CONTINUOUS HEATING OF FOULING-SENSITIVE MILK PRODUCTS – MICROWAVE TECHNOLOGY AS NEW APPROACH?

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Abstract

During processing of milk concentrates the microbiological load especially of thermophilic spore formers and their endospores may increase. Thus, not only the concentrate but also the thereof produced powder can contain high amounts of thermophilic spores that survive transport and storage and finally germinate upon reconstitution. High temperature treatments of milk concentrate prior to the evaporation process reduce the count of thermophilic spores (> 100 °C). However, such a treatment in the existing indirect heating plants is limited due to extended deposit formation, so called fouling. Only intensive cleaning procedures with heavy cleaning agents remove fouling from the plant. It is hypothesized that through the lack of hot walls continuous microwave heating of milk concentrates induces less fouling, retains the product properties, and diminishes the microbiological load of thermophilic spores.

Skim milk concentrates (31.1 g/100 g dry matter, obtained via reverse osmosis) were heated in a continuously operated microwave heating plant in pilot scale (f = 2450 MHz, volume flow = 150 L/h). Temperatures ranging from 85 to 115 °C were applied with holding time t = 5 s. The concentrates were analyzed by means of $L^*a^*b^*$ colour space and particle size by static light scattering.

Direct heating by microwave technology of skim milk concentrate within the designed process window was feasible, while product properties were maintained. Except yellowing of the samples, colour changes (browning) were not found. Particle formation was significantly reduced and low in comparison to indirect heating. The experimental results allow to set a process window for microwave heating of milk concentrates with minimal product changes. Thus, microwave technology presents a new method to heat fouling-sensitive milk products.

Introduction

Various food processing applications like drying, heating, thawing, blanching, baking, and sterilization can be realized by direct heating through microwave technology [1–3]. Microwaves induce a temporary reorientation of polarized molecules in a dielectric material that is exposed to an electromagnetic field. Through internal friction and the vibration of neighboring molecules heat is induced in the material [1, 4]. The frequencies of microwaves range from 300 MHz to 300 GHz, whereby frequencies of 915 MHz and 2.45 GHz are typically used for microwave ovens and industrial purposes worldwide [1, 3]. Microwave heating retains flavour and nutritional values of food products as it lacks heat transfer surfaces and involves short heat transfer with high heating rates, thereby allowing gentle heating (in comparison to indirect heating methods) [4, 5]. It has recently been demonstrated that continuous microwave heating of milk products reduces deposit formation by 90 % in comparison to indirect heating via tubular heat exchangers [6].

Concentrated milk products like skim milk concentrate are prone to aggregate and deposit formation upon heating, as they contain high amounts of dry matter, including minerals and (whey) proteins [7, 8]. The latter are heat-sensitive and denature with subsequent aggregate formation if they are evolved to high temperatures ($\geq$ 80 °C). Together with minerals, a deposit is formed at hot heat transfer surfaces in indirect heat exchangers, so called fouling [9]. However, the heating step is crucial as heating of skim milk concentrate before powder manufacture represents one approach to reduce high amounts of thermophilic endospores in milk powders [10]. This issue is challenging for milk powder manufacturers as spores were shown to persist in processing plants, were thus found in milk powders, what finally decreased their acceptance [11, 12]. Moreover, heat preservation of milk concentrates favours their direct distribution and skips energy-consuming drying processes [13]. Direct heating methods like steam injection have been successfully proven as heating methods for skim milk concentrate in pilot scale [7]. However, this method dilutes the concentrate by steam addition and the energy recovery is poor. Moreover, continuous microwave heating has been shown to be suitable for heating reconstituted skim milk concentrates up to 125 °C [6, 10]. This study aims to demonstrate the feasibility of continuous microwave heating for skim milk concentrate in terms of colour changes, deposit, and particle formation.

1. Material and Methods

1.1. Production of skim milk concentrate

Skim milk concentrate was produced by reverse osmosis (MMS Membrane Systems, Urdorf, Switzerland equipped with a spiral wound module 3839 KMS HRX 14.2 m^2 Koch Industries Inc. Lenntech B.V. Delfgauw, Netherlands) of pasteurized skim milk at 50 °C and 150 L/h until a trans membrane pressure (TMP) of TPM $\approx$ 4 MPa was reached. The following values represent the final product properties: dry matter 31.10 ± 0.26 g/100 g, pH 6.30 ± 0.09, and electrical conductivity 7.72 ± 0.08 mS/cm.

1.2. Microwave heating of skim milk concentrate

A continuously operated pilot scale microwave heating plant (µWaveFlow 0620hp, Pueschner GmbH & Co KG, Schwanewede, Germany) has been set up as shown by Graf *et al.* [6]. The power outputs of the single-mode applicator were 0.6 – 6 kW at $f = 2450$ MHz, while a quartz glass tube ($d_\mathrm{i} = 3 \cdot 10^{-3}$ m, $l = 0.5$ m) resembled the microwave heating section. A PT100 was used to measure the product outlet temperature directly after the heating section, representing the temperature the heating experiments were referred to. In order to pre-heat and cool the product, the microwave heating plant was connected to a pilot heating plant (tubular heat exchanger, 03T210, MSR 03210, Asepto GmbH, Dinkelscherben, Germany).

All heating experiments were performed according to a previously presented procedure [6], with a volume flow of $\dot{V}$ = 150 L/h, an over pressure of $p \approx 0.3$ MPa, a pre-heating temperature of 80 °C, a single-staged homogenization between pre-heater one and two at 15 MPa, a heat holding section of length $l = 2.2$ m (inner diameter $3 \cdot 10^{-3}$ m) after the heating, realizing 5 s heat holding time, and a cooling temperature of 15 °C. Within each experiment, the temperatures 85, 90, 95, 100, 105, 110, and 115 °C were set and held for 5 min, respectively, before samples were taken for further analyses. All heating experiments were performed in duplicate.

1.3. Analyses

1.3.1. L*a*b* colour space

The colour of heated and unheated skim milk concentrate was determined based on the CIE $L^*a^*b^*$ colour space with a Chroma meter CR-400 (Konica Minolta, Chiyoda, Japan), where L^* represents the lightness (0 = black, 100 = white) and b^* the shift from blue (-) to yellow (+). The instrument was calibrated as described by the manufacturer with a white calibration plate each day prior to the measurements. 10 mL sample was filled into a petri dish (ø 30 mm) that was placed on a white background to ensure equal light conditions.

For analysis, the yellow index (*YI*), representing the browning of a product, was calculated according to Equation 1 [14].

$$YI = 142.86 \cdot b^*/L^* \tag{1}$$

1.3.2. Particle size distribution

The particle size of skim milk concentrate was determined by static light scattering with a particle size analyser (LS 13 320, Beckman Coulter Corp., Brea, USA). The refractive index of the surrounding medium (distilled water) was set to 1.33 while the refractive index of the sample was set to 1.57, representing casein micelles. For data evaluation, the $d_{90,3}$ was chosen, representing the value to which 90 % of the particles are equal to or smaller, based on a volumetric particle size distribution

1.3.3. Statistical analyses

The arithmetic mean and standard error have been calculated for all analyses. If values are given, they have been rounded to two significant digits of the standard error. To evaluate significant differences, a two-sided t-test in Sigma Plot 12.5 (Systat Software Inc., San Jose, California, USA) with p = 0.05 was performed. All pilot plant trials were performed in duplicate, whereas all analyses were performed in triplicate.

2. Results and Discussion

In a first approach, skim milk concentrate was heated in a) the pre-heater to 80 °C, b) in an indirect heater (tubular heat exchanger, pilot heating plant) at 110 °C, and c) in the microwave heating plant at 110 °C in order to test the feasibility and evaluate the effect of the pre-heater on the product properties. The resulting particle sizes ($d_{90,3}$) are shown in **Table 1**. While the pre-heater ($d_{90,3} = 0.21 \pm 0.01$ µm) had no impact on the particle size, heating through microwave technology ($d_{90,3} = 0.40 \pm 0.04$ µm) caused a significant increase. However, the particle size is still acceptable and low in comparison to indirect heating that caused a 60-fold increase in particle size ($d_{90,3} = 14.4 \pm 2.0$ µm). Moreover, the pressure in the indirect heating plant increased immediately, indicating that heating of native skim milk concentrate in a tubular heat exchanger to $\vartheta \geq 110$ °C causes deposit formation and is thus not feasible. All continuing experiments were performed using solely the microwave heating plant connected to the tubular pre-heater und cooler.

Table 1. Particle sizes ($d_{90,3}$) of unheated and heated skim milk concentrate. Different letters within column indicate significant differences, tested by a two-sided t-test with $p = 0.05$.

Heat treatment	Temperature in °C	Particle size $d_{90,3}$ in µm
unheated	-	0.21 ± 0.01^{a}
Pre-heater	80	0.21 ± 0.01^{a}
Tubular heat exchanger	110	14.4 ± 2.0^{b}
Microwave	110	0.40 ± 0.04^{c}

2.1. Browning of skim milk concentrate upon heating

Within the $L^*a^*b^*$ colour space, the L^*-value is a measure for the lightness of a sample. Characteristic ingredients of milk products like the milk protein casein, that is present in micellar structures, fat globule size and distribution, as well as technological treatments like heating and homogenization generally influence the lightness and thus the L^*-value [15]. The b^*-value is a colour indicator where negative values represent blue and positive values yellow colour. In milk, coloured pigments like riboflavin, β-carotene, and lutein influence the b^*-value, depending on their concentration [15]. In general, an increase in b^*-value implies a more yellowish appearance of the samples.

Fig. 1a shows the L^*- and b^*-values of unheated and microwave heated skim milk concentrate ($\vartheta = 85 – 115$ °C). With increasing temperature, the L^*- and b^*-values increase, as well as the *YI*, shown in **Fig. 1b**. All values show highest increases at $\vartheta = 115$ °C, of about 8 (L^*-value), 5 (b^*-value), and 8 (*YI*). This implies that after microwave heating skim milk concentrate appeared lighter and showed a shift towards yellow colour, more pronounced with increasing heating temperature. The results are in accordance with our previous studies where reconstituted skim milk concentrate was heated by means of microwave technology. There, the L^*-value was 88.97 ± 0.46 (increased 0.6 upon heating) and b^*-value -0.24 ± 0.12 (increased 1.8 upon heating) at 115 °C and a dry matter of 31.5 g/100 g [6]. However, former studies that heated skim milk concentrate using indirect heat transfer to temperatures of 110 °C (5 and 10 min holding time) or even at 138-140 °C (3 s holding time), found visible browning accompanied with decreasing L^*-values, implying a darkening of the samples [16, 17]. Upon heating milk at elevated temperatures (> 100 °C), brown Maillard reaction products can be formed by sugars reacting with free amino acids. This reaction is enhanced in skim milk concentrate, as the dry matter and thus possible reaction partners are increased, as well as through enhanced heating temperatures and times [18].

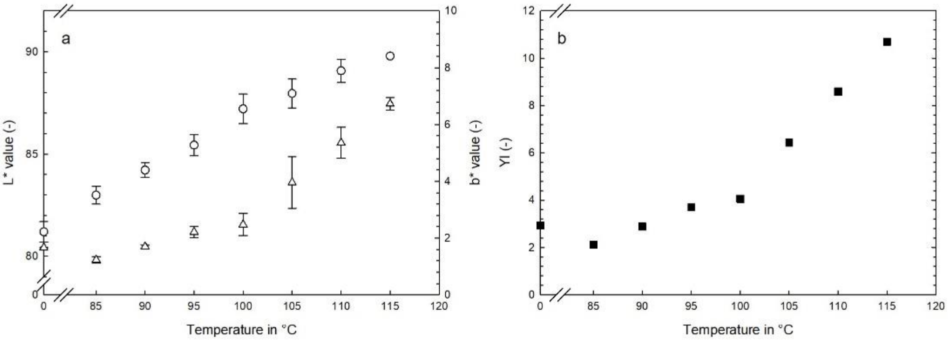

Fig. 1. Colour of microwave heated skim milk concentrate. a) L^* (○) and b^* (Δ) values and b) Yellow index (*YI*).

Although the observed increases in *YI* and *b**-values indicate a yellowing of skim milk concentrate after microwave heating, the increase in *L**-value proves that this effect has low significance, as only decreasing *L**values are attributed to Maillard reaction products and thus visible and intensive browning [18]. Moreover, it is well known that heat holding times below 5 min (at 110 °C) do not induce brown pigments through Maillard reaction [16]. Overall, the colour change in microwave heated skim milk concentrate was low and confirmed previous findings for heating reconstituted skim milk concentrate [6].

2.2. Particle formation in skim milk concentrate

Fig. 2 displays the particle size as $d_{90,3}$ of skim milk concentrate after heating with microwave technology. The $d_{90,3}$ represents the value to which 90 % of the particles are equal to or smaller. Upon heating, the $d_{90,3}$ of skim milk concentrate increased from 0.21 µm for unheated, 85, and 90 °C to 0.40-0.48 µm for heating temperatures ranging between 95-115 °C. One of the two performed experiments at 115 °C resulted in $d_{90,3} = 36.9$ µm.

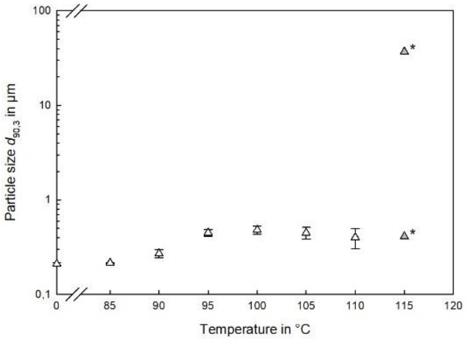

Fig. 2. Particle size $d_{90,3}$ of microwave heated skim milk concentrate. Grey triangles (marked with an asterisk) are depicted as single values.

Although the particle sizes increased significantly at heating temperatures ≥ 95 °C, they were comparable to skim milk concentrate heated by direct steam injection and still low compared to particle sizes of reconstituted skim milk concentrate heated by microwave technology ($d_{90,3}$ = 8-32 µm) in previous experiments [6, 13]. Dumpler and Kulozik [13] investigated the heat coagulation temperature of skim milk concentrate (obtained via reverse osmosis) in dependency of the pH. At a pH of 6.30 (pH of the skim milk concentrate used in this study) and a dry matter of 30 g/100 g, the heat coagulation temperature was 110 °C [13]. This possibly explains the observed difference in particle size at a heating temperature of 115 °C. Due to increased dry matter (31.1 g/100 g) and slight variations in pH, the heat coagulation temperature was exceeded in one trial, whereas in the other trial 115 °C were still below the heat coagulation temperature. For further trials, the pH should be adjusted to pH 6.4 or higher, as this increases the heat coagulation temperature to 120-130 °C, depending on the dry matter [13].

In general, microwave technology is a suited method to heat skim milk concentrate at elevated temperatures. It should be considered that not only the processing method but also the product properties (dry matter and pH) influence the particle formation upon heating.

3. Conclusion

This study investigated colour changes and particle formation upon microwave heating of skim milk concentrate (obtained via reverse osmosis). Regarding colour changes, microwave technology is suited to heat skim milk concentrate without visible browning. Moreover, deposit formation and enhanced particle formation were not detected. However, besides the processing method, the product properties play an important role concerning particle formation, as they e.g., influence the heat coagulation temperature of skim milk concentrate. Further experiments will focus on pH adjustment of skim milk concentrate prior to heating. Moreover, overheating in the microwave section should be prevented. Therefore, a temperature profile of the microwave heating section will be recorded with a height-adjustable fiber optic temperature sensor directly in the product, in order to gain deeper knowledge about the temperature profile upon heating.

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