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Spouted bed drying efficiency of bovine hydrolyzed collagen

Eficiência da secagem de colágeno hidrolisado bovino em leito de jorro

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Summary

Bovine hydrolyzed collagen (BHC) is an important food supplement normally consumed in the form of capsules or powder in order to stimulate the synthesis of collagen, promote health and assist in esthetics. The transformation of liquid foods into powders by drying is a difficult operation due to the complex physical and chemical changes resulting from the use of high temperatures, which may result in low drying efficiency and unwanted physicochemical and nutritional characteristics in the final product. In this work, a process engineering approach was used aiming to maximize the drying efficiency and investigate the potential of using a spouted bed on the drying performance of BHC. The effects of feed mode, type of inert material and use of an adjuvant on powder production efficiency were analyzed using a 2^3 factorial experimental design. A statistical analysis showed significant effects of all the independent variables on drying performance. The maximum powder production efficiencies were achieved using polypropylene as the inert material and atomization as the feed mode. Under the optimal process conditions, up to 85% efficiency was obtained, demonstrating that the spouted bed is a technically viable equipment for drying BHC.

Keywords: *Drying; Spouted bed; Hydrolyzed collagen; Efficiency; Peptides.*

Resumo

O Colágeno hidrolisado bovino (CHB) é um importante suplemento alimentar normalmente consumido na forma de cápsulas ou pós com o intuito de estimular a síntese de colágeno corpórea, promover a saúde e auxiliar na estética. A transformação de alimentos líquidos em pós através da secagem é uma operação difícil devido às complexas transformações físico-químicas decorrentes da elevada temperatura, o que pode resultar em baixa eficiência de secagem e características físico-químicas e nutricionais indesejadas no produto final. Neste trabalho, uma abordagem de engenharia de processos foi utilizada com o objetivo de maximizar a eficiência de secagem e investigar o potencial do secador de leito de jorro durante a etapa de secagem de CHB. Os efeitos do método de alimentação de pasta, tipo de inerte, bem como o uso de adjuvante foram analisados em relação à eficiência de obtenção de pó, a partir de um planejamento experimental fatorial 2^3. A análise estatística dos resultados revelou que todas as variáveis independentes apresentam efeito significativo sobre a eficiência de secagem. As máximas eficiências de produção de pó foram alcançadas com a utilização de polipropileno como material inerte e atomização como modo de alimentação. Nas condições ótimas de processo, foi obtida eficiência de até 85%, demonstrando que o leito de jorro é viável tecnicamente para secagem de CHB.

Palavras-chave: *Secagem; Leito de jorro; Colágeno hidrolisado; Eficiência; Peptídeos.*

1 Introduction

Collagen is a fibrous protein widely found in body tissues and comprises more than a quarter of the human protein mass. The function of collagen is related to the flexibility of the epithelial and connective tissues, as well as the strength of the bone tissue (RODWELL; KENNELLY, 2003).

Bovine hydrolyzed collagen (BHC) is a product of the enzymatic hydrolysis of collagen obtained from cattle skin and bones. It consists of a set of amino acids and bioactive peptides easily absorbed and distributed to the blood and other body tissues (WALRAND et al., 2008). BHC has received significant attention from pharmaceutical and food industries as it is generally recognized as a safe food by regulatory agencies. Also, clinical investigations indicated that orally administered collagen hydrolysate had a positive effect on cartilage regeneration, suggesting a potential therapeutic use of BHC in the treatment of some chronic diseases, such as osteoporosis and osteoarthritis (MOSKOWITZ, 2000).

The increase in BHC usage as a food supplement in capsule or powder form has promoted an increase in the collagen quality standards. The physicochemical and functional properties of the peptides are influenced by the processing conditions (ZENG et al., 2013). An improvement in the process steps and the development of process engineering are good alternatives to achieve the quality product requirements.

Drying is one of the most important steps during the production of collagen powder. At this stage, degradation by overheating must be avoided and high powder production efficiency is desirable.

The spray dryer has been used to carry out the drying process of collagen peptides (ZENG et al., 2013), since it is considered to be a good method to transform semi liquid foods into powders (BHANDARI et al., 1997). However, many disadvantages limit the employment of a spray dryer, such as the loss of aromatic compounds, thermal degradation, low efficiency and high energy demand (GREENWALD; KING, 1981).

On the other hand, the spouted bed could be an alternative way to dry hydrolyzed collagen. The spouted bed consists of a drying chamber, usually cylindrical, with a conical base. Under a stable spouting regime, the bottom inlet air promotes a cyclic movement of the inert particles, and three different internal regions can be identified: spout, fountain and annulus (MATHUR; EPSTEIN, 1974). The main advantages of the spouted bed dryer are excellent heat and mass transfer rates due to gas-solid contact, good particle movement and the possibility of drying heat sensitive materials, such as food pastes and slurries (FREIRE; SILVEIRA, 2009). This equipment has been successfully used to produce high quality food powders by drying pastes (ROCHA; TARANTO, 2009; FREIRE; SILVEIRA, 2009; MASSARANI et al., 1992; MEDEIROS et al., 2002; SOUZA, 2009; BRAGA; ROCHA, 2013).

The mechanism for drying pastes in a spouted bed is well-established. The process consists of filling a column with inert particles and under a stable spouting operation, feed the paste into the bed by an atomization or dripping mode. The inert surface is coated by the paste, forming a layer that gradually dries due to inert heat conduction and hot inlet air convection. The dried film is friable and broken up by the mechanical action of particle collisions and release from the inert surface, forming a powder that is entrained by pneumatic transport (FREIRE et al., 2009; MATHUR; EPSTEIN, 1974).

Despite the importance of the drying step, studies of the drying of hydrolyzed collagen are still scarce and the use of the spouted bed dryer totally unknown. The results reported in the scientific literature about collagen are all related to the characterization (SARBON et al., 2013) and functional properties of the hydrolysate (LI et al., 2009; SHON et al., 2011; GIMÉNEZ et al., 2009; WASSWA et al., 2007; KLOMPONG et al., 2007; HMIDET et al., 2011).

The lack of knowledge and information concerning the technical parameters about the drying of hydrolyzed collagen in a spouted bed, demonstrate the need for scientific and technological studies as a foundation for this procedure, and open new alternatives to the conventional drying process.

In this work, a process engineering approach aimed to investigate the efficiency of producing BHC powder using a spouted bed drying process. A full 2^3 factorial design was proposed and the independent variables were: feed method (dripping and atomization); use of an adjuvant (collagen formulation with and without maltodextrin) and kind of inert material (low density polyethylene (LDPE) and polypropylene (PP)). The response analyzed was the powder production efficiency.

2 Material and methods

2.1 Materials

The pastes for drying were prepared using hydrolyzed collagen powder (Gelita, Brazil) and distilled water and the procedure consisted of two steps. Firstly, BHC powder was manually dissolved in water (50 °C) with continuous stirring and secondly, the solution formed was ground in a colloidal mill (Osmec, Brazil) at 3470 rpm for 30 s to avoid cluster formation.

Maltodextrin (MoR-ReX 1910, Brazil) with 10 dextrose equivalents was used as the paste adjuvant. Two different pastes were prepared for the drying experiments: composition A was composed of 50% BHC and 50% water (w/w); and composition B was composed of 40% BHC, 50% water and 10% maltodextrin (w/w).

2.2 Characterization of the inert materials

The inert materials (LDPE and PP) were characterized for their granulometry, true density and contact angle. The granulometry of the inert particles was determined by the traditional sieving method, using the definition of Sauter to evaluate the mean diameter. Standard test sieves (Tyler 5, 6, 7 and 8) and a sieve shaker (Produtest, Brazil) were used. The true density was obtained using a helium pycnometer (Accupyc 1330, Micromeritics, USA), and the contact angles of the pastes for drying and the inert materials (wettability) were measured using a goniometer (Tantec angle contact meter, USA).

2.3 Experimental system

The experimental system was a cone-cylindrical spouted bed manufactured in acrylic Plexiglas®, as illustrated in Figure 1. The conical base had an included angle (α) of 60° and inlet air diameter (D_i) of 0.03 m. The internal diameter (D_v) of the cylindrical vessel was 0.2 m and the height (h) 0.5 m.

The components of the experimental system were: (1) blower, (2) cooler, (3) line pressure tap (static), (4) orifice plate pressure tap (differential), (5) silica gel fixed bed, (6) inlet temperature and humidity sensor, (7) electrical resistance, (8) pressure tap of the bed (differential), (9) temperature sensor, (10) temperature controller, (11) outlet temperature and humidity sensor, (12) spray/dripper nozzle, (13) spouted bed, (14) Lapple cyclone, (15) magnetic stirrer, (16) paste reservoir, (17) peristaltic pump, (18) compressed air supply line, (19) frequency inverter, (20) and (21) differential pressure transducers, (22) absolute pressure transducer, (23) data acquisition system, (24) computer. Labwiew™ 8.6 was used as the interface between the process and hardware.

Two kinds of inert beads were tested in the drying experiments: low density polyethylene (LDPE) and polypropylene (PP). Both PP and LDPE showed the same Sauter mean diameter (3.29 mm ± 0.01 mm) and irregular shape (pellets with sphericity values of 0.75 ± 0.07 and 0.79 ± 0.05 for PP and LDPE, respectively).

2.4 Fluid-dynamic tests

Fluid-dynamic tests were carried out using the two inert materials without paste feeding to define the minimum spouting velocity, operational air velocity, inert mass and atomization pressure.

The minimum spouting velocity was determined from the fluid-dynamic curves according to the method developed by Mathur and Epstein (1974). The criterion used to choose the operational air velocity, inert mass

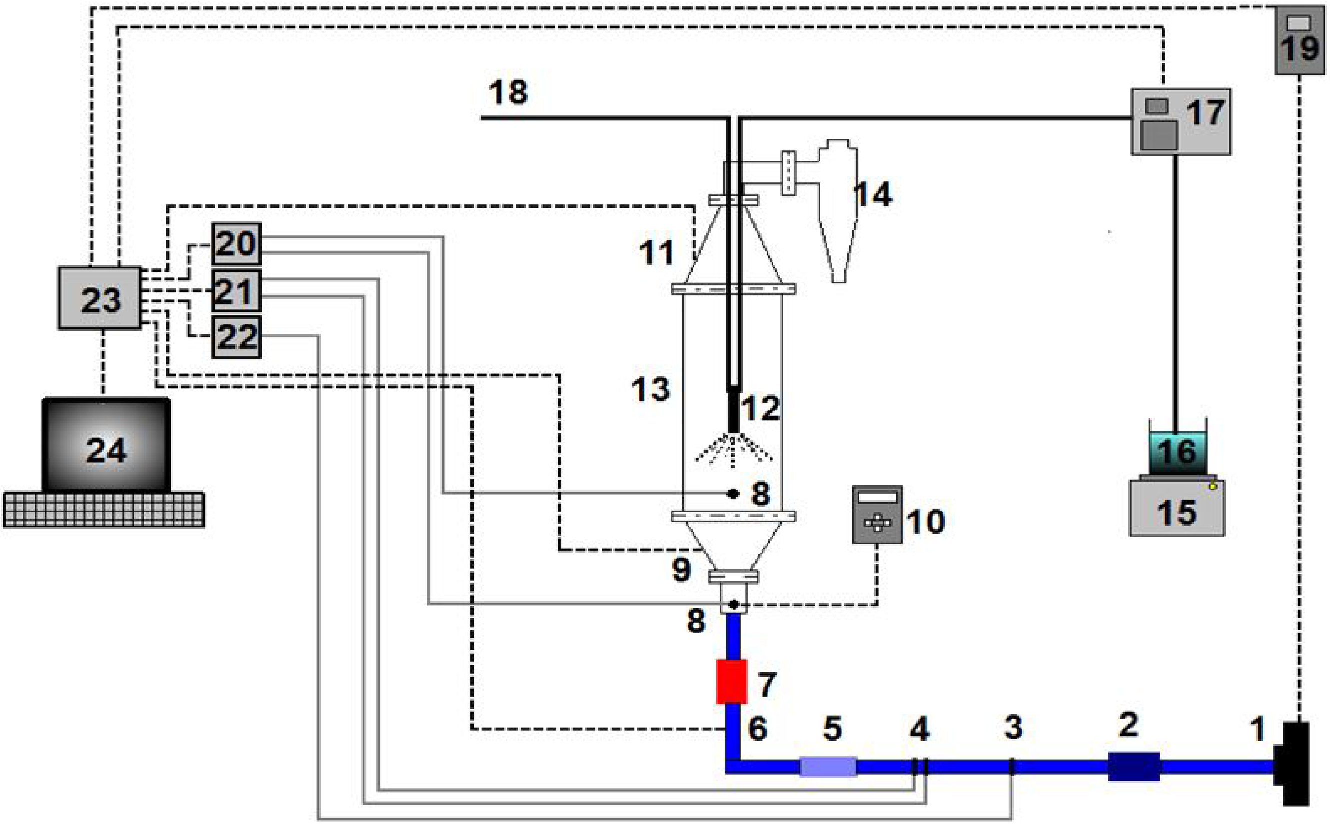

Figure 1. Cone-cylindrical spouted bed and experimental system.

and atomization pressure was based on both the dynamic curves and visual observations of stable spouting. The tests were carried out in duplicate.

2.5 Experimental procedure

The experimental system was turned on to heat the equipment before starting the drying experiments. The paste feeding procedure started after stabilization of the inlet and outlet air temperatures. During the experiments, the feed flow rate was maintained constant (4 mL/min) and the inlet air velocity varied in the range from 1.5 U_{ms} (0.55 m/s) to 1.55 U_{ms} (0.57 m/s) to maintain good movement of the inert material and ensure stable spouting dynamics.

The peristaltic pump was turned off after completing paste feeding (0.3 kg) and the drying process continued for another 20 minutes to ensure powder release and elutriation. Each experimental drying run lasted about one hour and forty minutes.

The independent process variables analyzed were: feed method (dripping and atomization), adjuvant addition (with and without maltodextrin) and kind of inert particle (LDPE and PP). The powder production performance was the response analyzed.

The powder production performance (ξ) was estimated from the ratio of dry powder mass recovered at the end of the process per total dry mass added to the bed, Equation 1 (ROCHA et al., 2009).

$$\xi = \frac{Mp\ C_{DS}}{W\ Cs\ t} \tag{1}$$

where: M_p = mass of powder recovered; C_{DS} = mass fraction of dry solid in the powder; w = mass flow rate; C_s = mass fraction of dry solid in the paste; t = feed time.

The powder moisture content was measured using gravimetric method n° 12/IV of the Instituto Adolfo Lutz (IAL, 2008) in an oven at atmospheric pressure and 105 °C and drying to constant weight.

3 Results

3.1 Operational process conditions

Preliminary tests were carried out in triplicate to define the fixed operational conditions for the mass of inert material, mass of paste added to the bed, air atomizing pressure, feed flow rate and inlet air temperature. Although important in the drying process, these variables were not included in the experimental design of this work, since it was not possible to vary them in a wide range without the spout collapsing. Visual observations of stable spouting and fountain conditions, as well as accumulation of powder in the cyclone were used as the criterion to select the fixed operational conditions. The results are shown in Table 1.

The bed height was tested for two inert materials, PP and LDPE. The best fluid dynamics and stable spouting for both materials was observed at 0.13 m ± 0.01 m of bed height, corresponding to an inert material load of 1.0 kg.

Low masses of inert material resulted in diluted spouting, whereas high loads resulted in unstable spouting. Instabilities in the fountain were also observed at atomization pressures higher than 69 kPa.

The minimum spouting velocity was determined from the fluid dynamics curves (T = 80 °C, M = 1.0 kg), obtained in duplicate and presented in Figures 2 and 3 (mean values).

The patterns of Figures 2 and 3 are in agreement with that reported by Mathur and Epstein (1974) for spouted beds. The minimum spouting velocity (U_{ms}) and maximum pressure drop (ΔP_m) for both inert materials were determined by the fluid dynamics graphic method, and are shown in Table 2.

The minimum spouting air velocity was the same (0.37 m/s) for both inert materials, however, the maximum pressure drop showed differences. The different maximum pressure drops may be explained by head losses generated by roughness, different heat transfer, dilation or other

Table 1. Fixed operational conditions in the spouted bed dryer.

Variable	Value
Mass of inert material	1.0 kg
Mass of paste	0.3 kg
Atomization pressure	69 kPa
Feed flow rate	4 mL/min
Air temperature	80 °C

Table 2. Fluid dynamics parameters for the spouted beds (LDPE and PP).

Variable	Value
Minimum spouting air velocity	0.37 m/s
Operational air velocity	0.55 m/s
Maximum pressure drop (LDPE)	800 Pa
Maximum pressure drop (PP)	1550 Pa

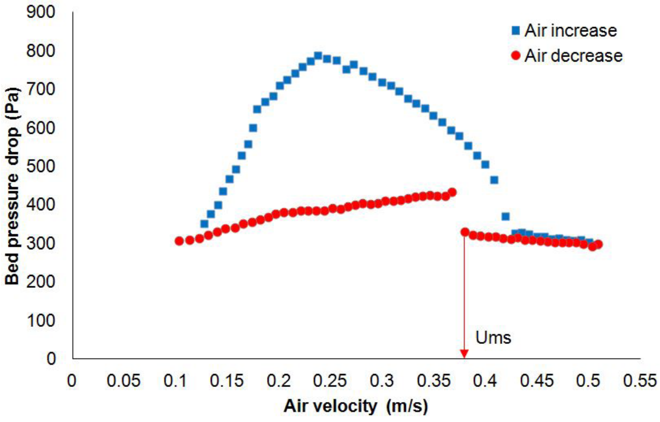

Figure 2. Fluid dynamics curve for LDPE.

inert material characteristics not measured in this work. The same minimum spouting velocity can be attributed to the same mean diameter, 3.29 mm ± 0.01 mm, similar sphericity, 0.75 ± 0.07 and 0.79 ± 0.05 and absolute density, 907.8 kg/m^3 and 921.8 kg/m^3, of PP and LDPE, respectively.

The operational air velocity chosen for the drying runs was defined as 1.5 U_{ms}, however, but it was varied a little, from 1.5 U_{ms} (0.55 m/s) to 1.55 U_{ms} (0.57 m/s), to ensure maintenance of the fountain stability in the wet bed during paste feeding.

3.2 Paste characterization

The moisture contents (w.b.) of the drying pastes A and B were equal to 54.00% ± 0.06%, and 54.51% ± 0.03%, respectively. Similar results for the moisture contents of pastes A and B can be attributed to similar moisture contents of BHC and maltodextrin.

The contact angles between the paste droplets and a smooth surface of PP and LDPE were also analyzed. Solution A resulted in contact angles of 88.55° ± 0.82° and 82.85° ± 0.82° with PP and LDPE, respectively, whereas solution B presented contact angles of 86.10° ± 2,0° with PP and 85.70° ± 0.99° with LDPE.

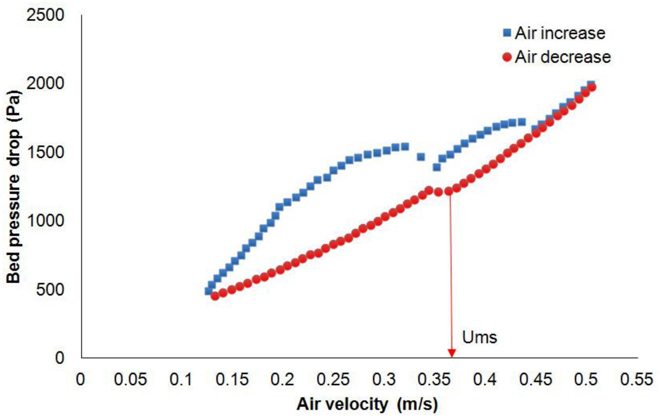

Figure 3. Fluid dynamics curve for PP.

Contact angles higher than 70° for solutions A and B with PP and LDPE indicate the great potential of these polymers as efficient inert materials in a spouted bed drying process. According to Rocha et al. (2009), contact angles higher than 70° are favorable to drying processes, whereas contact angles lower than 70° indicate that the solid may be coated by the liquid. In conclusion, the analyses of the contact angles showed that both polymers, PP and LDPE, could act as efficient inert materials.

3.3 Drying experiments

A 2^3 experimental design was proposed and carried out to analyze the process performance. The experiments were done in duplicate (total of 16 runs) and the other process variables were kept constant. Table 3 shows the levels of the independent variables and the average responses of the drying tests.

The highest powder production efficiency (85.98%) was achieved for run number 6, followed by run number 2 (83.23%). On the other hand, the lowest efficiency was observed for run number 3.

High powder production efficiencies obtained with both inert materials confirmed that both PP and LDPE were good polymeric resins for using in drying pastes in spouted beds, in agreement with the high contact angles, greater than 70°.

The results in Table 3 show that the process performance was better when using PP as the inert material than when using LDPE, and the powder production efficiency was visibly dependent on the feed mode in the case of LDPE, as can be seen from the low powder production efficiencies obtained for runs 1 and 3, 38.67% and 30.03%, respectively.

The low powder production efficiency observed in the experiments using dripping as the feed mode can be

Table 3. Variables, levels and responses of the experimental design.

Factor	-	+
(1) Feed mode	Dripping	Atomization
(2) Maltodextrin addition	No	Yes
(3) Type of inert material	LDPE	PP

Run	Feed mode		Maltodextrin		Inert material		Efficiency (%)	SD (%)
1	(-1)	dripping	(-1)	No	(-1)	LDPE	38.67	1.57
2	(+1)	atomization	(-1)	No	(-1)	LDPE	83.23	2.06
3	(-1)	dripping	(+1)	Yes	(-1)	LDPE	30.03	3.14
4	(+1)	atomization	(+1)	Yes	(-1)	LDPE	78.06	0.98
5	(-1)	dripping	(-1)	No	(+1)	PP	62.61	6.17
6	(+1)	atomization	(-1)	No	(+1)	PP	85.98	1.57
7	(-1)	dripping	(+1)	Yes	(+1)	PP	54.24	5.28
8	(+1)	atomization	(+1)	Yes	(+1)	PP	79.51	0.87

attributed to the heterogeneous paste distribution inside the bed. With this feed mode, the paste is continuously dripped onto the fountain by a drip nozzle, and could cause paste accumulation in the annulus-wall, with a consequent deposition of powder on the spouted bed wall. This effect was confirmed by visual observations, mostly for the pastes containing 10% of maltodextrin.

On the other hand, for the atomization feed mode the collagen paste is sprayed by a double fluid nozzle in a dispersive form onto the bed of inert material, and homogeneously onto the particle surface. The small droplets resulting from this feed mode produce a thin and weak layer over the inert material surface, collaborating with fast drying and breakage of the film, easy release and elutriation of the powder.

In contrast, Braga and Rocha (2013) reported different results for a similar process. The authors analyzed the drying of a blend of milk and black mulberry in a spouted bed and found low powder production efficiency with inert PP particles and the atomization method. Good results were observed by the authors using polystyrene (PS) as the inert material and dripping as the feeding method, resulting in a powder production efficiency higher than 60% and low moisture content of the product. The differences between the results of this work and those reported by Braga and Rocha (2013) can be attributed to different physicochemical characteristics of the drying pastes.

Medeiros et al. (2002) studied the influence of the chemical composition on the performance of pulp fruit drying in a spouted bed and noticed interferences of the paste composition on the dynamic regime stability and powder production efficiency. The absence of lipids in the collagen composition may result in limited motion of the particles and difficult drying under a dripping mode of feeding.

Figure 4 shows the results of all the experiments in comparison with a satisfactory efficiency line (dashed line, 50%) and good efficiency line (dotted line, 75%) for spouted bed dryers.

As shown by Figure 4, in at least three quarters of the experiments the powder production efficiency was greater than 50%, which is considered satisfactory performance, and good dryer performance (above 75%) could be seen in half the experiments.

The tests were done in duplicate to estimate the experimental error and evaluate the statistical significance of the effects. Table 4 shows the statistical effects of feed mode (1), adjuvant addition (2), inert material (3), and the interactions between them, on powder production efficiency, for a confidence level of 95%.

According to Table 4, the biggest effect (35.31) was attributed to the feed mode, and the second biggest effect to the type of inert material (13.09). In fact, the results show an average increase of 35% and 13% in powder production efficiency with the best feed mode and inert material, respectively. It can be seen that the interaction effect of these two variables (-10.99) was also significant at p = 0.05.

The effect of adding maltodextrin was negative and less significant on the efficiency (-7.16). This fact reflects the non-significant interaction effect of this variable with the others.

These results were reinforced by a Pareto chart for the effects of feed mode (1), maltodextrin (2) and type of inert material (3) on process efficiency, considering a confidence interval of 95%, as shown by Figure 5.

The Pareto chart is further statistical evidence of the significant influence of the three variables analyzed on the

Table 4. Single and interaction effects of the process variables.

Variable	Effect	SD	P
(1) Feed mode	35.31	0.4	0.007067
(2) Maltodextrin	-7.16	0.4	0.034808
(3) Type of inert material	13.09	0.4	0.019057
Interaction 1 and 2	1.34	0.4	0.180716
Interaction 1 and 3	-10.99	0.4	0.022699
Interaction 2 and 3	-0.2599	0.4	0.627241

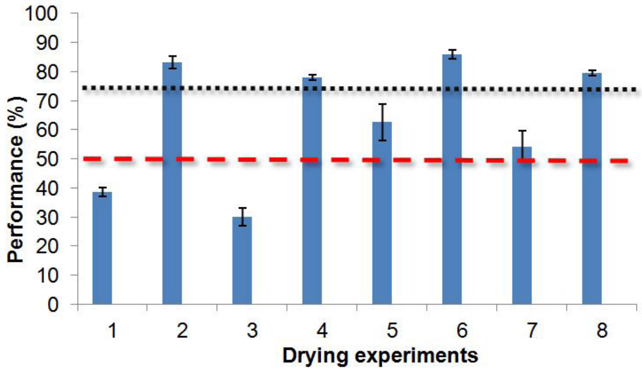

Figure 4. Comparison of the powder production efficiencies.

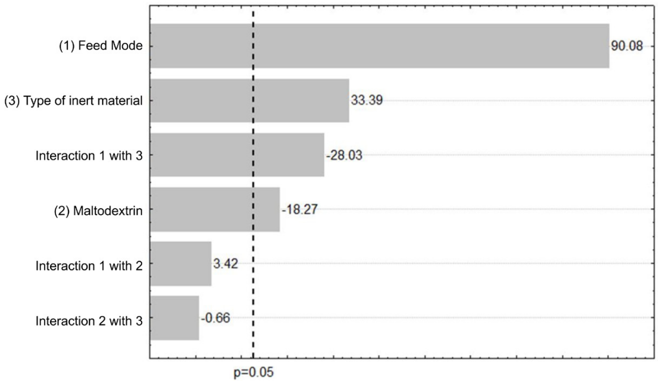

Figure 5. Pareto chart for the effects of feed mode, type of of inert material and adjuvant.

powder production efficiency. As shown by Figure 5, the interaction effects of maltodextrin with the other variables were not statistically significant. The vertical dashed line indicates the significance level of 95% ($p = 0.05$).

The results of high powder production efficiency for some drying conditions confirmed that the spouted bed could be a good way for drying BHC pastes. In addition, the good yield obtained is evidence that BHC could be used as an adjuvant to improve the powder production efficiency when drying food pastes.

4 Conclusions

In this study, the results demonstrated that the inert material, feed method and use of maltodextrin as an adjuvant were significant process variables and influenced the drying performance when drying BHC paste in a spouted bed. The effect of adding an adjuvant was less pronounced and negative. The inert material and feed method presented high and significant influences on the drying performance of BHC, and a correct combination of both improved the yield by up to 85%, showing great potential for the drying of hydrolyzed collagen in a spouted bed. In addition, the high powder production efficiency achieved suggests the use of BHC as an adjuvant to improve the process efficiency in the drying of food pastes.

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