contents were determined by a reversed phase HP series 1050 chromatograph (Hewlett Packard, Avondale, PA, USA). A HP UV-Visible detector at 272 nm was used as detector. A detailed description of the method is given by Vila Ayala et al. (2007). The oil stability index (OSI) of the CB samples was determined according to the Rancimat method as described more in detail in the AOCS Official Method Cd 12b-92 (Firestone, 2009). The color was determined in an automatic Lovibond PFX 880/P provided with a heater to avoid solidification of the CB. The analysis was performed at 70 °C and the color was expressed in the CIELAB coordinates: the lightness L\* (0: black to 100: white), a\* (negative values indicate green while positive values indicate red) and b\* (negative values indicate blue and positive values indicate yellow).

The DSC experiments were performed on a Q1000 DSC with Refrigerated Cooling System and autosampler System (TA instruments, New Castle, USA). It was calibrated with indium (TA instruments, New Castle, USA), azobenzene (Sigma-Aldrich, Bornem, Belgium) and undecane (Acros organiscs, Geel, Belgium) prior to analysis. Nitrogen was used to purge the system. A CB sample (5–15 mg) was hermetically filled in alodined aluminum pans. An empty pan was used as a reference. The sample was held isothermal at 65 °C for 10 min to insure a liquid state and to erase the crystallization memory of the sample. Then, it was cooled at 10 °C/min to 20 °C and held there for 240 min. Each analysis was executed in triplicate. The kinetics of the CB crystallization can be described by means of mathematical models. Foubert et al. (2002) developed a model for fat crystallization based on CB crystallization kinetics. The model was fitted to the integral of the main crystallization peak observed in DSC.

### 2.4. Chocolate production

The milk chocolates were produced in the UGent Cacaolab. Table 1 shows the standard recipe of the milk chocolate used in this study. Low fat cocoa powder with additional CB was used in preference to cocoa mass to reduce confounding effects from CB found within the cocoa mass (containing between 50–55% CB). In each production, a batch of 3.5 kg of chocolate was prepared. In the first step, sugar, milk powder, cocoa powder and a part of the CB were mixed in a planetary Vema mixer BM 30/20 (Vemaconstruct, NV machinery Verhoest, Izegem, Belgium). CB was added to obtain a fat content of 27%. In the next step, particle size was reduced by refining on an Exakt 80S 3-roll mill (Exakt Apparatebau GmbH & Co. KG, Norderstedt, Germany). The temperature of the rolls was set at 35 °C. The refined product was then conched in a Bühler Elk'olino conche (Richard Frisse GmbH, Bad Salzufflen, Germany). Conching consisted of two phases: a dry and a liquid conching phase. In the dry phase, the temperature was first set at 55 °C while mixing clockwise at 1200 rpm during 120 min. The next step consisted of a shearing phase of 240 min at 65 °C with anticlockwise rotation at 1200 rpm. In the liquid phase, the

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