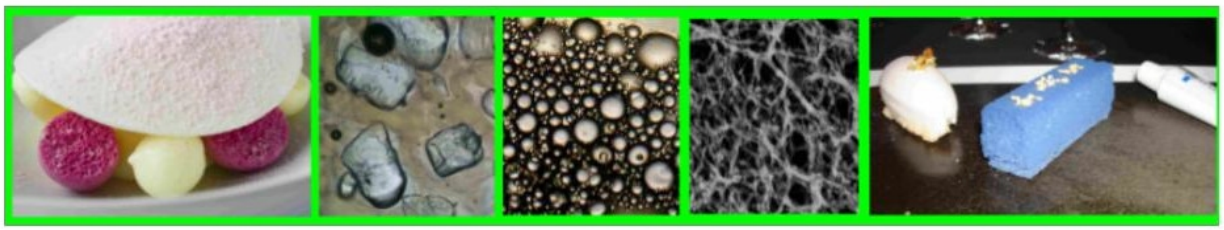

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Image for thought

The blooming in a dark chocolate

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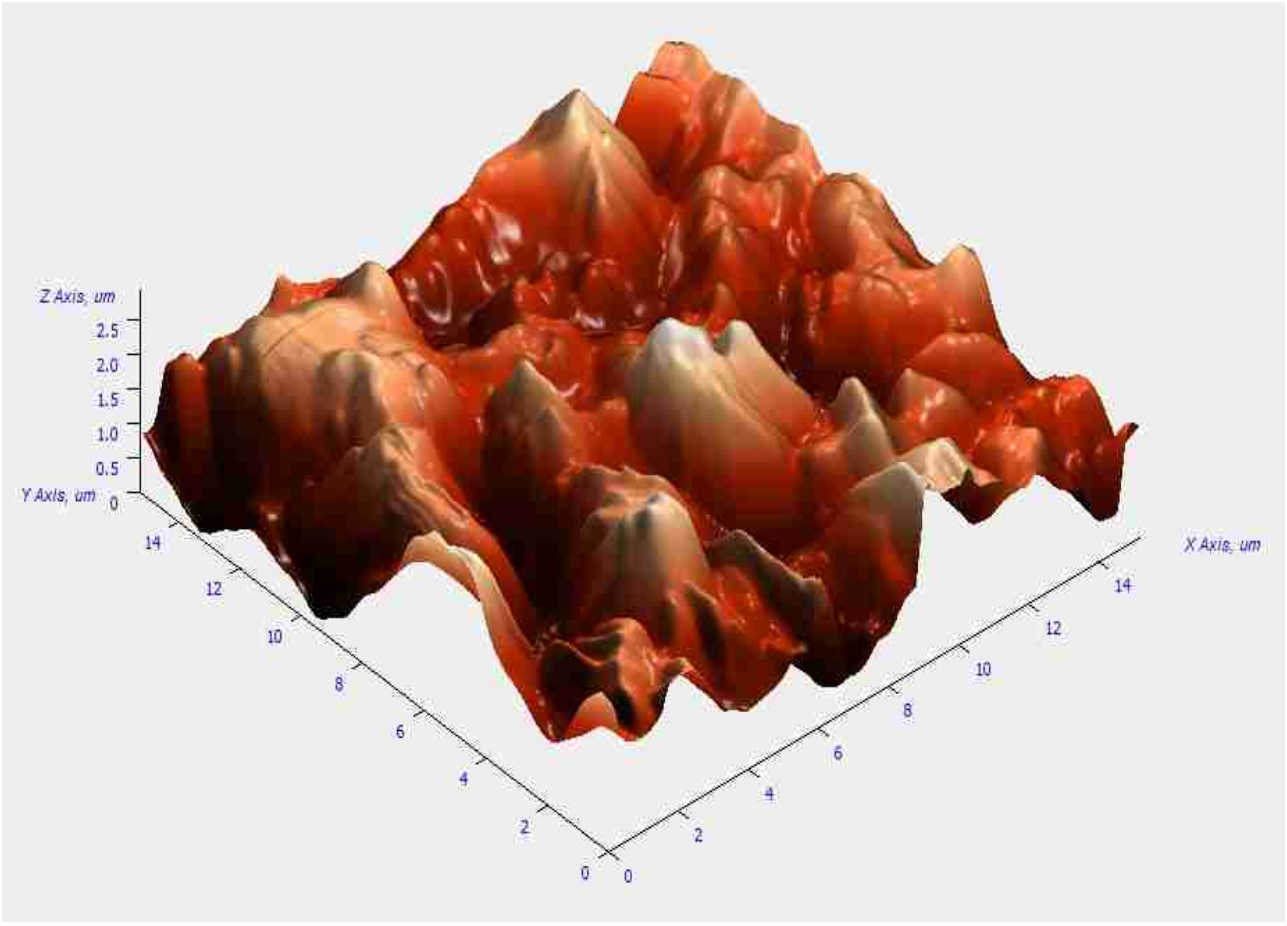

The main raw material for chocolate is cocoa beans which is rich in triglycerides with high melting temperature (Loisel *et al.*, 1998), and in bioactive compounds as flavonoids (Magrone *et al.*, 2017). To produce chocolate, fresh cocoa beans are submitted to fermentation and drying at farm level. Cocoa bean fermentation change polyphenols, phytosterols, free fatty acids, fatty acid profile and volatile compounds into beans, conveniently to chocolate production (Calvo *et al.*, 2021).

Chocolate is produced with varying amounts of cocoa solids (cocoa liquor, cocoa butter and cocoa powder), produced from cocoa beans, according to formulations and chocolate types (dark, milk and white, among others) (Shafi *et al.*, 2018). Traditionally, the dark chocolate contains at least 35 % of total cocoa solids, sugar and sometimes milk products, but, more recently the content of cocoa solids can exceed 80% (Merlino *et al.,* 2021).

To produce chocolate, the cocoa solids are mixed with sugar, milk, or other ingredients and the ensuing processing steps for obtaining the

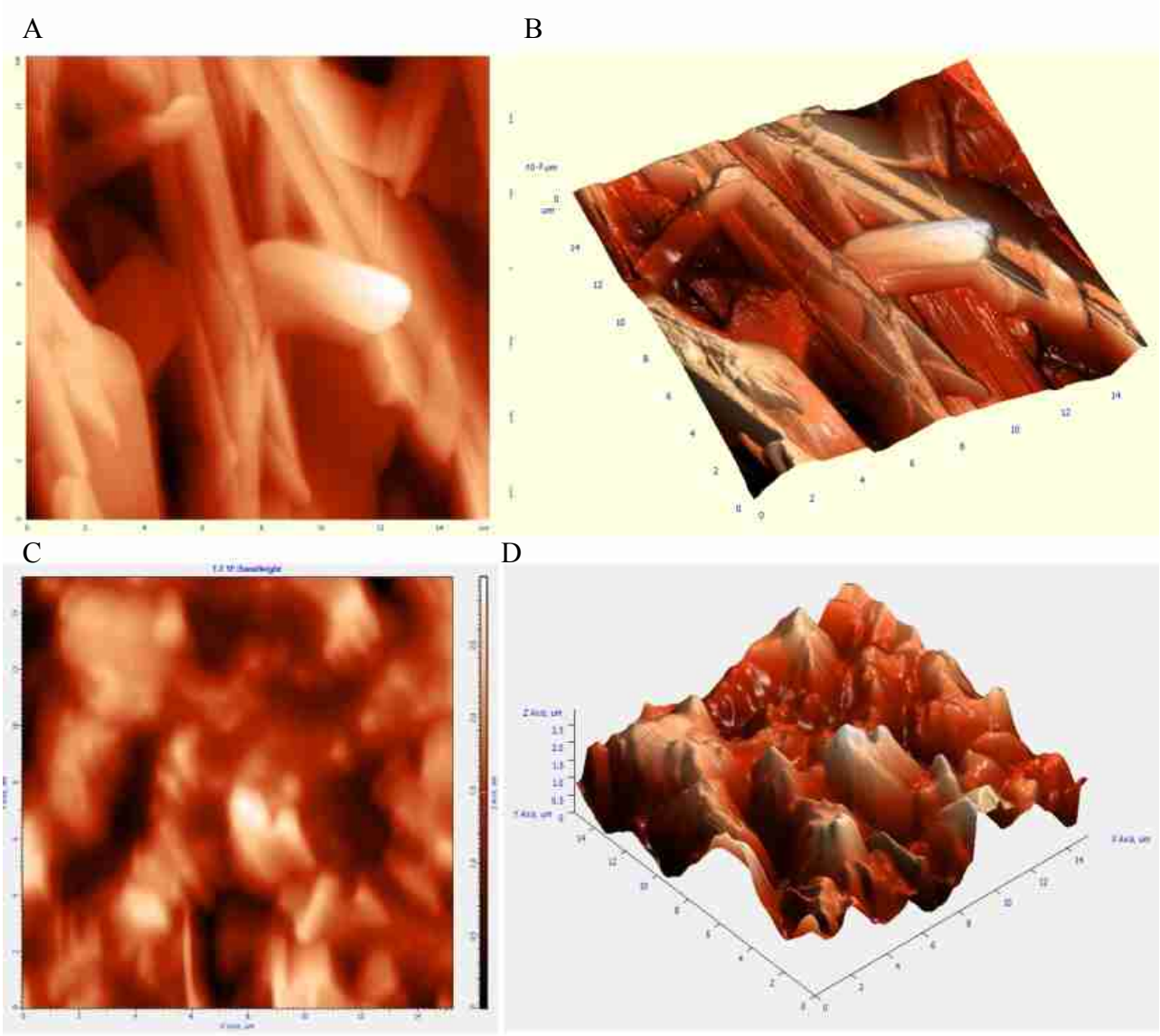

Figure 1.2D (A,C) and 3D (B,D) AFM micrography of the bloomed surface of dark chocolate samples submitted to temperature cycling treatments.

solid chocolate bar (Beckett, 2009). After mixing, the liquid chocolate paste is refined to further reduce particle sizes, ensuring flowability and adequate sensory properties. The chocolate is then conched (heating treatment under constant agitation in a thin layer of chocolate paste) to remove undesirable volatile compounds reducing acidity, further developing flavor as well as viscosity and texture. Chocolate undergoes tempering to promote adequate fat crystallization in its most stable form (Shafi *et al.*, 2018), so that the product presents the desirable brightness, hardness (snap) and shelf-life (avoiding fat bloom, for instance). Tempered chocolate is ready for molding into chocolate bars and packaging. Then, chocolate consists of a continuous fat phase where fine solid sugar, cocoa (and milk, if present) particles are suspended forming a semisolid product (Nightingale *et al.*, 2011).

Tempering is known to be a critical step for manufacturing of final products with well-defined organoleptic properties such as dark color, brightness, snaps, shelf-life properties and appreciated melting properties (in mouth but not in hands). This procedure is mainly based on time-temperature cycles allowing controlled cocoa butter phase transformations (Loisel *et al.*, 1998) from liquid phase to metastable crystals I (orthorhombic polymorph with melting temperature 16 to 18 °C) and II (hexagonal polymorph with melting temperature 22 to 24 °C) which transform slowly into crystals III (orthorhombic polymorph with melting temperature 24 to 26 °C) and IV (orthorhombic polymorph with melting temperature 26 to 28 °C) and then into the form V, a more stable polymorph (triclinic polymorph with melting temperature 32-34 °C) in well-tempering optimal conditions.

Under unfavorable storage conditions, such as temperature cycling, fat blooming can be developed at chocolate surface (Rousseau and Sonwai, 2008). Then, temperature fluctuations lead to rearrangement of triglycerides that make up the bulk of the chocolate matrix, forming a whitish coating on the surface of chocolate. Nevertheless, this is not only a visual default but also a texture change of the chocolate rendering it more brittle. Blooming is the main cause of quality loss in the chocolate industry (Nightingale *et al.*, 2011).

We used complementary experimental approaches for monitoring changes in calorimetric behavior, optical properties and surface morphology of a commercially available dark chocolate (85 % cocoa). A chocolate sample was exposed to tempering-cycles as following: 55 °C/30 min. (lipid crystal melting), 4 °C/1 night (refrigerator storage temperature), 23 °C/8 hours (room temperature), 4 °C/1 night, 23 °C/2hours, 4 °C/1week.

Here we reported only morphological results with 2D (Figures A, C) and 3D (Figures B, D) micrographs of blooming surfaces obtained by atomic force microscopy. We used an AFM set up (NT-MDT, Moscow, Russia) running in semi contact mode, scan rate at 0,7 Hz, cantilevers NSG01-NT-MDT, typical resonant frequency of 150 kHz and typical force constant of 5.1 N/m. This allowed to observe qualitative and quantitative information on fat blooming as related to nano-scale topographic evolution and bloom development under temperature-cycling.

Qualitatively, orthorhombic (Figure A, B) and triclinic (Figure C, D) crystal morphologies were observed. These crystals were produced as consequence of polymorphism transitions provoked by temperature changes. The morphological difference can be associated to the different emulsifier (lecithin) concentrations into the chocolate sample used.

And, quantitatively, the surface average roughness of bloomed surface can be calculated as the sum of the difference between the highest peaks and the average of the total peaks, which are around 190 nm, meaning that the formed structure was in nanoscale. Similar crystal morphologies were observed by Nightingale *et al.* (2011).

These observations confirm that the temperature cycle as well as the storage temperature interferes with the growth of fat bloom.

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