



Influence of fluidized bed process parameters and material properties on plant powder characteristics and reconstitution behavior

K. Kramm^a, S. Pietsch-Braune^{a,*}, V. Meunier^b, K. Haas^b, S. Heinrich^a

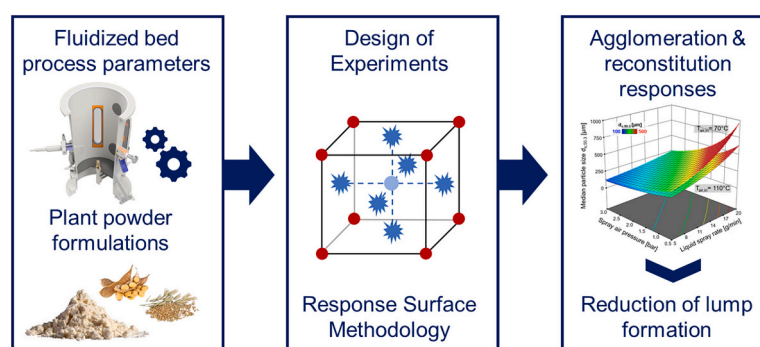
^a Hamburg University of Technology, Institute of Solids Process Engineering and Particle Technology, Denickestraße 15, 21073 Hamburg, Germany

^b Nestlé Research, Vers-Chez-Les-Blanc, Route du Jorat, 1005 Lausanne, Switzerland

HIGHLIGHTS

- Water activity and glass transition temperature impact the powder wettability.
- Depending on the formulation, different process parameter combinations were found for optimization.
- Correlation between contact angle and lump diameter was established.
- Powder formulations have the main influence on the formed lumps.

GRAPHICAL ABSTRACT



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ABSTRACT

Plant-based milk alternatives (PBMA) are increasingly used in food applications owing to their nutritional value and lower environmental impact compared with conventional dairy products. However, their successful reconstitution remains challenging because plant proteins and dietary fibers promote lump formation, thereby impairing wetting and dissolution. Although fluidized bed agglomeration is widely applied to improve the instant properties of food powders, the combined influence of formulation composition and process parameters on the reconstitution behavior of PBMA has not yet been systematically investigated. In this study, three representative PBMA formulations with different dominant compositional characteristics were agglomerated in a fluidized bed. A statistical Design of Experiments (DoE) approach was applied to investigate the interaction between formulation composition and fluidized bed process parameters on powder structure, wettability and lump formation during reconstitution. The results identified liquid spray rate and spray air pressure as the dominant process parameters governing agglomerate growth, while formulation composition strongly affected powder wettability. The protein- and fiber-enriched formulations exhibited larger contact angles and reduced wettability than the carbohydrate-rich reference formulation. Furthermore, contact angle correlated with lump diameter, indicating that wetting represents a rate-limiting step during powder reconstitution. The generated DoE models provide a

Abbreviations: DE, dextrose equivalent; DoE, Design of Experiment; PBMA, plant-based milk alternative.

* Corresponding author.

E-mail address: swantje.pietsch@tuhh.de (S. Pietsch-Braune).

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framework for formulation-specific adjustment of fluidized bed process parameters to tailor powder structure and minimize lump formation.

1. Introduction

The demand for plant-based milk alternatives (PBMA) has increased considerably in recent years, driven by health, environmental, ethical and animal welfare concerns [1–3]. In addition to providing valuable nutrients, PBMA are generally associated with a lower environmental impact than conventional cow's milk, including reduced greenhouse gas emissions and freshwater consumption, although the magnitude of these benefits depends on the raw materials and production systems employed. To improve shelf life, reduce transportation and storage costs, and facilitate their incorporation into a broad range of food applications, PBMA are increasingly commercialized as powder products. However, despite these practical advantages, the successful reconstitution of plant-based powders remains considerably more challenging than that of conventional dairy powders due to their more complex multi-component composition.

Powder reconstitution is a complex multi-step process comprising wetting, sinking, dispersion and dissolution [4]. Efficient reconstitution requires rapid penetration of water into the porous powder structure, followed by particle disintegration and complete dissolution. Insufficient wetting frequently leads to the formation of persistent lumps, which hinder water transport into the particle interior, delay hydration and negatively affect product quality and consumer acceptance. Consequently, understanding the mechanisms governing lump formation is essential for the rational design of instant plant-based powders.

The reconstitution behavior of food powders is strongly governed by both particle structure and material composition. Besides particle size and porosity, properties such as wettability, surface composition, moisture content and glass transition behavior determine liquid penetration and dispersion kinetics [5,6]. Compared with dairy powders, PBMA represent considerably more complex multi-component systems comprising carbohydrates, proteins, lipids and dietary fibers, whose interactions during reconstitution are still poorly understood [3,7]. In particular, plant proteins and dietary fibers may substantially alter wettability, moisture sorption and structural stability, thereby affecting the formation and persistence of lumps. Furthermore, plant proteins differ fundamentally from dairy proteins in their molecular structure, surface activity and hydration behavior, which may influence particle formation during spray drying and fluidized bed agglomeration as well as the subsequent reconstitution process.

Fluidized bed agglomeration is commonly employed to improve the instant properties of food powders due to its high heat and mass transfer rates, which enable short processing times at comparatively low temperatures [8,9]. Previous studies have consistently identified liquid spray rate, atomization conditions and drying kinetics as the key process variables governing agglomerate growth and structure [10–12]. Under the laboratory-scale conditions investigated by Hemati et al. [10], the nozzle position and the associated process parameters were identified as critical factors controlling agglomeration. The atomization process determines droplet size distribution, spray pattern and particle wetting and is therefore essential for the formation of stable liquid bridges between particles [11]. In agreement with these findings, Kramm et al. [13] demonstrated that spray air pressure primarily governs spray angle, radial mass distribution and droplet size distribution of a two-fluid nozzle. In addition to the atomization conditions, increasing liquid spray rates promote particle growth by enhancing particle wetting and adhesion during collisions [14,15]. Drying kinetics, which are largely controlled by the inlet air temperature, further determine the moisture content and consolidation of the agglomerates [10,16]. Besides these process parameters, agglomerate formation is strongly affected by the material properties, including particle size distribution, wettability,

solubility and formulation composition [15,16]. Consequently, understanding the interactions between formulation composition and process parameters is essential for the targeted design of powder properties.

Although numerous studies have investigated either formulation composition or fluidized bed operating conditions individually, the combined influence of both aspects on the structure formation and reconstitution behavior of PBMA has not yet been systematically investigated. This knowledge gap is particularly relevant because the increasing complexity of plant-based formulations limits the direct transferability of findings obtained for conventional dairy powders. Furthermore, quantitative investigations linking formulation composition, agglomeration process parameters and reconstitution behavior using a statistical Design of Experiments (DoE) approach remain scarce.

Therefore, the objective of this study was to systematically investigate the interactions between formulation composition and fluidized bed process parameters using a DoE approach. Accordingly, the objective was establishing the relationships between powder structure, wettability and lump formation during reconstitution. The results provide new insights into the coupled effects of formulation composition and agglomeration process parameters on powder structure and reconstitution behavior and thereby support the targeted design of plant-based powders with improved instant properties.

2. Materials and methods

2.1. Materials

The study was conducted using a plant-based alternative to conventional cow's milk. A simplified model system was developed to facilitate the transfer of the obtained findings to more complex formulations. The formulation consisted of glucose syrup (dextrose equivalent, DE 21), soy protein isolate, sunflower oil, and oat bran. To date, studies examining the combination of the plant-based ingredients soy protein isolate and oat bran within the complex matrix of carbohydrate and lipid sources have not been reported. The raw material Glucidex DE 21 (Azelis GmbH, Germany) was used as the raw material for the amorphous, hygroscopic polysaccharide glucose syrup DE 21. The soy protein isolate ProFam® 974 (ADM, USA) conducted as the protein source and has a crude protein content in dry matter according to DIN EN ISO 16634-1:2009 of 93.6% and an ash content of 4.50% with a moisture content of 5.1 g/100 g based on internal analyses. The lipid component is sunflower oil (Florin AG, Switzerland), which is characterized through gas chromatography flame ionization detector by a fatty acid content of palmitic acid C16:0 of 5.3%, stearic acid C18:0 of 2.7%, oleic acid C18:1 of 39.8% and linoleic acid C18:2 of 45.0%. The raw material OatWell® 28 (DSM-Firmenich AG, The Netherlands) was used to introduce the oat fiber β -glucan in the model system. The oat bran is featured by a β -glucan content of 28 g/100 g, a fiber content of 52 g/100 g, a protein content of 19.9 g/100 g and a Total Kjeldahl Nitrogen of 3.2 g/100 g through conducted measurements.

Three representative PBMA formulations (Table 1) previously

Table 1

Overview of the formulations PP1, PP2 and PP3 in regards to raw material composition.

Formulation	Glucose syrup DE 21 [%wt]	Soy protein isolate [%wt]	Sunflower oil [%wt]	Oat bran [%wt]
PP1	53	20	20	7
PP2	50	38	10	2
PP3	68	20	10	2

established and characterized by Kramm et al. [7] were selected as model systems to investigate the interaction between formulation composition and fluidized bed process parameters on powder structure and reconstitution behavior. Owing to constraints related to emulsion stability, spray drying performance, powder stability and processability, independent variation of individual ingredients while maintaining all remaining components constant was not feasible. Consequently, increasing the concentration of one component required corresponding adjustments to the remaining formulation. The objective was not to investigate isolated ingredient effects but to evaluate formulation-process interactions in realistic multi-component systems representative of industrial PBMA. Accordingly, PP1 represents a fiber-enriched formulation, PP2 a protein-enriched formulation, whereas PP3 serves as a carbohydrate-rich reference formulation. The selected formulations were intentionally retained from the previous study to enable direct comparison between the intrinsic material characteristics of the powders and their behavior during fluidized bed agglomeration and subsequent reconstitution. PP3 was selected as the carbohydrate-rich reference formulation because it contains the lowest protein and dietary fiber contents while representing the simplest formulation within the investigated design space. It therefore serves as the baseline for evaluating the influence of increasing protein (PP2) and dietary fiber (PP1) contents on powder structure and reconstitution behavior. Since the objective of this study was to investigate formulation-process interactions under practically relevant conditions, a classical one-factor-at-a-time design was intentionally not pursued.

Prior to spray drying, the PBMA feed emulsions were prepared by dispersing the formulation components in demineralized water under continuous stirring. The crude emulsions were subsequently treated using a rotor-stator dispersing device (T50 Ultra-Turrax, IKA-Werke GmbH & Co. KG, Germany) to ensure complete dispersion of the oat bran and pre-emulsification of the sunflower oil. The resulting emulsions were then homogenized using a two-stage high-pressure homogenizer (TwinPanda 600, GEA Group AG, Germany) before spray drying to obtain the PBMA powders used for following fluidized bed agglomeration. A detailed description of the powder production process is provided by Kramm et al. [7].

2.2. Fluidized bed agglomeration

For the production of agglomerates from previously spray-dried PBMA powder, the ProCell LabSystem fluidized bed (FB) equipped with a GF3 insert (Glatt GmbH, Germany) was used. The trials were conducted with a mesh distributor plate of 18 cm diameter and a slit diameter of 120 μm . A minimum bed volume of 1 L of PBMA powders was fluidized within the process chamber at a volumetric flow rate ranging from 80 to 100 m^3/h . The volumetric flow was continuously adjusted based on the size of the agglomerates formed to ensure homogeneous fluidization of the bed material. The air utilized for fluidization was preheated to temperatures between 70 and 110 $^{\circ}\text{C}$ by an electric heater before entering the process chamber. The agglomeration trials were conducted with the two-fluid nozzle 970 S4 (Düsen-Schlick GmbH, Germany) with a liquid insert size of 1.0 mm and an air cap position of 5. The nozzle settings were selected based on preliminary experiments and previous investigations [13]. The nozzle was configured in bottom-spray or top-spray position in the process chamber and operated at a spray air pressure ranging from 0.5 to 3.0 bar. Water at room temperature was used as a spraying agent at a liquid spray rate of 5–20 g/min for agglomerate production. The parameter settings for the fluidized bed process were derived from preliminary tests and literature data to facilitate efficient agglomeration of the PBMA powder formulations.

2.3. Design of experiments

A response surface methodology (RSM)-based Design of Experiments

(DoE) was performed using the software Design-Expert (Stat-Ease Inc., USA). DoE facilitates the generation of extensive information regarding the causes of significant variations in output responses with a limited number of runs through multiple factor changes within an experiment [17]. The Response Surface Methodology enables the description of linear and non-linear interactions among multiple factors, whereby process optimization can be implemented [18,19]. In Response Surface Methodology, independent variables can be categorized into categorical and numerical factors. Categorical factors represent qualitative characteristics and thus possess discrete values. Conversely, numerical parameters denote quantitative characteristics that function as continuous variables within the experimental design. The study was performed with the face-centered Central Composite Design, as the exceeding of the test space would otherwise lead to issues in practice due to unfeasible settings. Five factors were investigated, comprising three numerical factors (liquid spray rate, spray air pressure and inlet air temperature) and two categorical factors (formulation and nozzle position). The investigated factors and response variables are summarized in Table 2. The experimental design comprised 120 experimental runs, including replicated center points, distributed over two experimental blocks.

The adequacy of the fitted response surface models was evaluated by analysis of variance (ANOVA). Model significance was assessed using the F-test ($p < 0.05$), while model adequacy was verified using the lack-of-fit test. The goodness-of-fit and predictive capability of the selected models were evaluated using the coefficients of determination (R^2 , adjusted R^2 and predicted R^2). The statistical characteristics of the fitted response surface models are summarized in Table 3, while the complete ANOVA tables are provided in the Appendix.

2.4. Analysis methods

For characterization of the PBMA products, several measurement approaches were used.

2.4.1. Particle size measurement

The particle size distribution was analyzed using dynamic image analysis based on the 2-camera principle of the CamSizer XT (Retsch Technology, Germany). The measurement was conducted according to Kramm et al. [7] with a compressed air unit at a dispersion pressure of 90 kPa. Besides the particle size distribution and the characteristic parameters, the Sauter mean diameter was calculated from the distribution data using the software MATLAB. The Sauter mean diameter is defined as the particle diameter of a monodisperse material system, which possesses a total volume and surface area equivalent to those of the corresponding polydisperse system. The Sauter mean diameter $d_{3,2}$ can be calculated using the specific surface area S_v , the particle surface area $\bar{d}^2$, the Heywood factor f and the arithmetic means of the particle volume $\bar{d}^3$:

Table 2

Investigated numerical and categorical factors together with the response variables used in the response surface Design of Experiments (DoE).

Factors		
Factor	Type	Levels
A: Liquid spray rate	Numerical	5, 12.5, 20 g/min
B: Spray air pressure	Numerical	0.5, 1.75, 3.0 bar
C: Inlet air temperature	Numerical	70, 90, 110 $^{\circ}\text{C}$
D: Formulation	Categorical	PP1, PP2, PP3
E: Nozzle position	Categorical	Top-spray, Bottom-spray
Responses		
Sauter mean diameter ($d_{3,2}$)		
Moisture content		
Contact angle		
Lump formation		

Table 3

Statistical characteristics of the fitted response surface models.

Response	Selected model	Model p -value	Lack-of-fit p -value	R^2	Adjusted R^2	Predicted R^2
Sauter mean diameter	Reduced 2FI	<0.0001	0.2831	0.7215	0.6957	0.6538
Moisture content	Reduced Quartic	<0.0001	0.9880	0.7634	0.6997	0.6025
Contact angle	Reduced Quartic	<0.0001	0.9274	0.8306	0.7501	0.6309
Lump formation	Reduced Cubic	<0.0001	0.1096	0.6688	0.5559	0.3018

$$d_{3.2} = \frac{6}{S_v} = \frac{\bar{d}^3}{\bar{d}^2} \cdot \frac{1}{f} \quad (1)$$

The Heywood factor quantifies the ratio of the measured specific surface area of the particles to the specific surface area of a sphere with a diameter d [20]. To ensure repeatability, the measurements were performed at least three times.

2.4.2. Sorption behavior and moisture content determination

The sorption isotherm was established according to Dupas-Langlet et al. [21] to characterize the sorption behavior of PBMA powders and to examine the influence of moisture content on the properties of the powder. To achieve uniform moistening and drying, the PBMA powder formulations were placed in dishes. The analysis was repeated at least in duplicates to ensure reproducibility. Humidification of the powder was performed by storing the dishes in desiccators containing a saturated sodium bromide (NaBr) solution, which provides a water activity value a_w of 0.567, for durations of 2, 4, 8, 16, and 24 h. The water activity a_w indicates the availability of free water within the powdered material and is defined as the ratio of the water vapor pressure in the immediate environment of the food p_v to the saturation vapor pressure of pure water at the same temperature p_s :

$$a_w = \frac{p_v}{p_s} \quad (2)$$

The parameter can assume values ranging from 0, which signifies complete dryness of the product, to 1, which represents pure water. Once the product system has reached equilibrium, the relative humidity of the surrounding environment corresponds to the water activity of the material [21]. The equilibrium water activity was determined experimentally using an AQUALAB 4TE instrument (Meter Group, USA) and subsequently used together with the moisture sorption data to determine the glass transition behavior by fitting the Gordon-Taylor equation. Measurements were conducted for a duration of one hour, utilizing approximately 1 g of PBMA sample in duplicate at a temperature of 25 °C. Drying of the PBMA powders was achieved in silica desiccators for periods of 8 and 24 h. The weight difference of the conditioned powder is measured post-storage, allowing for the calculation of moisture content on a wet basis:

$$M_{wb} = 100 - \frac{m_{initial}}{m_{final}} \cdot (100 - M_{wb,initial}) \quad (3)$$

Here, $m_{initial}$ and m_{final} represent the sample masses before and after humidifying or partial drying. The initial moisture content on wet basis $M_{wb,initial}$ was determined using the thermogravimetric differential thermal analysis (TG-DTA) method. Further details regarding this methodology can be found in the study by Vuataz et al. [22]. Initially, 20 mg of the powder samples were heated in open pans from 25 °C to 220 °C at a heating rate of 2 °C/min. The mass loss observed at the inflection point in the thermogravimetric curve, which indicates the end of water release, corresponds to the initial moisture content on wet basis. The conversion of moisture content from wet basis M_{wb} to dry basis M_{db} is expressed as follows:

$$M_{db} = \frac{M_{wb}}{\left(1 - \frac{M_{wb}}{100}\right)} \quad (4)$$

The obtained measurement points are fitted with the Brunauer,

Emmett and Teller (BET) sorption isotherm in Eq. 5 [23]:

$$\frac{w'}{w'_m} = \frac{C \cdot a_w}{(1 - a_w) \cdot (1 + (C - 1) \cdot a_w)} \quad (5)$$

In the equation, w' describes the moisture content in dry basis and w'_m the amount of water required for the formation of a monomolecular water layer on the solid surface. C is the material-specific, temperature-dependent constant. The parameters w'_m and C are used as fitting parameters of the measured data.

2.4.3. Glass transition temperature

The measurement of the glass transition temperature was performed to determine the physical state of the powders in relation to water activity and moisture content. The glass transition temperature was determined using the Differential Scanning Calorimeter DSC Q2000 (TA Instruments, USA). Therefore, the powder sample was put in duplicates of a maximum of 30 mg into pre-weighed aluminum pans with a volume of 40 μ L and closed with a lid. The heating rate was set at 5 °C/min, while the cooling rate was 20 °C/min. A double scan method was conducted for the measurement to minimize the influence of relaxation enthalpy, thereby enhancing the visibility of the glass transition during evaluation. The glass transition temperature T_g was determined from the second scan and is defined as the onset of the step change in heat capacity. The resulting data are fitted using the Gordon-Taylor eq. (6), considering the moisture-related water content w , the Gordon-Taylor constant k , the glass transition temperature of the anhydrous material $T_{g,s}$ and of pure water $T_{g,w}$ [24]:

$$T_g = \frac{(1 - w) \cdot T_{g,s} + k \cdot w \cdot T_{g,w}}{(1 - w) + k \cdot w} \quad (6)$$

The glass transition temperature of pure water has been reported in the literature to be approximately −135 °C. [25,26].

2.4.4. Wettability measurement

To determine the wetting behavior, tablets were prepared from the particulate product. The pressing process creates a flat surface, which facilitates accurate measurement of the contact angle and minimizes the influence of surface roughness on the results. The press PWV 200 E (Paul-Otto Weber GmbH, Germany) was used to produce cylindrical tablets with a diameter of 20 mm. For this purpose, 4.5 g of sample material was weighed and strained with a force of 80 kN for two minutes, resulting in an applied pressure of 453 MPa. All tablets were prepared using identical compaction conditions to ensure the direct comparability of the contact angle measurements between the investigated formulations. Although migration of surface lipids during compaction cannot be completely excluded, the standardized preparation procedure was applied consistently for all samples. Consequently, any potential influence of compaction on the measured contact angles is expected to affect all formulations in a comparable manner. The wettability is assessed by the sessile drop method using the contact angle goniometer model 100-00-115 (Ramé-hart Instruments Co., USA). On the PBMA powder tablets, a water droplet of 10 μ L volume was pipetted and a video of the wetting process was recorded. The high-speed camera captures 2500 images over 600 s. All measurements were performed in at least triplicate. It should be noted that tablet preparation modifies the porous structure of the agglomerates. Consequently, the contact angle measurements are intended to provide a comparative assessment of the

intrinsic wettability of the different formulations rather than to directly represent the wetting behavior of the porous agglomerates during reconstitution. Therefore, the results should be interpreted comparatively rather than as absolute contact angle values.

2.4.5. Determination of lump characteristics

To evaluate the reconstitution process, the powder samples were dissolved in demineralized water, and the process was monitored using the conductivity probe PT1000 (Metrohm GmbH & Co. KG, Germany). A surface propeller with a diameter of 3 cm and six blades facilitated the incorporation of the powder into the dissolution medium at a stirring speed of 200 rpm. To minimize concentration gradients at the sensor, a magnetic stirrer measuring 2.5 cm in length and 0.8 cm in width is used and operated at a rotational speed of 800 rpm. Fig. 1 illustrates the schematic setup of the measurement method. The experiments were conducted in triplicates using 150 mL of demineralized water and 5 g of the sample powder. To ensure a steady state of the conductivity value, the measurement duration was set to 600 s.

Prior to the reconstitution experiments, formulation-specific calibration curves were established using completely dissolved PBMA suspensions with known solids concentrations. Linear regression was performed within the linear concentration range (0–15 wt%), resulting in coefficients of determination of $R^2 \geq 0.99$ for all formulations. The resulting calibration equations (Table A.5) were used to convert conductivity values into dissolved solids concentrations during reconstitution experiments. The relative lump formation was subsequently calculated as the proportion of undissolved powder ($m_{undissolved}$) with respect to the initially added powder mass $m_{initial}$:

$$L = \frac{m_{undissolved}}{m_{initial}} \cdot 100 = \frac{m_{initial} - m_{dissolved}}{m_{initial}} \cdot 100 \quad (7)$$

In addition to the proportional identification of lumps, the undissolved solids were characterized according to their particle size distribution, which was measured by the Mastersizer 3000 with the wet dispersion unit Hydro MV (Malvern Panalytical, United Kingdom) by taking samples after the conductivity measurement. Upon adding the sample to the dispersing unit, a stirrer operating at a speed of 1600 rpm was used to prevent settling or separation of the emulsion sample within the dispersant. The sample concentration was monitored through the parameter of obscuration and maintained consistently within a range of 1–5%. The refractive was set to 1.33 and the analysis was performed in triplicates according to the Mie theory which assumes homogeneous spherical particles in the diluted suspension [27].

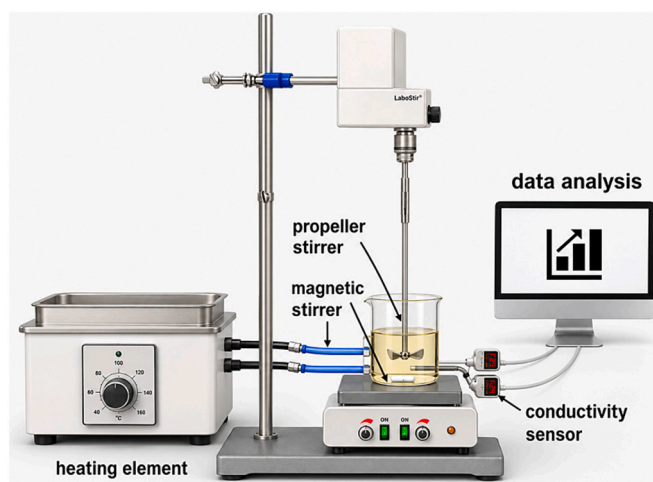


Fig. 1. Schematic setup of the conductivity measurement for the analysis of the powder dissolution behavior.

3. Results and discussion

To support the interpretation of the agglomeration results, the initial spray-dried PBMA powders were characterized prior to fluidized bed processing. The corresponding particle size distribution span, moisture content, bulk density and glass transition temperature are summarized in Table 4.

3.1. Sorption isotherm and glass transition temperature

Sorption isotherms were generated to investigate the influence of the moisture content on the material properties of the powder formulations and component behavior. The sorption isotherms shown in Fig. 2 (A) describe the relationship between the equilibrated moisture content of the sample on a dry basis and the water activity at a temperature of 25 °C. According to Dupat-Langlet et al. [21], the BET equation is most suitable for food powders in the water activity range of 0.05 to 0.5 and Abramović & Klofutar [28] also describe deviations from linearity at $a_w > 0.5$. Since glucose syrup DE 21 represented the predominant component of each PBMA formulation, the sorption behavior of the compositions is similar to the pure raw material [28,29]. The PBMA formulations are characterized by hygrocapacitive behavior, whereby PP1 with the highest content of oat bran in particular exhibits increasing differences from a water activity of 0.2 compared to the other recipes. Andrade et al. [30] attribute the differences in various water activities to the interactions of water molecules with the powder sample. In the low water activity range, the bound water is primarily absorbed by the hydrophilic and polar component groups and is thus not available as a plasticizer for the powder material. As the water activity increases, the interactions between surrounding water molecules and the product sample diminish, leading to instabilities [30]. The pronounced deviation observed for PP1 can be attributed to its altered formulation composition, particularly the higher dietary fiber content and the resulting changes in carbohydrate composition. Oat bran contributes both soluble β -glucan and insoluble components, which differ in their interaction with water molecules. Consequently, the observed sorption behavior should be interpreted as the combined effect of the overall formulation rather than the influence of a single ingredient.

The material properties of PBMA formulations are influenced by the temperature and moisture content of the environment due to the amorphous components. To identify the physical states, the glass transition temperature was determined as a function of moisture content (Fig. 2 B). The measured data points were fitted with the Gordon-Taylor eq. (6). An increase in the moisture content in the powder matrix reduced the glass transition temperature of the PBMA powders, as the adsorbed water acts as a plasticizer [31]. The plant product curves are similar to the pure carbohydrate raw material curve in literature [32–34]. In the low moisture range, PP2 is characterized by lower glass transition temperature values. However, PP1, with the highest proportion of oat bran in the PBMA formulation, indicates lower glass transition temperatures at powder moisture content of 6 g/100g_{wb}. Besides the dietary fiber fraction, the carbohydrate composition also contributes substantially to the glass transition behavior. Since glucose syrup represents the predominant component of all investigated formulations, while the plant protein and dietary fiber contents vary simultaneously,

Table 4

Characteristic properties of the initial spray-dried PBMA powders prior to fluidized bed agglomeration.

Property	PP1	PP2	PP3
Sauter mean diameter [μm]	47.5 ± 1.2	45.1 ± 0.9	38.1 ± 1.0
Span [–]	2.6 ± 0.1	2.7 ± 0.0	2.9 ± 0.1
Moisture content [g/100 g]	2.9 ± 0.4	3.2 ± 0.3	3.7 ± 0.6
Bulk density [g/L]	462.2 ± 34.0	377.0 ± 10.2	420.7 ± 23.3
Glass transition temperature [°C]	121.4	111.2	120.6

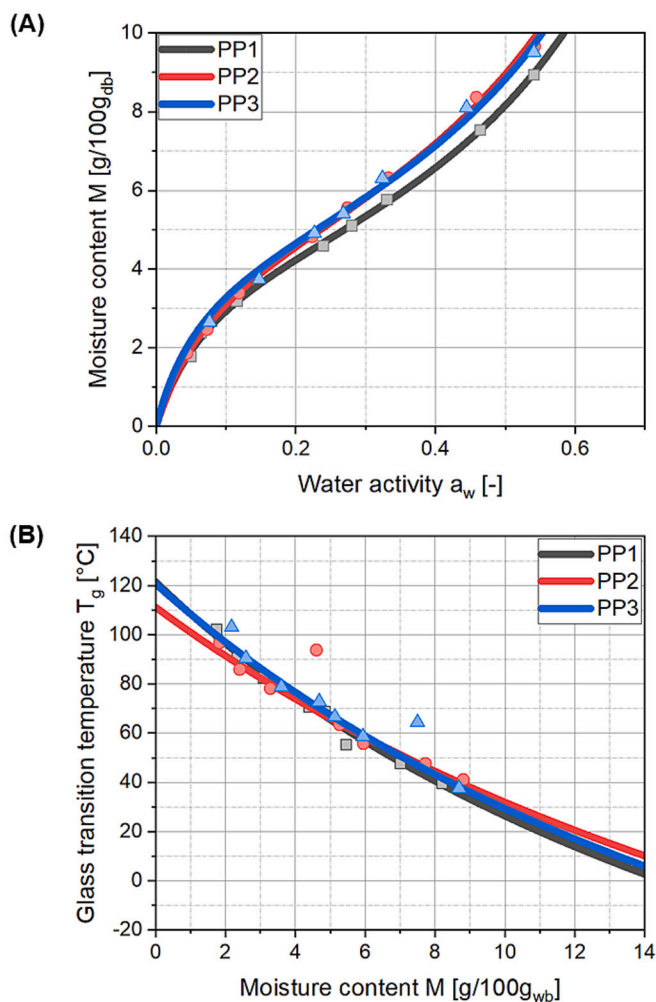


Fig. 2. (A) Sorption isotherm at 25 °C fitted with the BET model based on the generated measurement data on dry basis and (B) glass transition temperature with the fitted measurement points for the PBMA formulations PP1, PP2 and PP3.

the differences in glass transition temperature should be interpreted as the result of the overall formulation composition rather than the effect of an individual ingredient.

3.2. Parameter influence based on DoE

The respective responses are described in the following sections, while a comprehensive overview of variable interactions within the DoE can be found in the Appendix. The identified correlations among variables facilitate the production of customized products through fluidized bed agglomeration tailored to consumer needs.

3.2.1. Particle size

To understand the agglomerate growth as a function of the fluidized bed process parameters and the material properties, the characteristic particle size parameters after processing were incorporated into the DoE as responses. A quartic model with inverse square root transformation was used to characterize particle diameter. Therefore, the liquid spray rate and atomizing air pressure were identified in the ANOVA as the main impact parameters for all responses at $p < 0.05$. Fig. 3 illustrates the median particle diameter as a function of fluidized bed process parameters, averaged across PBMA formulations and nozzle positions. An increase in liquid spray rate combined with lower spray air pressures results in larger particle diameters. In the study by Kramm et al. [13], an

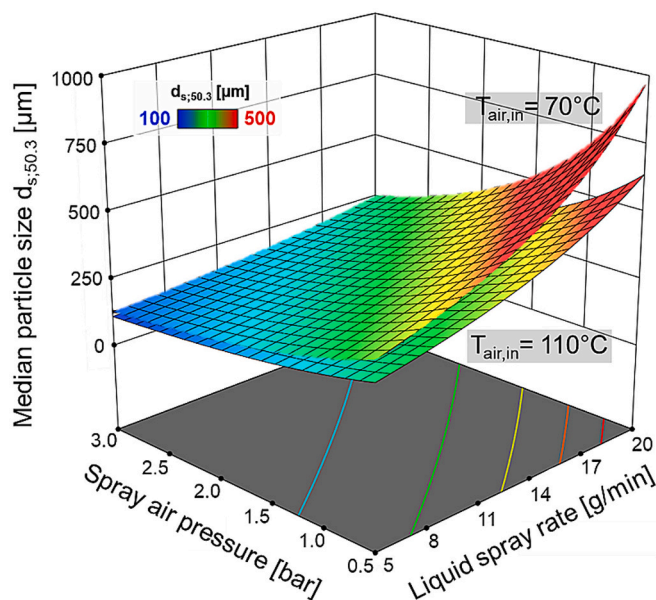


Fig. 3. Effect of spray air pressure and liquid spray rate at the minimum and maximum inlet air temperatures of 70 and 110 °C averaged over the nozzle position and PBMA formulations on the median particle size of agglomerates after processing.

increase in liquid flow rate is correlated with a more excentric droplet distribution and an increased spray cone angle. The combination of a high liquid spray rate with minimal spray air pressures leads to the largest droplet diameters at the nozzle tip [11], which increases the probability of agglomeration in the bed material [12]. Vengateson & Mohan [15] and Bouffard et al. [11] describe the association of reduced granule size and increased material abrasion with high atomizing air pressure. In addition to the atomized liquid feed, the temperature of the inlet air significantly influences moisture content within the powder bed, thus affecting final agglomerate size [11,16,35]. A lower inlet air temperature is characterized by a reduced evaporation rate and enhanced agglomerate growth, as depicted in Fig. 3. Hemati et al. [10] observed that most evaporation of sprayed liquid occurs near the atomizing nozzle. The continuously fluctuating relative humidity within the fluidized bed, resulting from evaporation, also impacts the formation of liquid bridges between particles by altering the cohesive properties of the fluidized bed material [10]. Additionally, rapid drying due to elevated inlet air temperatures correlates with fragile and non-compact solid bridges [12]. Besides the already mentioned process variables, agglomeration is affected by the position of the two-fluid nozzle and the PBMA formulations, which determine the fluidized bed process parameter settings during optimization through interactions. The observation in the DoE corresponds to the findings in the literature [10,15]. The two-fluid nozzle position in bottom spray configuration has a favorable effect on structuring by agglomeration over the recipe average. Besides the process parameters of the fluidized bed, the PBMA compositions also impact the responses of the characteristic particle size parameters.

Fig. 4 compares the measured Sauter mean diameters of the initial spray-dried powders with the DoE model predictions for the agglomerated powders obtained at an inlet air temperature of 70 °C, a liquid spray rate of 20 g/min and an atomizing air pressure of 0.5 bar, averaged over both nozzle configurations. The agglomeration process resulted in a substantial increase in particle size for all formulations. The Sauter mean diameter increased by approximately 17-fold for PP1, 12-fold for PP2 and 30-fold for formulation PP3 compared with the initial spray-dried powders. These formulation-dependent differences reflect the combined influence of powder composition and material properties,

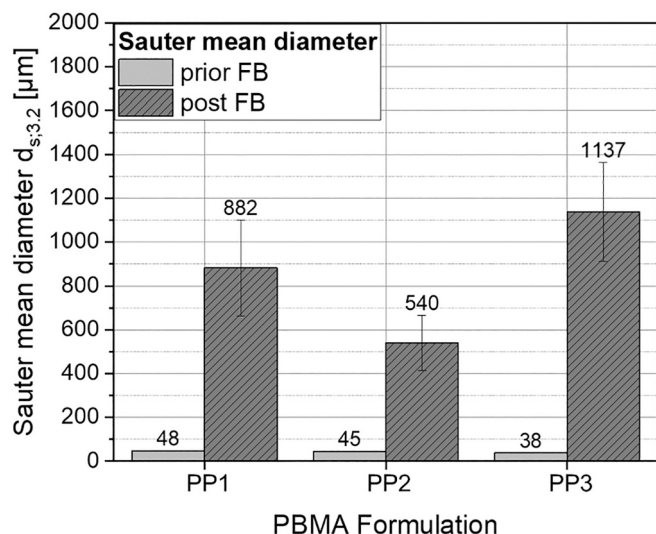


Fig. 4. Sauter mean diameter of the spray-dried powders before and after fluidized bed agglomeration for the three PBMA formulations. The agglomerated particle sizes correspond to the DoE model predictions for an inlet air temperature of 70 °C, a liquid spray rate of 20 g/min and an atomizing air pressure of 0.5 bar, averaged over both nozzle configurations.

including moisture sorption behavior, particle wettability and the physicochemical characteristics of the individual ingredients [7]. Since the investigated formulations differ simultaneously in their carbohydrate, protein and dietary fiber contents, the observed agglomeration behavior should be interpreted as the result of the overall formulation composition rather than the isolated effect of a single ingredient. The differences between the formulations therefore cannot be explained solely by the initial particle size or the concentration of a single component, but rather by the interaction between formulation composition and fluidized bed processing conditions. The study by Pont et al. [36] describe favored agglomerate growth at low contact angles. Hemati et al. [10] also describe a higher particle growth rate with smaller initial particle sizes of the bed material. The findings can be used as an additional explanation for the observed deviations. A comprehensive overview of the complete DoE results is presented in the Appendix. The analysis of the characteristic particle size parameters reveals that more pronounced agglomeration leads to smaller spherical particles with a reduced specific surface area and narrower size distribution compared to the smaller, slightly agglomerated powder particles. The results are consistent with the study by Vengateson & Mohan [15], which

demonstrated a decreasing particle size distribution with increasing liquid addition and consequently greater agglomerate formation.

Fig. 5 shows the direct applicability of the process through contour diagrams. To attain equivalent particle diameter, PP2 with the highest content of plant proteins requires higher liquid spray rates at generally lower spray air pressures compared to the other recipes. Furthermore, the influence of the process parameters exhibits varying intensities depending on the product composition.

3.2.2. Moisture content

The moisture content of the PBMA powder after processing in the fluidized bed impacts the product quality and has been investigated in more detail. The response was analyzed using a quadratic model with logarithmic transformation. The significant factors influencing moisture content with $p < 0.05$ were identified as the liquid spray rate, inlet air temperature, nozzle position, the interaction between liquid spray rate and inlet air temperature, the interaction of spray air pressure with PBMA formulations (two-factor interaction), and the interaction involving liquid spray rate, spray air pressure, powder recipe and nozzle position (four-factor interaction). However, the inlet air temperature and liquid spray rate exert the most substantial influence on moisture content. Fig. 6 indicates these parameters using the average of product compositions and nozzle position within the fluidized bed. An increase in fluidizing air temperature is associated with lower moisture content in the agglomerates, while higher liquid spray rates correspond to increased product moisture levels. Lipps & Sakr [16] identified the fluidization air temperature and liquid feed rate as the primary factors influencing the moisture content of the product within the fluidized bed, using Response Surface Methodology. Both parameters determine the particle moistening and the evaporation rate of the atomized liquid in the manufacturing process and can result in different growth kinetics depending on the product [16]. Palzer [35] describes a correlation between increased moisture content in product powders and larger average agglomerate sizes. The higher water addition with lower drying due to lower fluidization air temperatures increases the probability of moist particle collision and thus the agglomeration rate [12]. Based on the DoE correlation coefficient, the relation between the output factors of the characteristic particle size parameters and the moisture content of the PBMA powders could be confirmed. Higher product moisture contents were primarily observed when the liquid feed was atomized in the bottom-spray nozzle position. This resulted in higher moisture values associated with a tendency towards larger particle diameters, but the sphericity and specific surface area were reduced due to agglomerate formation. Furthermore, Bouffard et al. [11] reported that lower granulate abrasion occurred at higher bed moisture content within the fluidized bed. The combination of a high liquid spray rate and low spray air

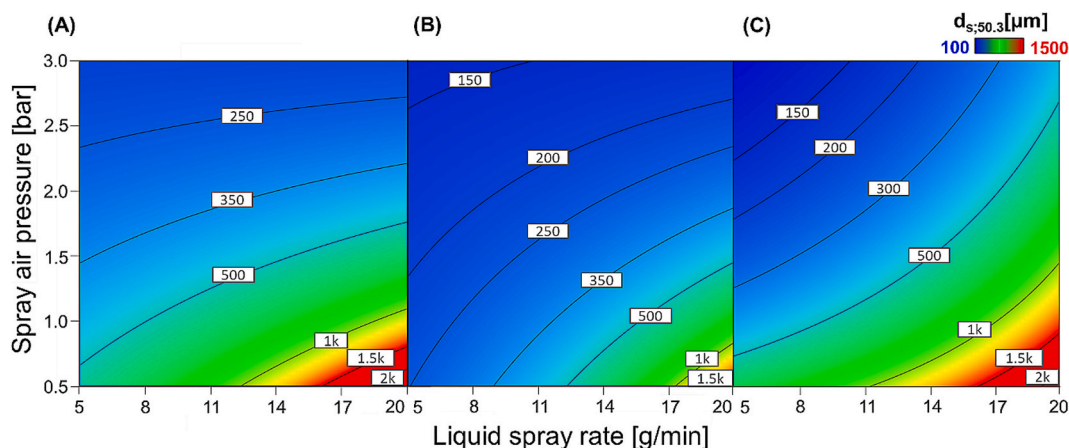


Fig. 5. Contour diagram of the median particle size as a function of the spray air pressure in relation to the liquid spray rate during fluidized bed processing at inlet air temperature of 70 °C for the nozzle position bottom-spray and the PBMA formulations (A) PP1, (B) PP2 and (C) PP3.

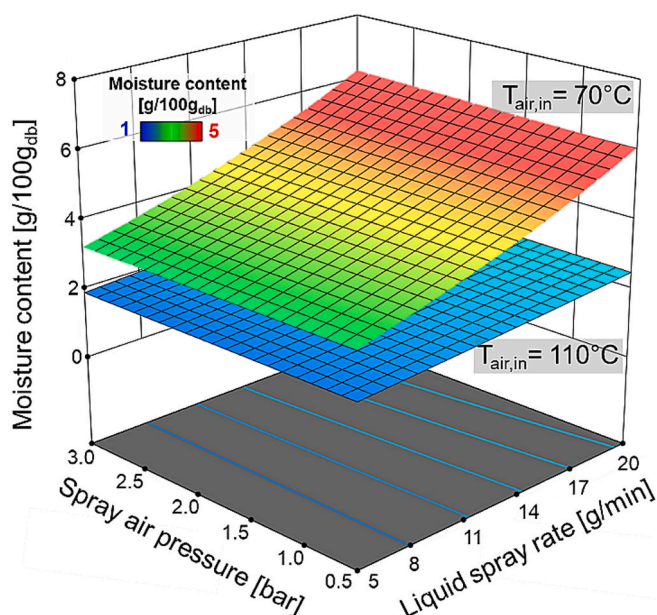


Fig. 6. Influence of spray air pressure and liquid spray rate at the minimum and maximum inlet air temperatures of 70 and 110 °C averaged over the nozzle position and PBMA formulations on the moisture content of the agglomerates after processing.

pressure of the two-fluid nozzle counteracts the abrasion through the formation of large liquid droplets.

Fig. 7 compares the moisture contents of the three PBMA formulations at constant liquid spray rate and inlet air temperature while varying the spray air pressure. As confirmed by the DoE analysis (see Table 3), the moisture content was primarily governed by the inlet air temperature and the liquid spray rate, whereas the main effect of spray air pressure was not statistically significant within the investigated design space. Therefore, the variations observed in Fig. 7 should be interpreted as formulation-dependent trends rather than statistically significant effects of atomization pressure. The slightly different moisture contents observed for the three PBMA formulations are likely related to differences in their sorption behavior and water-binding capacity (see Section 3.1), which are governed by the combined composition of carbohydrates, plant proteins and dietary fibers.

3.2.3. Wettability

The product quality of PBMA agglomerates was assessed by characterizing the powders based on the wetting process. Wettability was evaluated by contact angle measurements using compressed powder

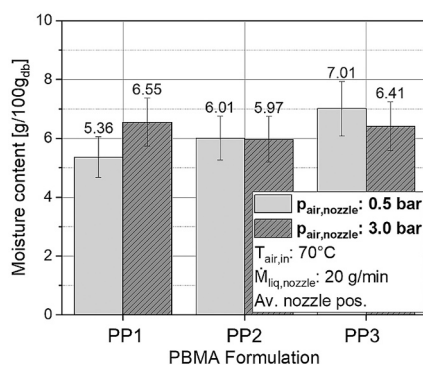


Fig. 7. Influence of the PBMA formulations in combination with the minimum and maximum spray air pressures of 0.5 and 3.0 bar with otherwise constant process parameters on the product moisture.

tablets. While the compaction process may have altered the powder structure and potentially induced surface lipids, these effects cannot be entirely excluded. Nevertheless, as all tablets were manufactured under identical compaction conditions, any associated changes are expected to be comparable among formulations. The contact angle data were analyzed using a quartic model with inverse transformation. Significant influencing variables identified on the response, with $p < 0.05$, included the liquid spray rate, fluidization air temperature, PBMA formulation, the interaction between formulation and nozzle position (four-factor interaction), the combination of atomization settings and powder composition (two-factor interaction), as well as the interaction of inlet air temperature with PBMA composition and nozzle position (three-factor interaction). However, the greatest influence on the contact angle is exerted by the powder formulation, followed by the inlet air temperature. Fig. 8 illustrates both input variables using contour diagrams. The PBMA products are characterized by capillary wetting at contact angles of less than 90°, indicating a positive capillary pressure [37]. The protein-enriched formulation PP2 exhibited the largest contact angles, while the fiber-enriched formulation PP1 also showed reduced wettability compared with the carbohydrate-rich reference formulation PP3. These differences likely reflect the combined influence of formulation composition, including changes in the relative proportions of carbohydrates, proteins and dietary fibers. Besides potential differences in particle surface properties, the hydration behavior and water-binding capacity of the individual ingredients may additionally contribute to the observed wettability. In particular, β -glucan has been reported to increase the viscosity of aqueous systems upon hydration, which may further impede the dispersion process during reconstitution. However, viscosity measurements were beyond the scope of the present study. Consequently, the individual contribution of each formulation component cannot be distinguished and the proposed mechanisms should be regarded as plausible explanations rather than direct conclusions from the present data. The contour diagrams indicate that fluidized bed process parameter settings for achieving effective wetting vary among formulations. Based on the comprehensive dataset generated, specific process parameter combinations were identified to influence the contact angle. For PP1, high liquid spray rates combined with medium spray air pressure and inlet air temperatures in the bottom-spray position of the two-fluid nozzle yielded the best wetting results within the investigated parameter range. Increased protein content in PP2 was associated with a process parameter combination characterized by a high liquid spray rate and high atomizing air pressure at low inlet air temperatures in top-spray configuration to minimize the contact angle. Conversely, reference formulation PP3 achieved the lowest contact angle values at a high liquid spray rate with a spray air pressure of 2 bar and a fluidizing air temperature of 85 °C in bottom-spray configuration.

In the study by Dupas et al. [5] a relationship between the reconstitution behavior, water activity and glass transition temperature was demonstrated for maltodextrins with different dextrose equivalent (DE) values. Therefore, the PBMA formulations are shown in Fig. 9 as a function of both material parameters. Although water activity and glass transition temperature are closely related through the moisture-dependent plasticization of the amorphous matrix and therefore exhibit similar trends, they describe different physicochemical properties of the powder. While water activity characterizes the thermodynamic state of the material, the glass transition temperature provides information on the physical state and molecular mobility of the amorphous components. Similar to the observations of Dupas et al. [5] the contact angle decreased with increasing water activity and the corresponding decrease in glass transition temperature. However, the glass transition temperature itself is not considered to directly determine the wetting behavior. Instead, the observed contact angles are attributed to the combined influence of moisture-dependent changes in the powder properties and the surface composition of the particles. Besides the moisture state, the distribution of hydrophilic carbohydrate-rich regions as well as surface-active proteins and hydrophobic lipid components at

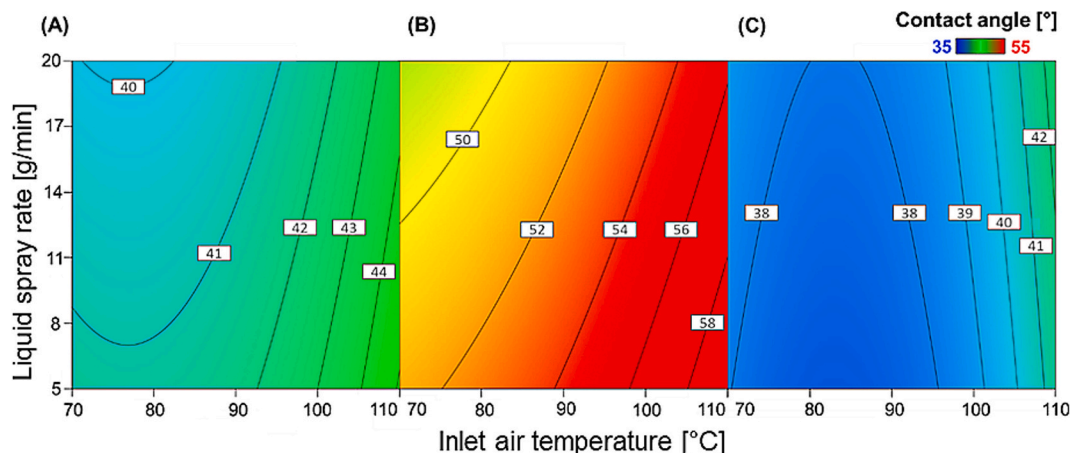


Fig. 8. Contour diagram of the contact angle as a function of the liquid spray rate in relation to the inlet air temperature during fluidized bed processing with a spray air pressure of 0.5 bar over the average of the nozzle position for the PBMA formulations (A) PP1, (B) PP2 and (C) PP3.

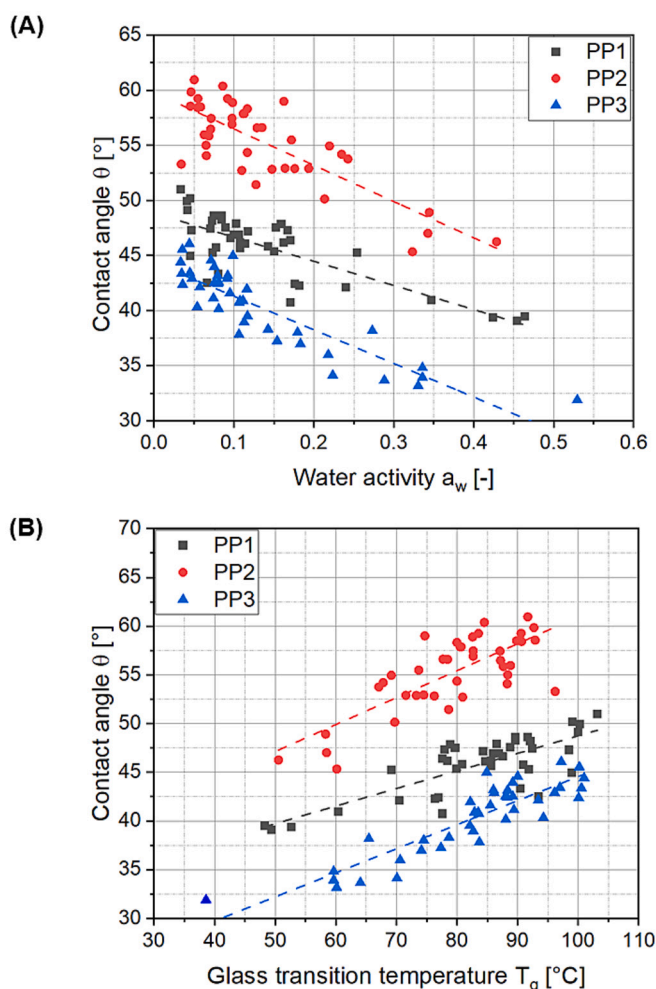


Fig. 9. Contact angle as a function of (A) water activity and (B) glass transition temperature of the tableted agglomerates of the PBMA formulations PP1, PP2 and PP3. Water activity and glass transition temperature are shown separately because they characterize the thermodynamic and physical state of the powders, respectively. The dashed lines indicate the overall trend.

the particle surface, capillary penetration and the subsequent dispersion mechanism. The material properties therefore affect liquid penetration into the porous particle structure and consequently the reconstitution

behavior [5]. For the investigated PBMA formulations, the results indicate that the wettability can be improved by controlling the moisture state of the powder through appropriate fluidized bed process parameters in combination with a suitable formulation composition.

3.2.4. Lump formation

A complete, lump-free dissolution process within an acceptable time is essential for the effective application of the product powder. To check these criteria, the agglomerated formulations were analyzed through conductivity measurements and particle size determination. Fig. 10 (A) indicates the interaction between the proportional lump formation and the Sauter mean diameter of the PBMA agglomerates. As the Sauter mean diameter of the product particles increases, lump formation decreases. A comparative analysis of the responses confirms this correlation. The formation and size of lumps are mitigated by agglomeration, which is associated with a decrease in sphericity, specific surface area, span, bulk density and an increase in hydraulic pore diameter (see Appendix). Dupas et al. [5] noted that larger pores between and within particles resulting from agglomerate formation facilitate capillary penetration. This increase in capillarity enhances the sinking rate and dispersibility of the powder sample. The numerical simulation with maltodextrin DE 19 performed in the study demonstrated that small particles in particular have more difficulty absorbing and dispersing water, leading to a prolonged reconstitution time and possibly the formation of lumps [5]. Within the investigated design space, lump formation was primarily governed by formulation composition. Compared with the carbohydrate-rich reference formulation PP3, both the protein-enriched (PP2) and fiber-enriched (PP1) formulations exhibited increased lump formation at comparable agglomerate diameters. These differences likely reflect the combined influence of the overall formulation composition, including the relative proportions of carbohydrates, plant proteins and dietary fibers, which simultaneously affect hydration behavior, moisture sorption and particle surface properties. Consequently, the observed behavior should not be attributed to the isolated effect of a single ingredient. The lower lump formation observed for PP3 is consistent with the higher carbohydrate content of this formulation and previous findings reported by Rieder et al. [6], although the contribution of the individual formulation components cannot be separated in the present study. In the study by Rieder et al. [6], an exponential correlation was ascertained between the lump quantity and the amount of soluble sugar used in the analyzed granulates. Additionally, a dependence of granule surface characteristics on residual lump quantity was described. The process parameter combinations used in the fluidized bed, which have previously demonstrated benefits for agglomerate formation (see Section 3.2), resulted in fewer lumps within PBMA compositions. However, Dupas et al. [5] reported no significant

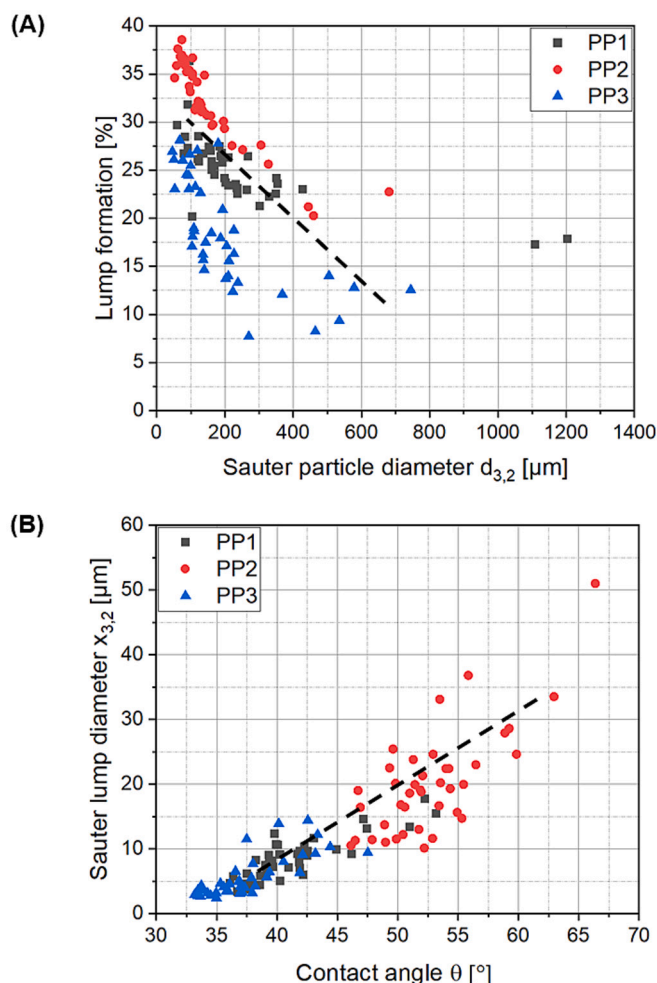


Fig. 10. (A) Lump formation in dependence of the Sauter mean diameter of the agglomerates before rehydration, (B) Sauter mean diameter of the remaining lumps after reconstitution related to the contact angle of the agglomerated powder formulations PP1, PP2 and PP3 at 20 °C; the dashed lines visualize the data trend.

influence of material moisture content on intrinsic dissolution rates. As indicated in the Appendix, product moisture has a slightly positive effect on lump characteristics due to a reduction in lump quantity and diameter.

Besides the described particle properties, the formation of lumps is also influenced by the wetting process. A poor wetting process is characterized by high contact angles and larger lump diameters, as illustrated in Fig. 10 (B). Consequently, the wetting of the bulk powder emerges as a critical factor in the reconstitution performance. The relationship between wettability and lump formation further emphasizes the importance of the overall formulation composition. The PP2 formulation exhibited the highest contact angles and largest residual lump size after reconstitution, likely due to the combined effects of plant protein, carbohydrate, and dietary fiber composition on surface properties and hydration behavior. The observed correlation therefore supports the conclusion that formulation composition governs wettability and consequently influences lump formation during reconstitution. According to Dupas et al. [5], viscous effects arising from particle composition impact the wetting process and, therefore, the complete dissolution of the powder. An increase in viscosity leads to slower solvent diffusion, which correlates with larger contact angles influenced by the complex structure of the particles. The larger the diameter of solid particles that remain undissolved after reconstitution, the broader the measured particle size distribution. The correlation can be explained by

the detected droplets of the dispersed phase. Since a high proportion of lump formation corresponds to a large diameter of the lumps, the same influences of the fluidized bed process parameters could be identified in the DoE (see Appendix). Besides differences in formulation composition, the molecular functionality of plant proteins, including their hydration behavior and interfacial properties, may also contribute to the observed differences in wettability and lump formation. Since protein structural properties as well as viscous effects were not investigated in the present study, these aspects should be addressed in future work.

4. Conclusion

In this study, a statistical Design of Experiments (DoE) approach was applied to systematically investigate, for the first time, the combined influence of formulation composition and fluidized bed process parameters on the reconstitution behavior of plant-based powders. Agglomerate growth was primarily governed by the liquid spray rate and atomizing air pressure. While the carbohydrate-rich formulation (PP3) produced the largest agglomerates, the protein-enriched formulation (PP2) exhibited reduced agglomeration and therefore required higher liquid spray rates combined with lower spray air pressures to achieve comparable particle sizes. These findings demonstrate that fluidized bed agglomeration requires formulation-specific process design rather than universal operating conditions. The moisture content of the agglomerated powders was mainly influenced by the inlet air temperature and liquid spray rate, with lower inlet air temperatures and higher spray rates resulting in increased residual moisture. Furthermore, bottom-spray processing was associated with higher product moisture contents. Powder moisture also affected wettability, as increasing water activity and the associated reduction in glass transition temperature resulted in lower contact angles. Formulation composition and inlet air temperature were identified as the dominant factors affecting wettability. Both plant proteins and dietary fibers were associated with increased contact angles and consequently reduced wettability. A strong correlation between contact angle and lump diameter suggests that wetting plays a key role during powder reconstitution. Furthermore, increasing agglomerate size consistently reduced lump formation for all investigated formulations, while the carbohydrate-rich formulation exhibited the lowest proportion of residual lumps after dissolution. Although the present study focused on understanding the relationships between formulation composition, process parameters and reconstitution behavior, product yield was not quantified. Nevertheless, no pronounced wall deposition, excessive filter losses or operational instabilities were qualitatively observed during the investigated experiments. Future studies should therefore combine product quality and reconstitution performance with quantitative yield analyses to further support industrial process optimization and scale-up.

Author note

During the preparation of this work, the authors used ChatGPT (OpenAI) to support the revision of the manuscript, including language editing, improvement of clarity and readability, and the formulation of responses to reviewers' comments. All scientific content, data analysis, interpretation of the results, and conclusions were developed and verified by the authors. The authors critically reviewed and edited all generated text and take full responsibility for the content of the published article.

CRediT authorship contribution statement

K. Kramm: Writing – review & editing, Writing – original draft, Methodology, Investigation, Conceptualization. **S. Pietsch-Braune:** Writing – review & editing, Supervision. **V. Meunier:** Writing – review & editing, Supervision, Funding acquisition. **K. Haas:** Writing – review & editing, Supervision. **S. Heinrich:** Writing – review & editing,

Supervision, Project administration, Funding acquisition.

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.

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Appendix A. Appendix

Table A.1

Complete ANOVA for the reduced two-factor interaction (2FI) model describing the Sauter mean diameter.

Source	df	F-value	p-value
Model	10	27.97	<0.0001
Liquid spray rate (A)	1	118.51	<0.0001
Spray air pressure (B)	1	109.66	<0.0001
Inlet air temperature (C)	1	8.43	0.0045
Formulation (D)	2	12.91	<0.0001
Nozzle position (E)	1	2.16	0.1449
A × D	2	3.99	0.0213
D × E	2	3.41	0.0366
Lack of fit	102	1.62	0.2831

Table A.2

Complete ANOVA for the reduced quartic model describing the moisture content.

Source	df	F-value	p-value
Model	25	12.00	<0.0001
Liquid spray rate (A)	1	77.40	<0.0001
Spray air pressure (B)	1	0.06	0.8102
Inlet air temperature (C)	1	181.67	<0.0001
Formulation (D)	2	1.00	0.3709
Nozzle position (E)	1	5.24	0.0244
A × B	1	0.22	0.6428
A × C	1	9.14	0.0032
A × D	2	1.84	0.1643
A × E	1	0.43	0.5131
B × D	2	3.80	0.0259
B × E	1	0.00	0.9850
D × E	2	0.34	0.7118
A × B × D	2	0.06	0.9422
A × B × E	1	0.00	0.9609
A × D × E	2	0.36	0.6996
B × D × E	2	1.14	0.3242
A × B × D × E	2	3.15	0.0472
Lack of fit	87	0.34	0.9880

Table A.3

Complete ANOVA for the reduced quartic model describing the contact angle.

Source	df	F-value	p-value
Model	38	10.32	<0.0001
Liquid spray rate (A)	1	7.84	0.0064
Spray air pressure (B)	1	1.21	0.2746
Inlet air temperature (C)	1	21.69	<0.0001
Formulation (D)	2	144.00	<0.0001
Nozzle position (E)	1	1.03	0.3141
A × B	1	2.67	0.1061
A × D	2	2.01	0.1406
A × E	1	0.10	0.7536
B × D	2	0.95	0.3916
B × E	1	0.75	0.3888
C × D	2	0.15	0.8640
C × E	1	2.80	0.0984
D × E	2	6.11	0.0034

(continued on next page)

Table A.3 (continued)

Source	df	F-value	p-value
B ²	1	2.74	0.1020
C ²	1	4.08	0.0467
A × B × D	2	1.06	0.3511
A × B × E	1	3.46	0.0667
A × D × E	2	0.69	0.5043
B × D × E	2	0.58	0.5648
C × D × E	2	0.06	0.9462
B ² × D	2	0.95	0.3910
C ² × D	2	0.68	0.5121
C ² × E	1	0.89	0.3484
A × B × D × E	2	3.92	0.0237
C ² × D × E	2	4.12	0.0198
Lack of fit	74	0.49	0.9274

Table A.4

Complete ANOVA for the reduced cubic model describing lump formation.

Source	df	F-value	p-value
Model	30	5.92	<0.0001
Liquid spray rate (A)	1	0.51	0.4767
Spray air pressure (B)	1	25.13	<0.0001
Inlet air temperature (C)	1	0.71	0.4032
Formulation (D)	2	2.15	0.1225
Nozzle position (E)	1	1.08	0.3017
A × C	1	0.01	0.9068
A × D	2	8.45	0.0004
A × E	1	9.15	0.0033
B × C	1	18.19	<0.0001
B × D	2	2.26	0.1109
B × E	1	3.26	0.0742
C × D	2	2.21	0.1155
C × E	1	4.32	0.0406
D × E	2	0.36	0.6971
A ²	1	3.47	0.0657
C ²	1	0.33	0.5698
A × C × D	2	6.01	0.0036
A × C × E	2	14.02	0.0003
B × D × E	2	3.27	0.0428
A ² × D	2	3.98	0.0221
C ² × D	2	7.71	0.0008
Lack of fit	82	2.64	0.1096

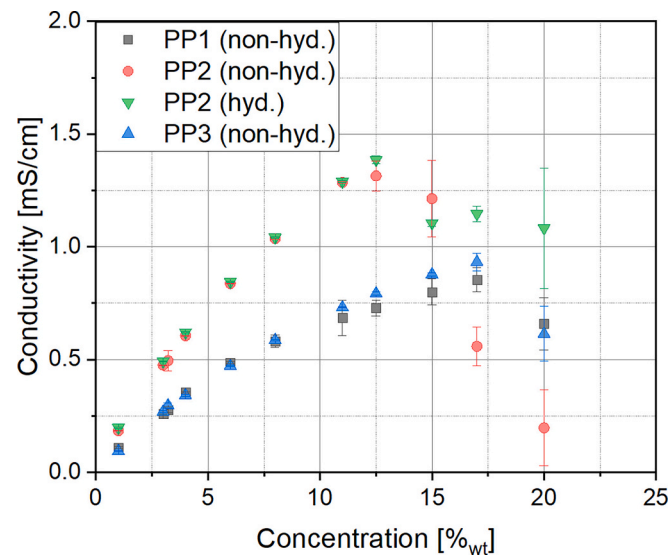
**Fig. A.1.** Conductivity values as a function of the concentration of the dissolved powder formulations PP1, PP2 and PP3 at 20 °C, considering the hydration state of the solution.

Table A.5

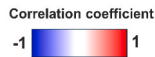
Correlation between the steady-state conductivity values and the adjusted powder concentrations for the pilot plant dried formulations PP1, PP2 and PP3 using a linear regression of the form $y = a + b \cdot x$.

Formulation	Calibration equation	R ²
PP1	($y = 0.034 + 0.070 \cdot x$)	0.99
PP2	($y = 0.094 + 0.124 \cdot x$)	0.99
PP3	($y = 0.034 + 0.070 \cdot x$)	0.99

Table A.6

Overview of the correlations generated with the response surface methodology presented using the correlation coefficient.

	$\dot{M}_{\text{liq, nozzle}}$	$\rho_{\text{air, nozzle}}$	$T_{\text{air, in}}$	Formulation	Nozzle pos.	$d_{s,3,2}$	$d_{s,50,3}$	S_s	$S_{v,s}$	SPAN _s	M_{db}	ρ_b	d_h	θ	Lump formation	$d_{i,3,2}$	$d_{i,4,3}$	SPAN _i
$\dot{M}_{\text{liq, nozzle}}$				X	X													
$\rho_{\text{air, nozzle}}$				X	X													
$T_{\text{air, in}}$				X	X													
Formulation	X	X	X		X	X	X	X	X	X	X	X	X	X	X	X	X	X
Nozzle pos.	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X	X	X
$d_{s,3,2}$				X	X													
$d_{s,50,3}$				X	X													
S_s				X	X													
$S_{v,s}$				X	X													
SPAN _s				X	X													
M_{db}				X	X													
ρ_b				X	X													
d_h				X	X													
θ				X	X													
Lump formation				X	X													
$d_{i,3,2}$				X	X													
$d_{i,4,3}$				X	X													
SPAN _i				X	X													



Data availability

Raw data were generated at the Institute of Solids Process Engineering and Particle Technology of Hamburg University of Technology, Germany and Nestlé Research and Development in Lausanne, Switzerland. Derived data supporting the findings of this study are available from the corresponding author S. Pietsch-Braune on request.

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