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# Effects of high pressure on anhydrous milk fat crystallization in emulsion

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Anhydrous milk fat crystallization was compared either in bulk phase or in oil in water emulsion by studying melting curves obtained by calorimetry measurements. Emulsions were submitted to a high pressure (HP) treatment (200 MPa/10 min). An aging period of 72 h was required to obtain the highest amount of crystallized fat at 4 °C. Emulsification promoted the crystallization of low melting point (LMP) triglycerides. The effect of HP on anhydrous milk fat crystallization depended on the aging time. For short aging periods (30 min and 1 h), HP treatment favored the crystallization of LMP triglycerides, thus amplifying the effect of emulsification compared with the crystallization in bulk phase. However, for a long aging time, *i.e.* 48 h, the amounts of crystallized matter in emulsion with or without HP treatment were comparable. This result was interpreted in terms of a different polymorphic form crystallizing under HP.

**Keywords:** oil in water emulsion; anhydrous milk fat crystallization; high pressure; melting enthalpy

## 1. Introduction

In various food products, the fat phase is found in a crystallized form at the temperature of use or of storage. The fat physical characteristics influence the rheological properties and the stability of the products [1–3]. In food, the fat phase is frequently found in a dispersed form such as oil in water (O/W) emulsions (ice cream, whipped topping). The mechanism of fat crystallization is different in the bulk phase and in emulsified systems so that the fat behavior in emulsion cannot simply be deduced from that in the bulk phase [4]. Crystallization in emulsion depends on various parameters such as the fat globule size, the presence of surfactants (proteins and low molecular weight emulsifiers) and the technological processes applied. It has been shown that high pressure (HP) treatment preserves the quality of treated food compared with conventional heat treatment [5]. Moreover, HP treatment of emulsions is less destabilizing than heat treatment [6]. Thus, the

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aim of this study was to evaluate the impact of HP treatment on the crystallization of anhydrous milk fat in the form of an O/W emulsion and to compare the obtained results with that obtained in the bulk phase.

## **2. Materials and methods**

### **2.1. Emulsion preparation**

Primary O/W emulsions were composed of 70 wt.% milk fat and 12 wt.% sodium caseinate adjusted to 100% with water. The sodium caseinate solution and the fat were preheated separately at 60 °C, mixed and immediately homogenized at 60 °C using a Couette cell (Ademtech SA, France) with a gap of 200 μm and an applied shear rate of 7420 s<sup>-1</sup>. The obtained emulsion was diluted with water containing sodium caseinate and sodium azide (0.08 wt.%) to set the fat concentration to 22.5 wt.% and the sodium caseinate concentration to 5 wt.% in aqueous. This emulsion was conserved at 4 °C for several weeks. The size distribution of the fat droplet was characterized by photo microscopy (Olympus BX51) and static light scattering using a Coulter LS 230 apparatus.

### **2.2. High pressure treatment**

The emulsion at 60 °C (to limited the crystallization) was placed in a plastic pouch, and hydrostatically pressurized in a 5 L sample compartment (containing a water-glycerol mixture at 4 °C) by means of a hyperbar experimental apparatus Framatome (France). The HP treatment (200 MPa/4 °C/10 min) was carried out on vacuum packaged emulsions (2 mL). The temperature of the sample decreased at 9 °C at the beginning of the pressurization stage then at 4 °C at the treatment end. After HP treatment, the treated samples were placed at 4 °C for crystallization time.

### **2.3. Differential scanning calorimeter**

The thermal behavior of milk fat in the bulk phase and in emulsion with and without the HP treatment was determined using a micro differential scanning calorimeter (DSC) VII instrument (SETARAM). Samples (≈100 mg) aged at 4 °C for 30 min, 1 h, 48 h or 72 h were sealed in aluminum pans. The samples were heated from 4 to 60 °C at a rate of 0.5 °C/min. Heat flow was recorded as a function of increasing temperature. For each sample, the experiment was conducted at least in triplicate. For enthalpy calculation, the base-line must still stable. The percentage of the crystallized matter in the emulsion was calculated according to the proportion of fat in the emulsion.

## **3. Results and discussion**

The emulsion based on anhydride milk fat and stabilized by sodium caseinate was used as dairy product model. The fat globule size analysis revealed the existence of a single population with a mean diameter of 2 μm. Because the droplet size may influence the fat thermal behavior [7], we checked that the fat particle size in the emulsion remained unchanged in the conditions of the thermal analysis and after HP treatment.

The DSC melting curve of anhydrous milk fat showed a complex pattern due to the extreme wide diversity of fatty acids and triacylglycerols (TG) that constitute this type of fat. The anhydrous

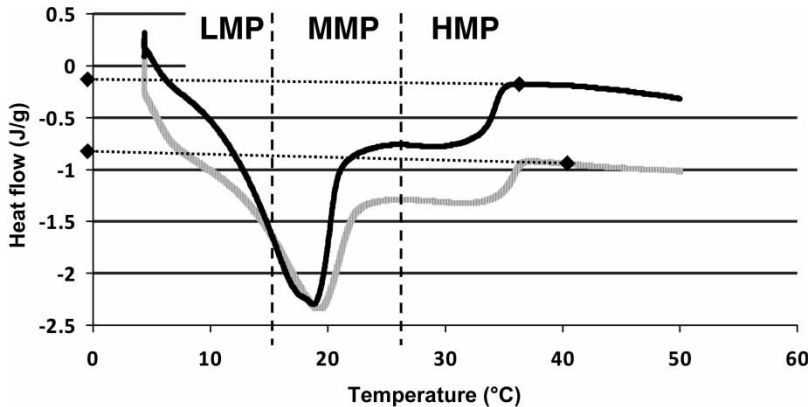


Figure 1. Endothermic peaks obtained by DSC during melting of anhydrous milk fat in the bulk phase (black line) and in O/W emulsion (grey line) after 72 h aging period at 4 °C. The samples were untreated by HP.

Table 1. Effect of the aging time at 4 °C and the HP treatment on enthalpies of DSC melting curves for the emulsified milk fat.

|                     | Enthalpy (J/g of fat) |              |              |
|---------------------|-----------------------|--------------|--------------|
|                     | 30 min                | 1 h          | 48 h         |
| Untreated emulsion  | 50.06 ± 2.29          | 60.61 ± 0.86 | 91.47 ± 1.58 |
| HP-treated emulsion | 120.42 ± 3.99         | 93.73 ± 1.93 | 89.71 ± 2.11 |

milk fat usually exhibits a melting range ranging from about  $-40^\circ\text{C}$  to  $40^\circ\text{C}$ . In our case, because the experiments started at  $4^\circ\text{C}$ , only part of the TG was in a crystallized state. As already reported in the literature, the milk fat melting curves allowed to distinguish three main factors: *i.e.* low melting point (LMP) TG ( $< 15^\circ\text{C}$ ), medium melting point (MMP) TG ( $< 26^\circ\text{C}$ ) and high melting point (HMP) TG (between  $26^\circ\text{C}$  and  $40^\circ\text{C}$ ) (Figure 1). A similar melting curve pattern was obtained for emulsified milk fat (Figure 1). The melting enthalpy of bulk fat was lower than that of the emulsified one ( $61.33 \pm 0.54 \text{ J/g}$  of fat vs  $92.80 \pm 4.12 \text{ J/g}$  of fat, respectively). This showed that emulsification promoted fat crystallization and specifically that of the LMP TG as suggested by the appearance of a shoulder in the melting curve below  $15^\circ\text{C}$ . Moreover, the area under the peak of LMP and MMP in the case of emulsified milk fat was about twice that of the non-emulsified fat ( $66.16 \pm 1.93 \text{ J/g}$  of fat vs  $34.49 \pm 0.86 \text{ J/g}$  of fat, respectively). The aging period at  $4^\circ\text{C}$  influenced the amount of crystallized matter. Increasing the aging time from 30 min to 48 h induced an increase of the melting enthalpy values by more than 80% (Table 1).

For a short aging period (30 min and 1 h) at  $4^\circ\text{C}$ , HP treatment seemed to favor crystallization as revealed by higher melting enthalpy values compared with untreated emulsions. However, upon 48 h of aging, with or without the HP treatment, melting enthalpy values of the emulsified fat were similar. This suggested that the increased amount of fat that first crystallized under HP was in an unstable physical state (presumably  $\alpha$  polymorphic form) that undergoes an  $\alpha$  to  $\beta'$  polymorphic transition during the aging period.

#### 4. Conclusion

The thermal behavior of milk fat studied by differential scanning calorimetry showed significant variations as a function of its physical state (bulk phase or emulsion). Moreover, HP treatment

influenced the amount of crystallized matter and presumably the polymorphic form of some TG. Further experiments are required to better understand the role of parameters such as pressure, temperature and time treatments on milk fat crystallization.

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