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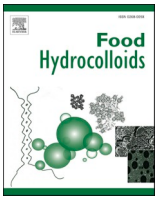

Spray-dried microfibrillated cellulose particles as texture modifier in liquid foods and their effect on rheological, tribological and sensory properties

Annelies E. Blok [a,*], Dieuwerke P. Bolhuis [a], Krassimir P. Velikov [b,c,d], Markus Stieger [a]

[a] Food Quality and Design, Wageningen University, P.O. Box 17, 6700 AA, Wageningen, the Netherlands
[b] Unilever Innovation Centre Wageningen, Bronland 14, 6708 WH, Wageningen, the Netherlands
[c] Institute of Physics, University of Amsterdam, Science Park 904, 1098 XH, Amsterdam, the Netherlands
[d] Soft Condensed Matter, Debye Institute of Nanomaterials Science, Utrecht University, Princetonplein 5, 3584 CC, Utrecht, the Netherlands

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ABSTRACT

Microfibrillated cellulose (MFC) has potentisal to be used as clean label texture modifier in foods due to its structural and mechanical properties. These properties deteriorate upon drying of MFC dispersions due to aggregation of the microfibrils. In this study dried MFC particles were prepared by spray-drying MFC dispersions in a surplus of maltodextrin to prevent hornification. The aim of this study was to evaluate the effect of MFC particle concentration and MFC:maltodextrin ratio of dried MFC powders on rheological, tribological and sensory texture properties of liquid foods. Scanning Electron Microscopy demonstrated that after spray-drying, MFC powders with polydisperse particle size distribution were obtained (1–30 μm). Upon suspension of spray-dried MFC powder in water, maltodextrin dissolved in the aqueous continuous phase whereas spherical MFC networks retained their shape and co-existed in a mixture with individual fibrils. Spray-dried MFC powders were added to skimmed milk and tomato soup at different concentrations. With increasing concentration of dried MFC particles, shear viscosity, consistency index K, storage and loss modulus of skimmed milks and tomato soups increased whereas flow index n decreased. Addition of spray-dried MFC particles to milks and soups significantly ($p < 0.05$) increased sensory thickness and creaminess. Milks displayed similar tribological properties irrespective of MFC particle concentration, which was presumably caused by exclusion of the MFC network from the tribological gap. Rheological properties, thickness and creaminess increased more effectively upon addition of low MFC:maltodextrin particles compared to particles with high MFC:maltodextrin ratio. We conclude that spray-dried microfibrillated cellulose particles can be used as thickener or fat replacer in liquid foods.

1. Introduction

Cellulose is the most abundant renewable biopolymer on earth, consisting of long chains of β-(1–4)-linked D-glucose assembled into microfibrils (Wüstenberg, 2014). It can be obtained from agricultural crops and waste streams such as fruit and vegetable pulp or peels, which offers environmental benefits (e.g. Koul, Yakoob, & Shah, 2021; Usmani et al., 2021; Zain, Yusop, & Ahmad, 2014). The water insolubility of unrefined cellulose however hampers its application in foods and has led to the development of several cellulose nanomaterials, including rod-like cellulose nanocrystals (CNC) and microfibrillated cellulose (MFC). In contrast to CNC, which undergoes hydrolysis to remove amorphous cellulose, microfibrillated cellulose consists of both crystalline and amorphous cellulose (Lavoine, Desloges, Dufresne, & Bras, 2012). MFC is prepared by disintegration of cellulose fibres using mechanical treatment (usually high-pressure homogenisation, microfluidization or grinding) resulting in delamination of the cellulose fibres and the release of individual microfibrils. In polar liquids such as water, an entangled fibrous network is formed and a stable dispersion is obtained (Siró & Plackett, 2010). The mechanical properties of the fibrillated material are improved due to its enlarged surface area, which makes MFC dispersions suitable as texture modifiers in foods (e.g. Blok et al., 2021; Gómez et al., 2016; Ström, Öhgren, & Ankerfors, 2013).

MFC has thus far mostly been applied and studied in dispersed form. Transport and storage of MFC dispersions is inefficient and expensive, which hampers the development of foods with MFC as texture modifier. One of the main challenges of applying MFC in food products has been the aggregation of microfibrils upon drying (Déléris & Wallecan, 2017).

* Corresponding author.
E-mail address: annelies.blok@wur.nl (A.E. Blok).

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The enhanced surface area of MFC gives rise to the exposure of additional hydroxyl groups, which can form strong hydrogen bonds with hydroxyl groups of adjacent cellulose molecules. Upon drying of MFC dispersions, water removal causes the cellulose fibres to move closer to each other and aggregate, a phenomenon referred to as hornification. Hornification induces irreversible binding of the fibrils and increases the crystallinity of the material. The process essentially reverses the fibrillation procedure, resulting in inferior mechanical properties upon resuspension of dried MFC. Several studies investigated how to improve the drying of MFC. Different drying and preparation methods have been compared (e.g. Eyholzer et al., 2010; Jiang & Hsieh, 2014; Peng et al., 2013; Silva et al., 2021; Yan et al., 2016) and drying MFC in the presence of other components has been described (Butchosa & Zhou, 2014; Cantiani, Knipper, & Vaslin, 2002; Lowys, Desbrieres, & Rinaudo, 2001; Missoum, Bras, & Belgacem, 2012). A promising method to prevent hornification was proposed recently by Velásquez-Cock et al. (2018), who mixed MFC dispersions with various amounts of maltodextrin prior to drying. The described method includes removal of maltodextrin by vacuum filtration and hot water rinsing following redispersion of the dried material yielding stable MFC dispersions. However, the processing steps required for the removal of maltodextrin might limit the applicability of these processes for incorporation of MFC in foods.

In the present study, MFC was prepared in a surplus of maltodextrin and subsequently spray-dried to obtain dried MFC particles. MFC and maltodextrin were spray-dried in two different ratios. The two types of spray-dried MFC were applied in two liquid foods: skimmed milk and instant tomato soup. The effects of concentration of spray-dried MFC particles and MFC:maltodextrin ratio on rheological, tribological and sensory texture properties of both liquid foods were determined in order to assess whether spray-dried MFC particles can be used as texture modifier in liquid foods.

2. Materials & methods

2.1. Preparation of spray-dried MFC particles

Two types of spray-dried MFC powders were prepared differing in MFC:maltodextrin (MFC:MD) ratio: (i) 1:25 with 3.8 wt% MFC in the final powder (MFC:MD-Low) and (ii) 1:7.5 with 11.6 wt% MFC in the final powder (MFC:MD-High) (Table 1). The MFC:MD ratios used in this study were selected to represent a broad range of MFC:MD ratios (3-fold difference between MFC content in spray-dried powder). The MFC: maltodextrin ratio for the low-ratio powder (1:25) was selected based on preliminary experiments (data not shown) that demonstrated that this was the lowest MFC:MD ratio at which an effect on rheological, tribological and sensory properties could be expected. The high MFC:MD ratio (1:7.5) was chosen as higher MFC:MD ratios could not be spray-dried, since the viscosity of the suspension became too high. Citrus fibre powder (HERBACEL® AQ® Plus, Herbafood Ingredients, Werder, Germany) containing ~65 wt% cellulose (Fechner, Fenske, & Jahreis, 2013) was dispersed in demineralised water using a L5M-A Silverson laboratory mixer operated at 8000 rpm for 15 min (Silverson Machines Ltd., Chesham, United Kingdom). A second mixing step of 15 min was performed after addition of maltodextrin (Glucidex® 29, Roquette, Lestrem, France), followed by addition of tricalcium phosphate (anhydrous ($Ca_3(PO_4)_2$; Brenntag, Essen, Germany) and mixing for 2 min. The mixture was subsequently homogenised using a lab scale high-pressure microfluidizer (Microfluidizer M-110S, Microfluidics™, Newton, MA, USA) equipped with a 200 μm Z-cell at 900 bar, to obtain microfibrillated cellulose. This was followed by a second passage through the same microfluidizer equipped with an 87 μm Z-cell at 1200 bar. The resulting slurry was spray-dried (Mini Spray Dryer B-290, Büchi Labortechnik AG, Flawil, Switzerland) using a 1.5 mm two-fluid nozzle having an atomizing pressure of 2 bars. The inlet (controlled) and outlet (measured) air temperatures were 165 °C and 60–75 °C, respectively. The flow rate was set at 0.3–0.5 L/h (=15–25% pump). Different set flow rates were used because the actual flow rate depends on the viscosity of the sample. Lower flow rates were used when the measured outlet temperature became lower than 65 °C. A gas flow rate of 30 was used and the nozzle cleaner was set at 5. The composition and other details of the dispersions and the resulting spray-dried powders can be found in Table 1. The obtained spray-dried powders were stored in glass jars at room temperature in the dark for up to three months until further use.

Table 1
Composition of the two spray-dried MFC powders differing in MFC:maltodextrin (MFC:MD) ratio before and after spray-drying (in wt%).

	Before spray-drying		After spray-drying[a]	
	MFC:MD-Low (1:25)	MFC:MD-High (1:7.5)	MFC:MD-Low (1:25)	MFC:MD-High (1:7.5)
Citrus fibre	0.3	1	3.8	11.6
Maltodextrin	7.5	7.5	94.3	86.7
Calcium phosphate	0.15	0.15	1.9	1.7
Demineralised water	92.05	91.35	[a]	[a]

[a] Moisture content after spray-drying was not quantified and the composition after spray-drying is therefore expressed based on 100 g dry matter (weight per 100 g dry matter).

2.2. Sample preparation

Spray-dried MFC powders were dispersed at different concentrations in skimmed milk (0% fat milk, Albert Heijn, Zaandam, the Netherlands) and instant tomato soup (Knorr tomato drink bouillon (<0.5% fat), Unilever, London, United Kingdom). Table 2 presents the final composition of the liquid foods. The concentration of maltodextrin in all liquid samples was constant (7.5 wt%) to standardise potential effects of maltodextrin on flavour and mouthfeel. Differences in rheological, tribological and sensory properties between samples are therefore related to MFC. In order to compare the two types of spray-dried MFC powder (MFC:MD-Low vs MFC:MD-High), a concentration of MFC:MD-High was selected to match the final MFC concentration to that in M-L8 and S-L8 (Table 2). Commercial full-fat milk (3.6% fat; Albert Heijn, the Netherlands) was added as a reference to the skimmed milk sample set.

For the skimmed milks, spray-dried MFC powder and maltodextrin were gradually added to the skimmed milk under continuous stirring on a magnetic stirrer plate at room temperature. Samples were stirred at 300 rpm for 45 min, until all ingredients were fully dissolved. Instant tomato soups were prepared according to the instructions on the product. 175 ml of boiling water was added to 8.1 g instant soup powder and stirred for 5 min at room temperature. The soup was subsequently cooled using an ice bath and sieved to remove pieces of herbs and undissolved ingredients. Spray-dried MFC and maltodextrin were gradually added to the soups while stirring at room temperature (300 rpm) for 60 min until all ingredients were dissolved.

2.3. Scanning Electron Microscopy (SEM) and Cryo Scanning Electron Microscopy (cryoSEM)

Spray-dried MFC powders were characterised using Scanning Electron Microscopy (SEM). For this the powders were fixed on SEM stubs using carbon adhesive tabs (EMS Washington USA) and sputter coated with 12 nm Tungsten (Leica EM SCD 500, Vienna, Austria). SEM images were recorded using a FEI Magellan 400 at an acceleration voltage of 2.0 kV and 13 pA. Secondary electrons (SE) were detected using an Everhart-Thornley detector at a working distance of ~4.7 mm.

The behaviour of spray-dried MFC powder in aqueous matrices was characterised using cryoSEM on a dilute suspension of MFC:MD-Low in water. For the cryoSEM images a small droplet of the suspension was placed between aluminium (HPF) platelets (Wohlwend, Sennwald, Switzerland). The sample was then frozen by plunging in liquid ethane.

Table 2
Composition of the liquid samples used for rheological, tribological and sensory analyses. Skimmed milks (M) and instant tomato soups (S) were used as liquid matrices. Full-fat milk (M-FF) was used as a commercial reference product for set of milks. Two types of MFC powder were added at varying concentrations: MFC:MD-Low (L: 2, 4, 8 wt%) and MFC:MD-High (H: 2.6 wt%).

		MFC:maltodextrin ratio				Final composition of milks and soups	
	Sample	Low (1:25) (wt%)	High (1:7.5) (wt%)	Maltodextrin (MD) (wt%)	Matrix (soup / milk) (wt%)	MFC (wt%)	Maltodextrin (MD) (wt%)
Milk	M-0	-	-	7.5	92.5	-	7.5
	M-L2	2	-	5.6	92.4	0.075	7.5
	M-L4	4	-	3.7	92.3	0.15	7.5
	M-L8	8	-	-	92.0	0.3	7.5
	M-H2.6	-	2.6	5.3	92.1	0.3	7.5
	M-FF	-	-	-	100 (full-fat)	-	-
Soup	S-0	-	-	7.5	92.5	-	7.5
	S-L2	2	-	5.6	92.4	0.075	7.5
	S-L4	4	-	3.7	92.3	0.15	7.5
	S-L8	8	-	-	92.0	0.3	7.5
	S-H2.6	-	2.6	5.3	92.1	0.3	7.5

The frozen samples were transferred to a cryo-preparation system (MED 020/VCT 100, Leica, Vienna, Austria) onto a sample stage at −92 °C in high vacuum. In this cryo-preparation system the samples were freeze-fractured and kept for 15 min at −92 °C for ice sublimation and etching of the samples. After coating with 12 nm of tungsten, the samples were transferred under vacuum to the field emission scanning microscope (Magellan 400, FEI, Eindhoven, the Netherlands) on the sample stage at −120 °C. SEM images were recorded at an acceleration voltage of 2.0 kV and 13 pA.

2.4. Rheological properties

Rheological properties of the samples were determined using an MCR 302 rheometer (Anton Paar, Graz, Austria) equipped with a concentric cylinder (ø = 17 mm). A double gap measuring system (ø = 26.7 mm) was used for M-0 and M-FF due to the low viscosity of these samples. To determine rotational shear flow properties, shear viscosity was measured while increasing the shear rate from 1 to 1000 s^{-1} in 50 logarithmic steps. The Ostwald-de Waele power law model was fitted to the shear-thinning zones of the flow curves (viscosity at shear rates ranging from 1 s^{-1} to 100 s^{-1}) and used to determine consistency index K and flow index n: $\sigma = K \times \dot{\gamma}^n$, in which σ = shear stress (Pa), $\dot{\gamma}$ = shear rate (s^{-1}), K = consistency index ($Pa{\cdot}s^n$) and n = flow index. Mean square errors (MSE) from the averaged flow curves were determined to indicate the goodness of the fit. Strain sweeps (oscillatory shear flow) were performed by increasing the strain from 0.1 to 100% at a constant oscillation frequency of 1 Hz. The values of G′ and G″ at 1% shear strain were extracted from the strain-sweeps. All rheological measurements were performed in triplicate at 35 °C to mimic the temperature in the oral cavity. Each measurement was preceded by a 5 min waiting step to allow for structural relaxation of the sample after loading.

2.5. Tribology: Friction properties

Tribological (friction) properties were determined using an MCR 302 rheometer (Anton Paar, Austria) equipped with a tribological set-up (T-PTD 200). A ball-on-three-pins set-up was used with a glass ball and three polydimethylsiloxane (PDMS) pins. 600 μl of the sample was loaded into the sample cup. Measurements were performed in triplicate at 35 °C. Each measurement consisted of two runs in which the rotational speed was increased from 0.0001 to 2200 rpm (equivalent to 0.00004–1000 mm/s). Each run was preceded by a 5 min resting period to allow for structural relaxation of the sample. A normal force of 1 N was applied during runs and waiting steps. New pins were conditioned by performing one run (0.0001–2200 rpm) with 1 ml demineralised water, followed by a run with 1 ml of the respective sample without MFC particles (M-0 or S-0). Data from the second run was used for analysis, as the first run showed variation due to running-in effects and adjustments in the alignment of the pins. Maximum friction μ_{max} was determined as the maximum friction coefficient in the boundary regime. Since for soups the mixed regime was considered to start immediately after reaching the maximum in the boundary regime (μ_{max}), the sliding speed at which the mixed regime starts was extracted as the speed at which μ_{max} occurred. As μ_{max} did not represent the end of the boundary regime for milks, the start of the mixed regime for these samples was determined visually by the researcher as the sliding speed at which a precipitous negative deflection in the friction curve is observed. The slope in the mixed regime was determined as the slope between the start of the mixed regime until the friction measured at 1000 mm/s.

2.6. Sensory evaluation

Participants were recruited from Wageningen and surroundings. Individuals were excluded from participation in the study when they were allergic or intolerant to milk or when they had diabetes. Women were excluded from participation when they were pregnant or

Table 3
Sensory attribute definitions and examples of products with high and low intensity of the attributes.

Sensory attribute	Definition	Examples
Thickness	The amount of force needed to make the sample flow in the mouth (the easier the sample flows in the mouth, the thinner the sample)	Low: water High: Greek yoghurt
Creaminess	The degree to which the sample provides a silky, smooth, velvety, rich, full mouthfeel (which is often associated with the presence of fat)	Low: water High: (un)whipped cream

breastfeeding. Participants (n = 72; 18 male, 53 female, 1 other) were between 18 and 37 years old (mean 24 ± 3 y), had a BMI between 17.9 and 28.7 kg/m^2 (mean 21.9 ± 2.6 kg/m^2) and were non-smokers. Participants from 17 different countries were recruited, and most were students of Wageningen University. Participants received financial reimbursement upon completion of the study. The study did not meet the requirements to be reviewed by the Medical Research Ethical Committee of The Netherlands according to the "Medical Research Involving Human Subjects Act" of The Netherlands. The study was conducted in agreement with the ethics regulations laid out in the Declaration of Helsinki (2013). All participants gave written informed consent.

The consumer panel evaluated milks and soups in two sessions of 45 min on different days. The order in which participants attended the two test sessions was randomised. Samples were evaluated using the rank-rating method, in which all samples were presented simultaneously and evaluated for one attribute at a time. Samples were evaluated for thickness and creaminess, see Table 3 for attribute definitions and examples of products low and high in intensity of the respective attribute. The order in which participants evaluated the two attributes was randomised, new samples (with different 3-digit codes) were provided when participants moved to evaluation of the second sensory attribute. Samples within a sample set (5 samples for soup, 6 samples for milk) were presented simultaneously in random order, in transparent 30 ml cups labelled with random 3-digit codes. Participants were instructed to focus on the mouthfeel of the samples, disregarding any potential differences in appearance or flavour. Participants evaluated the samples by tasting the samples and placing the 3-digit codes corresponding to the samples on a 100 mm unstructured line scale representing the intensity of the attribute. In this way all samples were placed on the same line scale, which was anchored *'weak'* on the left and '*strong'* on the right end. A comment box was provided in case participants had any additional comments about the sample set. Participants were allowed to expectorate the sample after evaluation, and were asked to rinse their mouth with water between different samples. Between evaluation of the first and second attribute, participants cleansed their palate with crackers and water. Each test session was preceded by a familiarisation task to become acquainted with the test methodology and the attributes to be evaluated. Data was collected in English in EyeQuestion® (Logic8, the Netherlands).

2.7. Data analysis

Results from instrumental analyses were reported as mean values with standard deviation. One-way ANOVA and Tukey post-hoc analyses were performed to determine significant differences in rheological and tribological properties between samples prepared from the same matrix (milk or soup). Results from sensory evaluation of thickness and creaminess intensity were reported as mean values with standard error. Thickness and creaminess intensities (scale 0–100) were analysed using Repeated Measures ANOVA, with sample as fixed factor and participant as random factor. Bonferroni post-hoc analyses were performed to determine statistically significant differences between the samples. Pearson correlations between sensory thickness and creaminess were calculated for milks and soups separately. Pearson correlations between sensory and instrumental data were computed using the means of the pooled datasets (n = 11). Data analysis was performed using RStudio (version 4.0.2) using the packages lmerTest (Kuznetsova, Brockhoff, & Christensen, 2017), emmeans (Lenth, 2021) and Hmisc (Harrell & Dupont, 2018). A significance level of α = 0.05 was used.

3. Results & discussion

3.1. Morphology of spray-dried MFC powders differing in composition before and after resuspension

Scanning Electron Microscopy images of the two types of spray-dried MFC powders revealed that both powders have a polydisperse particle size distribution (Fig. 1a–b). Both powders contain smaller and larger particles, varying in size between ~1 and 30 μm. In the current study, a surplus of maltodextrin was added to MFC dispersions to improve the redispersibility of spray-dried MFC. The presence of polymers such as maltodextrin on the cellulose microfibrils prevents the formation of agglomerates during drying (hornification) (Lowys et al., 2001; Velásquez-Cock et al., 2018).

Fig. 1c and d shows the morphology of spray-dried MFC powder (MFC:MD-Low) that has been resuspended in water. The powder particles swell and adopt a more spherical shape upon suspension in water.

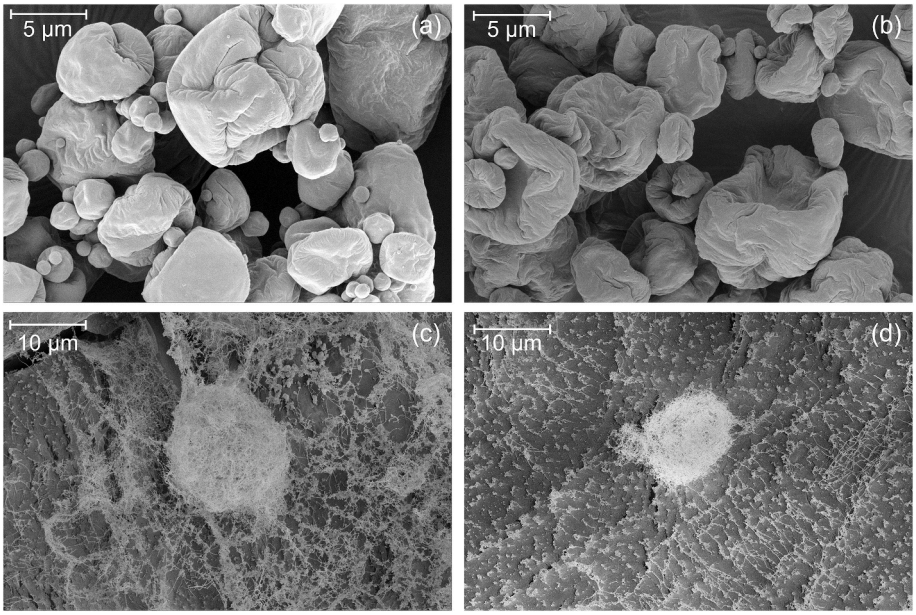

Fig. 1. Scanning Electron Microscopy (SEM) images of (a) MFC:MD-Low and (b) MFC:MD-High spray-dried MFC particles at 10000x magnification. Panel (c) and (d) show cryoSEM images of MFC:MD-Low after suspension in water at 5000x magnification (please note that no cryoSEM images of MFC:MD-High after suspension in water are shown).

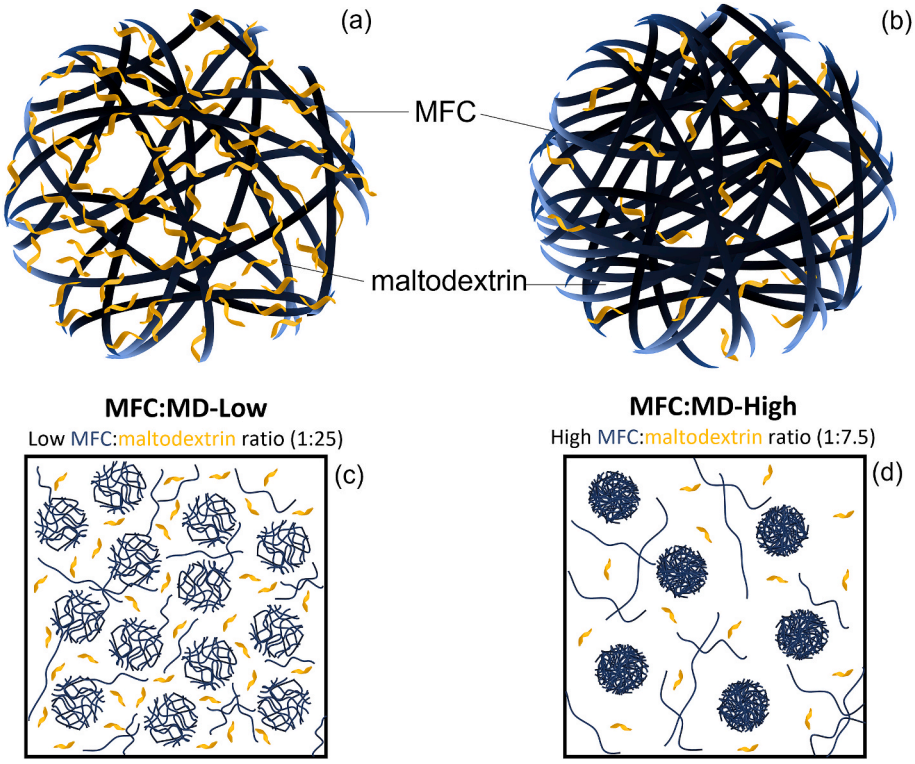

Fig. 2. Schematic overview of the proposed differences in morphology between spray-dried powders with (a,c) low MFC:maltodextrin ratio (1:25, MFC:MD-Low) and (b,d) high MFC:maltodextrin ratio (1:7.5, MFC:MD-High). The MFC network is displayed in dark blue, maltodextrin in yellow. Figure a and b represent spray-dried MFC powder before suspension in water, higher MFC:maltodextrin ratio results in particles with more entanglements between cellulose microfibrils. Figure c and d represent MFC particles after suspension in water, and spherical MFC networks are surrounded by individual microfibrils and aggregates thereof. It is hypothesised that at constant MFC content, fewer and denser particles might be formed at higher MFC:maltodextrin ratio. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Three-dimensional spherical networks of entangled MFC are observed with particle sizes of approximately 10 μm. These MFC particles stay intact after suspension in water and are surrounded by individual microfibrils and small networks thereof. Two possible explanations for the presence of individual microfibrils and aggregates thereof are: (i) not all MFC contributes to the formation of spherical particles, or (ii) some of the spherical MFC networks are not sufficiently strong to maintain their shape upon suspension and therefore collapse or disintegrate. It is expected that maltodextrin is present in the voids within the MFC network and that suspension of the powders results in dissolution of maltodextrin in the continuous aqueous phase (Fig. 2a–b). The spherical particles formed by the MFC network can potentially modify the texture of the liquid matrix in which the particles are suspended. We speculate that the lower maltodextrin content in MFC:MD-High enhances entanglement between the microfibrils during the spray-drying process. A larger number of contact points due to increased entanglement might strengthen the network, and these particles therefore might be denser and more rigid compared to MFC:MD-Low particles. We hypothesise that at constant MFC content within a food, fewer (*i.e.* lower volume fraction) but denser particles might be formed at high MFC:MD ratio compared to a lower ratio (Fig. 2c–d).

3.2. Rheological properties

3.2.1. Viscosity

All samples (soups and milks) displayed shear-thinning behaviour with the exception of skimmed milk and soup without MFC particles (M-0, S-0; Fig. 3), which displayed near-Newtonian behaviour. The Ostwald-de Waele model fitted the shear-thinning zones (viscosity at shear rate between 1 s^{-1} and 100 s^{-1}) of all samples well (see Table 4 for mean square errors). An increase in the concentration of MFC:MD-Low resulted in an increase in shear viscosity and consistency index *K*, and a lower flow index *n* (Fig. 3, Table 4). Although the final MFC (0.3 wt%) and maltodextrin content (7.5 wt%) of samples with 2.6 wt% MFC:MD-High were identical to those with 8% MFC:MD-Low (M-L8 and S-L8), flow curves of M-H2.6 and S–H2.6 overlapped with flow curves of M-L4 and S-L4. This suggests that MFC:MD-High powder is less effective at enhancing viscosity of liquid foods, and is therefore a less effective thickener than MFC:MD-Low. We speculate that this difference in rheological properties between high and low MFC:MD ratio powders might be caused by density differences of the respective MFC networks of these powders and consequently the number of spherical MFC particles that can be formed with the same amount of MFC (Fig. 2). Spray-

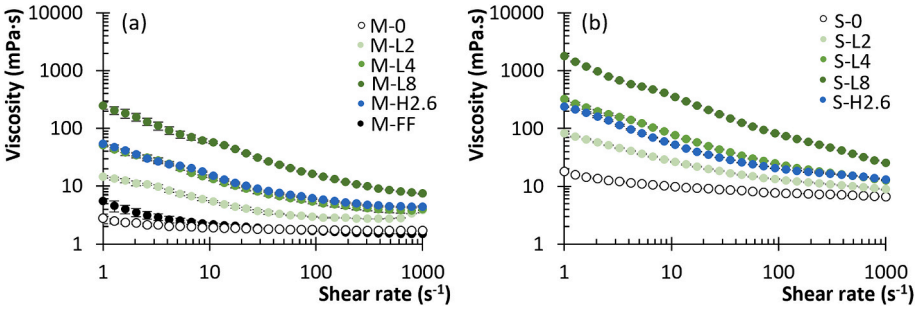

Fig. 3. Flow curves of (a) milks and (b) soups to which two types of spray-dried MFC were added at various concentrations. Sample codes and composition are summarised in Table 2. Means are presented with error bars representing standard deviations (triplicates).

Table 4
Rheological properties of milks and soups to which two types of spray-dried MFC were added at various concentrations (mean ± standard deviation). Flow index n and consistency index K were determined by fitting the Ostwald-de Waele power law model to the shear-thinning zone of the flow curves of the experimental data (viscosity at shear rates between 1 s^{-1} and 100 s^{-1}). The goodness of the fit of the Ostwald-de Waele power law model to the experimental data is represented by the Mean Square Error (MSE). Sample codes and composition are summarised in Table 2.

		Rotational shear flow			*Oscillatory shear flow*	
	Sample	Flow index *n*	Consistency index *K* (Pa·s^n)	Fit to power-law model: MSE (Pa^2)	Storage modulus G' (Pa) (at 1% strain and 1 Hz)	Loss modulus G'' (Pa) (at 1% strain and 1 Hz)
Milk	M-0	0.92 ± 0.02^{a}	0.002 ± 0.000^{b}	$8.81 \cdot 10^{-6}$	0.01 ± 0.01^{c}	0.04 ± 0.03^{b}
	M-L2	0.63 ± 0.04^{c}	0.014 ± 0.001^{b}	$1.11 \cdot 10^{-4}$	0.10 ± 0.02^{c}	0.06 ± 0.05^{b}
	M-L4	0.48 ± 0.00^{de}	0.049 ± 0.004^{b}	$4.47 \cdot 10^{-4}$	0.41 ± 0.10^{bc}	0.14 ± 0.07^{b}
	M-L8	0.40 ± 0.05^{e}	0.233 ± 0.043^{a}	$6.43 \cdot 10^{-4}$	3.77 ± 0.12^{a}	0.57 ± 0.02^{a}
	M-H2.6	0.51 ± 0.02^{d}	0.050 ± 0.003^{b}	$4.22 \cdot 10^{-4}$	0.62 ± 0.18^{b}	0.16 ± 0.05^{b}
	M-FF	0.77 ± 0.05^{b}	0.004 ± 0.001^{b}	$3.06 \cdot 10^{-5}$	0.18 ± 0.30^{c}	0.10 ± 0.14^{b}
Soup	S-0	0.83 ± 0.01^{a}	0.015 ± 0.000^{e}	$1.89 \cdot 10^{-4}$	0.02 ± 0.03^{d}	0.11 ± 0.02^{c}
	S-L2	0.59 ± 0.00^{b}	0.076 ± 0.001^{d}	$1.71 \cdot 10^{-3}$	0.27 ± 0.12^{d}	0.23 ± 0.02^{c}
	S-L4	0.42 ± 0.01^{c}	0.309 ± 0.014^{b}	$2.40 \cdot 10^{-3}$	1.90 ± 0.10^{b}	0.63 ± 0.04^{b}
	S-L8	0.34 ± 0.00^{d}	1.630 ± 0.004^{a}	$1.63 \cdot 10^{-2}$	21.67 ± 0.58^{a}	3.40 ± 0.12^{a}
	S–H2.6	0.43 ± 0.01^{c}	0.225 ± 0.008^{c}	$9.65 \cdot 10^{-3}$	1.03 ± 0.06^{c}	0.48 ± 0.02^{b}

[a-e] For each liquid matrix (milk/soup), samples sharing the same letter within a column are not significantly different from each other ($p > 0.05$).

dried MFC powder with high MFC:maltodextrin ratio contains relatively more MFC and therefore a lower proportion of maltodextrin. As maltodextrin impairs agglomeration of MFC, more junction zones might be formed between cellulose microfibrils at higher MFC:maltodextrin ratio and a stronger and denser MFC network might be formed. At constant MFC content fewer of such denser particles might be acquired compared to lower MFC:maltodextrin ratios, resulting in lower viscosity.

3.2.2. Storage and loss moduli at 1% strain

Storage and loss moduli (G' and G'') at 1% strain obtained at a frequency of 1 Hz of skimmed milks and tomato soups increased with increasing concentration of spray-dried MFC particles (Table 4). Milks and soups without spray-dried MFC powder (M-0, S-0) displayed liquid-like behaviour with loss modulus G'' being larger than storage modulus G'. Upon addition of spray-dried MFC powder more elastic behaviour was observed, as storage modulus G' was larger than loss modulus G'' in the linear viscoelastic regime under these test conditions. A clear increase in storage moduli G' and G'' was observed upon increasing the concentration of MFC:MD-Low. This was expected, as a higher volume fraction of spray-dried MFC particles results in increased flow resistance. Storage and loss moduli were higher for soups than for milks. It should be emphasised that M-L2, M-L4 and M-H2.6 exhibited only weak viscoelastic behaviour under the current test conditions. Storage and loss moduli were strongly positively correlated with shear viscosity parameters ($\eta 10s^{-1}$; $\eta 50s^{-1}$ and $\eta 100s^{-1}$, consistency index K; Table 5).

3.3. Tribological properties

Tribological properties of milks and soups are displayed in Fig. 4 and Table 6. Milks to which varying concentrations of spray-dried MFC were added displayed comparable friction properties, as their friction curves largely overlap. The fact that no significant differences in friction parameters were observed between milks differing in concentration of added spray-dried MFC and those without MFC suggests that the spherical MFC particles were not entrained between the PDMS pins and the glass ball (Table 6). We hypothesise that the high shear rates arising in the tribological set-up disrupt the spherical network formed by MFC. Individual microfibrils and aggregates thereof are released into the continuous phase, where these can form volume-spanning networks similar to those in never-dried MFC dispersions. Previously, such MFC networks have been postulated to be excluded from the contact zone due to their large hydrodynamic volume (Blok et al., 2021). Exclusion of the particles implies that tribological properties of the continuous phase of the milk samples were determined instead. Considering the continuous phase consists of skimmed milk with maltodextrin, the friction properties may therefore be governed by the proteins present in the milk (Chojnicka-Paszun, de Jongh, & de Kruif, 2012). Friction properties of full-fat milk on the other hand are significantly lower than those of the other milk samples over the entire speed range. This most probably results from the higher fat content of full-fat milk, as fat can form a lubricating film on the tribological surfaces. Although the increase in friction coefficient from 10 mm/s onwards appears to be the start of the hydrodynamic regime, this is refuted by the decline in friction coefficient starting around 400 mm/s. It is speculated that the lubricating fat film is disrupted at high sliding speeds due to starvation. The amount of lubricant in the contact zone might be insufficient to maintain complete separation of the tribological surfaces.

In contrast to milks, tribological properties of soups depended on the concentration of MFC powder added to the soup (Fig. 4, Table 6). We speculate that the higher viscosity of soups facilitated entrainment of the fluid between the tribo-pair surfaces, increasing the distance between them. This increase in gap size might potentially have promoted entrainment of MFC particles and fragments thereof. The presence of such polymer networks would affect the tribological properties of the sample, and could promote the transition from boundary to mixed lubrication at lower sliding speeds (Cassin, Heinrich, & Spikes, 2001). An alternative speculative explanation would be the presence of other polymers in the soup (i.e. starch and guar gum). After disruption of the spherical MFC particles due to the high shear in the tribological contact zone, fragments of MFC networks might have been able to interact with these biopolymers. As S-L8 contains more MFC compared to S-L2, more interactions between MFC and the other biopolymers might have occurred in soups with higher concentrations of MFC powder. The interactions between MFC network fragments and other polymers might have resulted in stronger network formation and higher viscosity, which would in turn affect the tribological properties of the soup such as an earlier transition from boundary to mixed lubrication.

It should be noted that the PDMS pins used in the tribological set-up do not closely resemble the tongue tissue due to differences in hydrophilicity and roughness and no saliva was added to samples for the tribological measurements. Still the combination of a glass ball and PDMS pins is considered the best option to determine oral lubrication, and the fact that these materials are used in the majority of studies facilitates comparison between various research groups (Rudge, Scholten, & Dijksman, 2019).

3.4. Sensory properties

A linear increase in thickness intensity was observed upon increasing

Table 5
Pearson correlations between sensory and instrumental parameters. Data from milks and soups were pooled (n = 11).

	Thickness	Creaminess	η_{10s}^{-1}	η_{50s}^{-1}	η_{100s}^{-1}	Flow index *n*	Consistency index *K*	Storage modulus G' [a]	Loss modulus G" [a]	μ_{max}	Start of mixed regime	Slope mixed regime
Thickness	–	0.974***	0.710*	0.711*	0.708*	−0.864***	0.707*	0.707*	0.707*	n.s.	n.s.	n.s.
Creaminess		–	0.736**	0.732*	0.725*	−0.848***	0.736**	0.730*	0.732*	n.s.	n.s.	n.s.
η_{10s}^{-1}			–	0.997***	0.992***	n.s.	1.000***	0.988***	0.999***	n.s.	n.s.	n.s.
η_{50s}^{-1}				–	0.999***	n.s.	0.995***	0.975***	0.994***	n.s.	n.s.	n.s.
η_{100s}^{-1}					–	−0.605*	0.989***	0.964***	0.987***	n.s.	n.s.	n.s.
Flow index *n*						–	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.
Consistency index *K*							–	0.990***	0.994***	n.s.	n.s.	n.s.
Storage modulus G' [a]								–	0.993***	n.s.	n.s.	n.s.
Loss modulus G" [a]									–	n.s.	n.s.	n.s.
μ_{max}										–	n.s.	n.s.
Start of mixed regime											–	0.854***
Slope mixed regime												–

Asterisks indicate statistically significant differences: (*) $p < 0.05$; (**) $p < 0.01$; (***) $p < 0.001$, n.s. = not significant.
[a] Storage modulus G′ and loss modulus G″ were determined at 1% strain at a frequency of 1 Hz.

the concentration of MFC:MD-Low in milks and soups (Figs. 5 and 6). The only exceptions were M-L2 and S-L2, which were not significantly thicker than milk or soup without MFC particles (M-0 and S-0). Moreover, thickness of commercial full-fat milk (M-FF) was not significantly different from skimmed milk without MFC particles to which 7.5 wt% maltodextrin was added (M-0). Similar to thickness, creaminess intensity of milks and soups increased linearly with increasing concentration of MFC:MD-Low (Figs. 5 and 6). While the presence of 2% spray-dried MFC:MD-Low did not affect thickness of soups and milks, creaminess of soup was significantly enhanced by addition of 2% MFC:MD-Low (corresponding to 0.075 wt% MFC) compared to soups without added spray-dried MFC particles. This effect on creaminess appears to be matrix-dependent, as it was not observed for milk. Milk presumably possesses a certain degree of intrinsic creaminess, which was not surpassed by addition of a small amount of MFC particles. No significant differences in creaminess were observed between M-0, M-L2 and M-FF, which was not expected. Full-fat milk contains more fat than the milk samples prepared from skimmed milk, and full-fat milk was therefore expected to have a creamier mouthfeel. It should be recalled that M-0 contained 7.5 wt% maltodextrin and had similar viscosity to M-FF (Fig. 3a), which might explain why M-FF was not perceived as thicker nor creamier than M-0. Milks and soups to which the highest concentration of MFC particles were added (M-L8 and S-L8) were significantly thicker and creamier than all other samples. Remarkably, M-H2.6 and S–H2.6 displayed thickness and creaminess intensities comparable to those of M-L4 and S-L4, while MFC and maltodextrin content of these samples were matched to those of M-L8 and S-L8. The analogy between these samples is in line with results on the samples' rheological properties (Fig. 3, Table 4). The similarity between M-H2.6, S–H2.6, M-H4 and S–H4 is hypothesised to be caused by differences in the (number of) particles formed upon spray-drying, as illustrated in Fig. 2.

The increase in thickness intensity upon increasing the concentration of MFC:MD-Low is fairly comparable for milk and soup, while the slopes for creaminess show modest differences between the two liquid matrices (Fig. 6). For soup a stronger increase in creaminess was achieved upon addition of MFC particles compared to milk. Initial creaminess for soup without MFC particles (S-0) started at lower creaminess intensity but ended at an intensity higher than that of milk at high MFC particle concentration (S-L8). This implies that the skimmed milk used in the current study was generally perceived as creamier than the tomato soup before addition of MFC particles, but soups became creamier more rapidly once MFC particles were added. Addition of MFC particles thus results in a more efficient enhancement of creaminess in soups compared to milks, which is reflected by the slope (Fig. 6b) that was 76% larger for soup compared to milk (6.7% vs 3.8% higher creaminess per wt% MFC:MD-Low; Fig. 6b). This suggests that an interaction effect exists between creaminess and the matrix in which the particles are suspended. As thickness and creaminess intensities increase concurrently (Figs. 5 and 6) the results moreover suggest that perceived creaminess is largely determined by the perceived thickness of the samples, which is in accordance with literature (e.g. Blok, Bolhuis, & Stieger, 2020; Janhøj, Frøst, & Ipsen, 2008; Kokini & Cussler, 1983). The significant positive correlations found between sensory thickness and creaminess intensities substantiate this postulation: $r = 0.53$ ($p < 0.001$) for milks, $r = 0.64$ ($p < 0.001$) for soups (data not shown). Pooling the two datasets reveals an exceptionally strong correlation between thickness and creaminess intensities ($r = 0.974$, $p < 0.001$; Table 5), which is a result of these correlations being based on the mean values ($n = 11$) instead of the raw sensory data ($n = 792$). The results demonstrate that spray-dried MFC particles enhance sensory thickness and creaminess of liquid foods and can thus be used to modify texture of such foods, for instance as thickener or creamer.

Strong positive correlations were found between the sensory texture attributes and the majority of rheological parameters ($r = 0.707$–0.974; Table 5), which demonstrates that rheological properties were important drivers of sensory perception of thickness and creaminess. On the other hand no statistically significant correlations were observed between the sensory texture attributes and tribological properties of the samples. This can be attributed to the fact that friction properties could not be determined as intended due to exclusion of MFC particles from the tribological gap. It can, however, not be excluded that friction in the mouth may be different from that in the tribological set-up used in this study. In the oral cavity, MFC particles might be present between the tongue and the palate where these could contribute to creaminess through lubrication.

4. Conclusions

The present work examined the effect of concentration and MFC:maltodextrin ratio of spray-dried MFC powders on rheological, tribological and sensory texture properties of liquid foods. Spray-dried MFC powders were prepared in a surplus of maltodextrin at two ratios and applied at different concentrations in skimmed milk and instant tomato soup. An increase in the concentration of spray-dried MFC resulted in higher viscosity, consistency index *K*, storage and loss modulus (*G'*, *G"*) at 1% strain and a lower flow index *n*. At concentrations above 2 wt% (corresponding to 0.075 wt% MFC), weak gel-like behaviour was observed in milks and soups. Increasing the concentration of spray-dried MFC particles substantially increased perceived thickness and creaminess of milks and soups. Creaminess was positively correlated with perceived thickness, and both sensory attributes were strongly correlated with rheological parameters.

SEM images demonstrated that both powders yielded a polydisperse particle size distribution. Three-dimensional spherical MFC networks

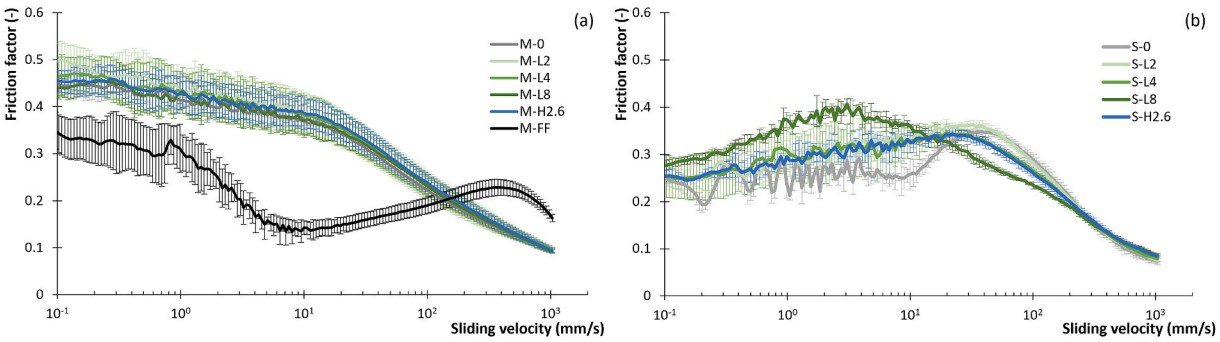

Fig. 4. Tribological properties of (a) milks and (b) soups to which two types of spray-dried MFC were added at different concentrations. Means are displayed with error bars representing standard deviations (triplicates). Sample codes and composition are summarised in Table 2.

Table 6
Means ± standard deviations of triplicate measurements of tribological properties of milks and soups to which two types of spray-dried MFC were added at different concentrations (for sample codes and composition: see Table 2). μ_{max} represents the maximum friction coefficient in the boundary regime. For soups, the start of the mixed regime was defined as the speed at which μ_{max} occurred, while this parameter was determined visually by the researcher for milks. The slope in the mixed regime was defined as the slope between the start of the mixed regime and the friction at 1000 mm/s.

	Sample	Maximum friction in boundary regime μ_{max}	Sliding speed representing start of mixed regime (mm/s)	Slope in mixed regime (10^{-4} s/mm)
Milk	M-0	0.46 ± 0.03^{ab}	12.6 ± 0.0^{b}	-2.67 ± 0.06^{b}
	M-L2	0.51 ± 0.03^{a}	13.4 ± 1.9^{b}	-2.75 ± 0.37^{b}
	M-L4	0.48 ± 0.03^{a}	19.4 ± 0.6^{b}	-2.51 ± 0.28^{b}
	M-L8	0.47 ± 0.03^{ab}	23.4 ± 0.0^{b}	-2.39 ± 0.22^{b}
	M-H2.6	0.46 ± 0.03^{ab}	18.3 ± 1.2^{b}	-2.63 ± 0.25^{b}
	M-FF	0.39 ± 0.04^{b}	469.2 ± 26.5^{a}	-1.12 ± 0.15^{a}
Soup	S-0	0.35 ± 0.01^{b}	37.0 ± 4.2^{a}	-3.14 ± 0.19^{a}
	S-L2	0.36 ± 0.01^{b}	27.6 ± 5.4^{a}	-3.25 ± 0.15^{a}
	S-L4	0.36 ± 0.02^{b}	21.1 ± 6.1^{a}	-3.05 ± 0.26^{a}
	S-L8	0.41 ± 0.01^{a}	2.0 ± 0.2^{b}	-3.42 ± 0.10^{a}
	S–H2.6	0.35 ± 0.01^{b}	20.7 ± 3.6^{a}	-2.91 ± 0.08^{a}

[a-b]: For each matrix (milk/soup), samples sharing the same letter within a column are not significantly different from each other ($p > 0.05$).

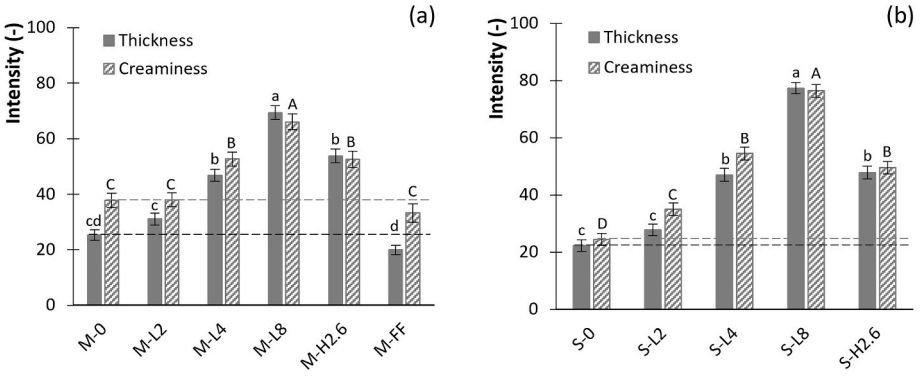

Fig. 5. Mean (±SEM) perceived thickness and creaminess intensities in (a) milks and (b) soups differing in concentration and type of added spray-dried MFC. Samples sharing the same letter are not significantly different from each other ($p > 0.05$). Sample codes and composition are summarised in Table 2.

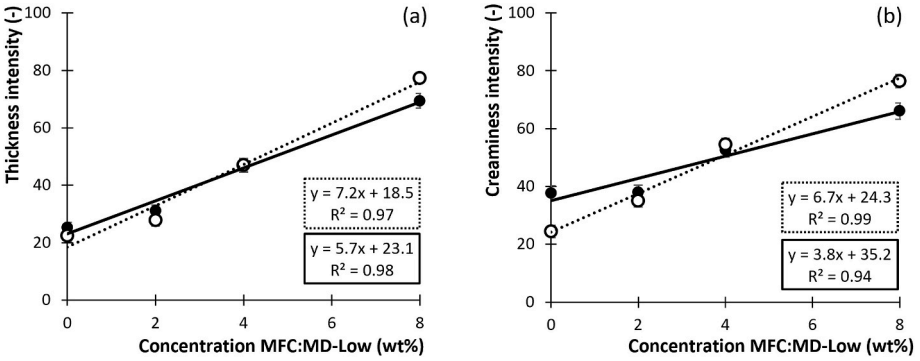

Fig. 6. Intensity of (a) thickness and (b) creaminess as a function of concentration of MFC:MD-Low particles (mean ± SEM) added to milk (filled symbols) and soup (open symbols). Linear trendlines are plotted to guide the eye.

were formed that did not disintegrate upon suspension in water, while maltodextrin dissolved in the continuous phase. High MFC:maltodextrin ratio particles were less effective at enhancing rheological and sensory texture properties of liquid foods compared to particles with low MFC: maltodextrin ratio, probably due to the lower volume fraction of particles that can be obtained with the same amount of MFC. No clear lubrication effect of the dried MFC particles could be established, due to exclusion of the particles from the tribological gap.

Spray-drying of MFC in the presence of maltodextrin prevents hornification and improves transportation and storage efficiency compared to MFC dispersions, which may provide financial benefits compared to liquid MFC dispersions. Based on the results presented, we conclude that spray-dried MFC particles can be used in liquid foods as thickener or even as a fat replacer due to their effect on rheological properties, sensory thickness and creaminess. One of the advantages of this material is its easy incorporation in foods by food manufacturers compared to the use of liquid MFC dispersions. The material furthermore shows potential as a thickener or creamer for direct use by consumers. In case spray-dried MFC particles are used to reduce the energy density of foods, for example in low-fat foods, replacing maltodextrin by less calorie dense materials should be considered.

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Author statement

Annelies E. Blok: Conceptualization, Methodology, Investigation, Formal Analysis, Writing – Original Draft.

Dieuwerke P. Bolhuis: Conceptualization, Methodology, Supervision, Writing – Review & Editing.

Krassimir P. Velikov: Conceptualization, Methodology, Writing – Review & Editing.

Markus Stieger: Conceptualization, Methodology, Supervision, Writing – Review & Editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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