# Effect of refining degree on particle size, sensory and rheological characteristics of anhydrous paste for ice creams produced in industrial stirred ball mill 

Marcello Fidaleo ${ }^{\text {a }}$, Nicoletta A. Miele ${ }^{\text {b, * }}$, Stefania Mainardi ${ }^{\text {c }}$, Vincenzo Armini ${ }^{\text {d }}$, Roberto Nardi ${ }^{\mathrm{e}}$, Silvana Cavella ${ }^{\mathrm{b}, \mathrm{d}}$<br>${ }^{\text {a }}$ Department for Innovation in Biological, Agro-food and Forest Systems, University of Tuscia, 01100 Viterbo, Italy<br>${ }^{\mathrm{b}}$ Center of Food Innovation and Development in the Food Industry, University of Naples Federico II, 80055 Portici, Italy<br>${ }^{\text {c }}$ MEC3-OPTIMA SRL, 47832 San Clemente, Italy<br>${ }^{\text {d }}$ Department of Agricultural Sciences -University of Naples Federico II, 80055 Portici, Italy<br>${ }^{\mathrm{e}}$ RO.NA. Food Technology Consulting, 00049 Velletri, Italy

## A R T I C L E I N F O

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#### Abstract

Refining of an anhydrous hazelnut and cocoa based paste for ice cream in a stirred ball mill was characterized in terms of energy use and product viscosity, particle size and sensory characteristics. With a specific milling power of 0.03 kW per kg of paste, the fineness of practical interest, $22.4-30.4 \mu \mathrm{~m}$, was reached in 225-150 min, respectively. The pastes showed a time-independent pseudoplastic behavior with $n$ and $K$ in the range $0.62-0.69$ and $14.2-27.2 \mathrm{~Pa} \mathrm{~s}^{\mathrm{n}}$, respectively. The particle size distribution (PSD) curves, as determined by laser diffractometry, were mainly unimodal, with $90 \%$ percentiles [ $D(\mathrm{v}, 0.9)$ ] higher than the micrometer readings, suggesting the presence of flat particles, as confirmed by scanning electron microscopy. Most of the textural attributes showed a small difference among the samples with fineness $22.4-30.4 \mu \mathrm{~m}$, thus suggesting a product with fineness, $\mathrm{D}(\mathrm{v}, 0.9)$ and specific energy equal to $30.4 \mu \mathrm{~m}, 41.8 \mu \mathrm{~m}$ and $0.074 \mathrm{kWh} / \mathrm{kg}$, respectively, as optimal.


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## 1. Introduction

Nut creams can be defined as complex systems made up of different solid particles (sugar, cocoa powder, milk whey, milk powder, nuts, etc.) dispersed in a continuous fluid (oil) (Glicerina, Balestra, Pinnavaia, Dalla Rosa, \& Romani, 2013). They include nut butters and nut spreads that contain at least 90 and $40 \mathrm{~g} / 100 \mathrm{~g}$ nut ingredients (Shakerardekani, Karim, Mohn Ghazali, \& Chin, 2013), respectively, but also other nut based products, like sweet creams used as filling, anhydrous pastes for ice cream preparation or spreadable products, such as hazelnut chocolate based spreads. Textural, color, and flavor properties of these products play a major role in consumer appeal, buying decisions and eventual consumption (Di Monaco, Giancone, Cavella, \& Masi, 2008). Depending on the specific product, the most desired characteristics are a good

[^0]spreadability across a wide temperature range, a creamy taste, no fat-phase separation and a good oxidative stability.

Anhydrous pastes based on nut creams used for the preparation of ice creams are close to nut based spreads for the recipe, but they are not spreadable across a wide temperature range and become more liquid than a spread at $20-25^{\circ} \mathrm{C}$, this being obtained by varying their fat phase composition (Birkett, 2009). They could also resemble a filling cream, that is generally fat and sugar based and represents an important component in different confectionary foods, in which it provides taste, texture and adhesion of the baked items (Miele, Di Monaco, Masi, \& Cavella, 2015).

The production process of nut creams requires a step for the reduction of solid particle size. The main aim of this unit operation, called refining, is to reduce the solids to a specific particle size that depends on the specific product. For example, in the case of chocolate, the size of largest solid particles has a direct influence on rheological (flow properties) and sensory (mouthfeel grittiness) properties. Chocolate with particle size above $35 \mu \mathrm{~m}$ becomes gritty or coarse in the mouth resulting in lower acceptability (Petković, Pajin, \& Tomić, 2013).

Refining is mainly carried out with roll refiners, typical of the chocolate industry, or, more recently and for small productions, with stirred ball mills. In this last process, nut cream ingredients to be milled are fed into a jacketed tank containing the grinding media, typically stainless steel balls. The refining product and the balls are then agitated by a rotating shaft with arms. The product can also be recycled through the ball mill several times (Alamprese, Datei, \& Semeraro, 2007). During this process of mixing, grinding and recirculation, the size of the solid particles reduces and their surface is wrapped by the fat phase (Petković et al., 2013), conferring to the cream its rheological and textural properties.

The rheological behavior of this kind of product is affected both by several process parameters (temperature, agitation rate, milling time) (Petković et al., 2013) and by formula composition (Glicerina et al., 2013; Lončarević et al., 2016). These factors act also on the particle size distribution of the product under refining (Lončarević et al., 2016). The same considerations can be applied to chocolate refining in stirred ball mills (Alamprese et al., 2007; Lucisano, Casiraghi, \& Mariotti, 2006). Since the particle size of the product affects overall perception of the final product, it is really important to optimize this attribute (Birkett, 2009). An accurate definition of target particle size of the product and corresponding method of measurement are essential, especially to avoid. Not only does overgrinding reduce productivity but also has significant implications for energy consumption, considering that the energy required to reduce particle size rises dramatically as particle size set point decreases.

In literature, it was not possible to find any work dealing with the refining of anhydrous paste for ice cream or similar products in stirred ball mills. Thus, the main objective of this work was to evaluate the effect of stirred ball mill refining degree on particle size, sensory and rheological characteristics of this product and also on specific energy consumption.

## 2. Materials and methods

### 2.1. Materials

Cocoa and hazelnut based anhydrous paste samples were produced in an Italian confectionery factory using an industrial plant (Mazzetti Renato Srl, Italy) as described below. The following formulation with reference to 100 g of paste was used: seed oil $(27 \mathrm{~g})$, hazelnut paste ( 8 g ), cacao powder ( 11 g ), milk powder and derivatives $(7.5 \mathrm{~g}$ ), lecithin ( 0.6 g ), sugar ( 31 g ), ice sugar ( 14 g ), other ( 0.9 g ).

### 2.2. Grinding equipment, operation and energy consumption

The paste production line allowed the production of 2400 kg for each production cycle. It was composed of a mixer, a holding tank, three ball mills and a tank to store the refined paste. The stirred ball mills (model WAFA400, Mazzetti Renato Srl, Italy) allowed the production of batches of 400 kg of paste. They consisted of jacketed cylindrical tanks containing $9.5-\mathrm{mm}$ diameter stainless steel balls, a vertical shaft with horizontal arms for stirring and a recycling pump. Refining was carried out at a starting temperature of $42^{\circ} \mathrm{C}$, at an agitator shaft speed of 50 rpm , recycling the mass through the bed of balls at a flow rate of $4640 \mathrm{~kg} / \mathrm{h}$.

At various time intervals ( $60,90,120,150,195,225 \mathrm{~min}$ ) during the process, samples of 1 kg of refining paste were withdrawn and stored at $20^{\circ} \mathrm{C}$ in air-tight plastic containers before being used for the sensory analysis, the determination of the rheological behavior and measurement of the particle size. At the same time intervals the electric current flowing into the motor connected to the vertical shaft was measured by a digital clamp meter (model ELD200N,

Eldes Instruments Srl, Italy).
The power $(\mathrm{P})$ absorbed by the asynchronous three-phase motor connected to the stirring shaft assembled with the ball mill was calculated according to Eq. (1) (Alamprese et al., 2007):
$P=V I \sqrt{3} \cos (\phi)$
where V and I are the electric voltage and current and $\cos (\phi)$ is the power factor of the motor ( 0.84 in this case).

The specific energy ( E ) consumed by the ball mill for grinding as a function of time ( t ) was calculated according to Eq. (2):
$E=\frac{\sum_{i=1}^{i=n} V I_{i} \sqrt{3} \cos (\phi) t_{i}}{M}$
where the summation index $i$ refers to the refining time interval, $\mathrm{I}_{\mathrm{i}}$ and $t_{i}$ to the corresponding electric current and time, $M$ to the mass of refining paste.

### 2.3. Particle size measurement

A Mastersizer laser diffraction particle size analyzer equipped with Hydro 3000 dispersion unit (Malvern Instruments, Worchestershire, UK) was used. About 0.1 g of paste was analyzed at ambient temperature ( $20 \pm 2^{\circ} \mathrm{C}$ ). The sample was added into the disperdant (vegetable oil) until $18-20 \%$ obscuration was obtained. Stirring was conducted to ensure that particles were independently dispersed. Size distribution was quantified as relative volume of particles in size bands and presented as size distribution curves. Several indexes of the particle size distribution (PSD) based on the volume of particles were estimated, including the volume weighted mean $D[4,3]$, and the 10,50 and 90 percentiles $D(v, 10), D(v, 50)$ and $D(v, 90)$, respectively.

For each refining time two different samples were analyzed and for each sample 15 measurements were performed.

In the production area, the micrometer method was used to measure the particle size of refining paste. Samples of the refining paste were withdrawn from the ball mill and analyzed immediately in five replicates by a digital micrometer (model IP65, Mitutoyo, IL, USA). A second measurement procedure consisted in dispersing the paste sample (approximately 1 g ) in 1 g of white mineral oil before being placed on the jaws of the micrometer (Lucisano et al., 2006).

### 2.4. Viscosity measurement

Viscosity of paste samples was determined by a rotational rheometer ARES-LS (Rheometrics Inc., Piscataway, NY). The flow curves were carried out at $30{ }^{\circ} \mathrm{C}$ using a plate-plate system. In steady state conditions, after a pre-shearing of 500 s at $2 \mathrm{~s}^{-1}$, the apparent viscosity was measured as a function of increasing shear rate from 2 to $50 \mathrm{~s}^{-1}$ ( Iramp ) within 180 s , then of decreasing from 50 to $2 \mathrm{~s}^{-1}$ (II ramp) (Glicerina, Balestra, Dalla Rosa, \& Romani, 2016). Between the two ramps samples were sheared for 30 s at $50 \mathrm{~s}^{-1}$.

The obtained flow curves were fitted with the model of Ostwald de Waele (Power Law model), as reported by Glicerina et al. (2013). Three replications for each sample were performed.

### 2.5. Sensory analysis

Texture profile was performed with a panel of nine trained judges. Judges were trained for 10 h . Approximately 10 g of each sample was served in two different containers (plastic petri dishes and cups), identified by three-digit random codes and presented in a monadic sequential order. The assessors were provided with a

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[^0]:    * Corresponding author. Center of Food Innovation and Development in the Food Industry, University of Naples, Federico II. Via Università 133, 80055 Portici, Italy.

    E-mail address: nicolettaantonella.miele@unina.it (N.A. Miele).

