

**Increasing energy efficiency in microencapsulation of soybean oil
by spray drying****Aumento da eficiência energética na microencapsulação de óleo
de soja por spray drying**

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ABSTRACT

The aim of this study was to evaluate the influence of inlet air temperature of spray drying on microparticles properties and heat loss on drying chamber in sense to select the conditions that promote the preservation of functional compounds and improve energy efficiency. The energy efficiency of the process was evaluated varying inlet air temperature (T) from 110 to 150 °C and airflow (AF) from 20 to 40kg/h, being the energy efficiency, the response variable. The

microspheres presented a continuous surface and no cracking, which hinders the diffusion of air and reduces the probability of triglycerides oxidation in the nucleus. The heat loss curves were plotted considering the principles of mass and energy conservation applied to both input and output currents. Further, the thermodynamic parameters were continuously monitored during the spray dryer. So, the selected operational conditions ($T = 130\text{ }^{\circ}\text{C}$, $AF = 20\text{ kg}\cdot\text{h}^{-1}$) allowed to achieve high encapsulation efficiency (95%) and saving energy compared to conventional processes using inlet air temperatures above $180\text{ }^{\circ}\text{C}$, a significant improvement to thermo-sensitive compounds.

Keywords: drying temperature; lower temperature; spray drying; encapsulation efficiency; heat loss; thermo-sensitive compounds

RESUMO

O objetivo deste estudo foi avaliar a influência da temperatura do ar de entrada da secagem por aspersão nas propriedades das micropartículas e a perda de calor na câmara de secagem no sentido de selecionar as condições que promovem a preservação de compostos funcionais e melhoram a eficiência energética. A eficiência energética do processo foi avaliada variando a temperatura do ar de entrada (T) de 110 a $150\text{ }^{\circ}\text{C}$ e o fluxo de ar (AF) de 20 a $40\text{ kg} / \text{h}$, sendo a eficiência energética, a variável resposta. As microesferas apresentaram uma superfície contínua e sem rachaduras, o que dificulta a difusão do ar e reduz a probabilidade de oxidação de triglicerídeos no núcleo. As curvas de perda de calor foram plotadas considerando os princípios de conservação de massa e energia aplicados às correntes de entrada e saída. Além disso, os parâmetros termodinâmicos foram continuamente monitorizados durante o secador por aspersão. Assim, as condições operacionais selecionadas ($T = 130\text{ }^{\circ}\text{C}$, $AF = 20\text{ kg}\cdot\text{h}^{-1}$) permitiram alcançar alta eficiência de encapsulação (95%) e economia de energia em comparação aos processos convencionais usando temperaturas do ar de entrada acima de $180\text{ }^{\circ}\text{C}$, uma melhoria significativa compostos termo-sensíveis.

Palavras-chave: temperatura de secagem; temperatura mais baixa; secagem por pulverização; eficiência de encapsulamento; perda de calor; compostos termo sensíveis

1 INTRODUCTION

Spray drying is not a novel technique and it has been successfully used in food industry for more than a century (O'Sullivan *et al.* 2019, Percy 1872), and the most used parameter variations were temperatures from 160 to $200\text{ }^{\circ}\text{C}$. However, there are situations where the most recommended configuration cause unwanted modifications in the product, as in the functional compounds, focus of our studies. Then it is necessary to return to the bases of the technique to adjust the parameters to each particular case.

Though widely used, the spray drying process consumes a significant amount of energy due to the evaporation of water in the material. It is a challenge in the food industry to minimize this high consume, making the process more cost-effective and its products, more competitive.

Spray drying has been widely used to protect highly unsaturated oils from oxidative rancidity (Nickerson and Chang 2018, Martínez *et al.* 2015, Bae and Lee 2008). Unfortunately, even with the short residence time (1-3 seconds), thermo-sensitive compounds (e.g. functional compounds and polyunsaturated oils) can be harmed at such temperatures. If a spray drying process can be accomplished at lower temperatures without compromising the drying efficiency, it can be applied to thermo-sensitive material with lower damaging, decreasing the energy consumption of the process, combining quality and a minor cost, a factor of relevance in industrial process.

To microencapsulate lipids, ingredients that provides protection and stabilizes the microparticles are necessary. In this case, an emulsion is previously carried out with oil, encapsulating ingredient and water. High molecular weight carbohydrates are the most common and cheapest encapsulating material, although some emulsions blending carbohydrates and protein have been showing promising results (Nickerson and Chang 2018).

During the microencapsulation of poly-unsaturated lipids by spray drying, the control of process conditions is fundamental to obtain a product with the desired physical and functional characteristics. Among the operational parameters, the input currents as drying temperature, airflow, and feed flow stand out as the main operational control variables. The effect of thermodynamics properties of inlet air in the characteristics of the microencapsulated particles has been underexplored in scientific literature. Few authors (Ng *et al.* 2013, Tonon *et al.* 2008) have investigated and compared the effect of lower temperatures (138 - 202 °C) in vegetable oil microencapsulation.

Thus, the objective of this study was to evaluate the influence of inlet air temperature of spray drying on microparticles properties and the heat loss on the drying chamber to select the conditions that promote the preservation of functional compounds in polyunsaturated oils and, simultaneously, to improve energy consumption during its microencapsulation by spray drying.

2 MATERIAL AND METHODS

2.1 MATERIALS

An oil-in-water emulsion was prepared using 7.5% pressed soybean oil (Organic[®]), 7.5% maltodextrin (DE5, GLOBE[®] 1805, Corn Products Brazil) and 22.5% modified starch (Capsul[®] AKY-800, National Starch).

2.2 DENSITY

The average density of the emulsion was estimated by picnometry. This assay was replicated five times.

2.3 PROCESSING

The processing was conducted in a lab scale spray dryer LabPlant SD06. The process was evaluated with a combination of five different inlet air temperatures (110, 120, 130, 140 and 150 °C) and five different inlet airflows (20; 25; 30; 35 and 40 kg/h), totalling 25 experiments.

The emulsion was pumped into the drying chamber at a controlled rate of 485 mL/h, and sprayed by a 0.7 mm diameter nozzle. An exact process time of 10 min was defined for each experimental condition. The microencapsulated powder was collected in amber glass flasks in dark cabinet at room temperature. The simplified scheme is illustrated in Figure 1.

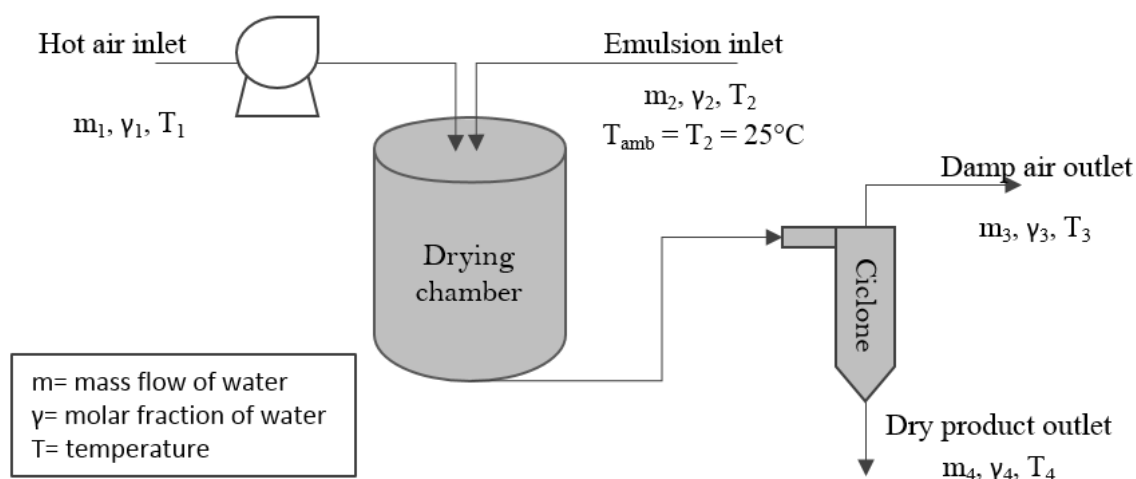


Figure 1. Simplified spray dryer operation scheme.

2.4 HEAT LOSS EVALUATION

The classical equations of mass and energy conservation were applied to analyse the drying process. The drying chamber was considered as control volume and, in order to perform the energy balance, some hypotheses were adopted: i) air was considered an ideal gas and ii) the thermal properties of the emulsion were similar to the properties of water. All thermodynamic properties were taken from specific tables in the Chemical Engineer's Handbook (Perry and Chilton, 1980). The molar fraction of water vapor in the inlet air (γ_1) was obtained from thermodynamic data recorded in ambient air, using psychometric charts (Luikov, 1982).

The global energy balance equation expresses the enthalpy conservation of all the incoming and outgoing currents:

$$\dot{m}_1 H_1 + \dot{m}_2 H_2 + \dot{Q}_p = \dot{m}_3 H_3 + \dot{m}_4 H_4 \quad (1)$$

Newton's semi-empirical cooling equation:

$$q_s'' = h * (T_s - T_w) \quad (2)$$

The energy consumed to boil the water from 25 °C (ambient temperature):

$$Q_{Ev} = m. \lambda + m. c. \Delta T \quad (3)$$

$$Q_{Exc} = Q_{Ev} + \dot{Q}_p \quad (4)$$

The heat loss in the drying chamber and the global coefficient of convective heat transport were determined by replacing the experimental data in the energy balance equation (Eq. 1) and Newton's convective transport equation (Eq. 2).

The excess of energy (Q_{Exc}) was calculated by the difference between the quantity of heat produced in each condition (\dot{Q}_p , Eq. 1) and the quantity of heat needed to evaporate the water in the emulsion in each assay (Q_{Ev} , Eq. 3).

Dry matter

Determined according to the Association of Official Analytical Chemists (AOAC 2005), method 930.15.

Encapsulation efficiency:

The encapsulation efficiency was calculated as the ratio of the encapsulated oil and the total oil content as described by Carneiro *et al.* (2013).

Morphology of the microparticles

The microphotographs of the oil powder were performed in scanning electron microscopy (SEM) TM3030 plus Hitachi, using an acceleration voltage of 15 kV with increase of 2000x.

3 RESULTS

The density of the suspension was 1.12 (± 0.02) g.mL⁻¹, and the energy calculated to water evaporation in each assay was in about 51 cal.s⁻¹. Figure 2 shows the excess of heat in every condition, where line zero is equivalent to 51 cal.s⁻¹. Except to the operational conditions at T = 110 °C/AF = 20 kg.h⁻¹ and T = 120 °C/AF = 20 kg.h⁻¹, all other conditions provided enough energy to evaporate all water in the emulsion. From this line up, as the temperature and airflow are increased, the more energy is wasted, since it is not used to withdraw the moisture from the product. This figure 2 also confirms that a greater amount of energy is lost by convection in the drying chamber when operating at higher temperatures and higher inlet flows. This phenomenon is best observed in higher temperatures.

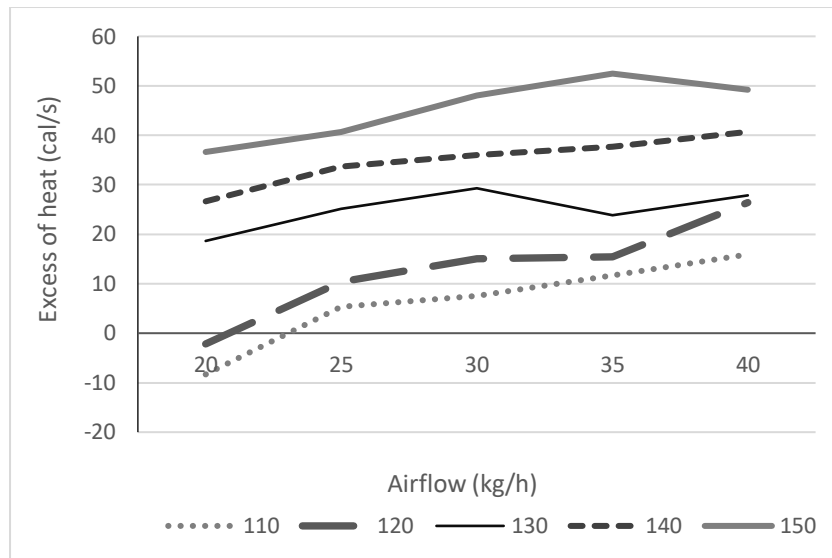


Figure 2. Excess of heat in the chamber as a function of flow and inlet air temperature.

In Figure 3 it was observed that the outlet air temperature generally increases with the inlet airflow. As the airflow increase more heat is introduced into the system, whereas only part of the energy is used for water evaporation. It is important to note that even the highest recorded outlet temperature (80 °C) is considerably lower than the inlet air temperature, contributing to lower thermal degradation of the core. The outlet temperature is the temperature reached in the microparticle surface during the water evaporation, and it corresponds to the wet bulb temperature. In general, microparticle humidity decreased with increasing temperature and increasing airflow, being higher at 110 °C (6.0 to 6.5%) and lower at 150 °C (3.4 to 5.2%; Figure 4).

Considering that the energy of the air exceeds the energy required to evaporate all the water from the emulsion, the high humidity of the microparticles can be reduced to achieve more conservative results (less than 3%) reducing the feed rate of the mixture. This decision reduces the operational capacity of the plant but increases the oxidative stability of the microparticles.

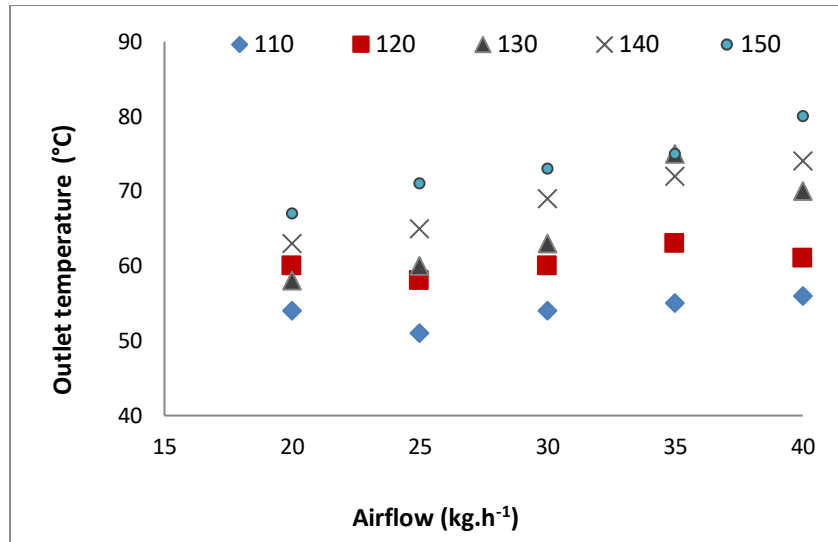


Figure 3. Air and product outlet temperature as a function of inlet airflow and temperature.

As inlet air temperature increases, it causes a larger temperature gradient (ΔT) between the air and the droplets, providing more heat energy to the system (Goula and Adamopoulos, 2008). Thus, ΔT shows a negative correlation with the powder's moisture, as can be seen in Figure 4. In Fig. 4, we can observe that from 110 to 130 °C, the moisture decreased 23%, and after 130 °C this effect was not significant. The higher ΔT also influences in microparticles' drying speed. So in higher temperatures the extra heat quickly forms a crust on particle surface that acts as a barrier to water diffusion. As result, in higher temperatures the humidity is not as low as we expected (Ng *et al.* 2013).

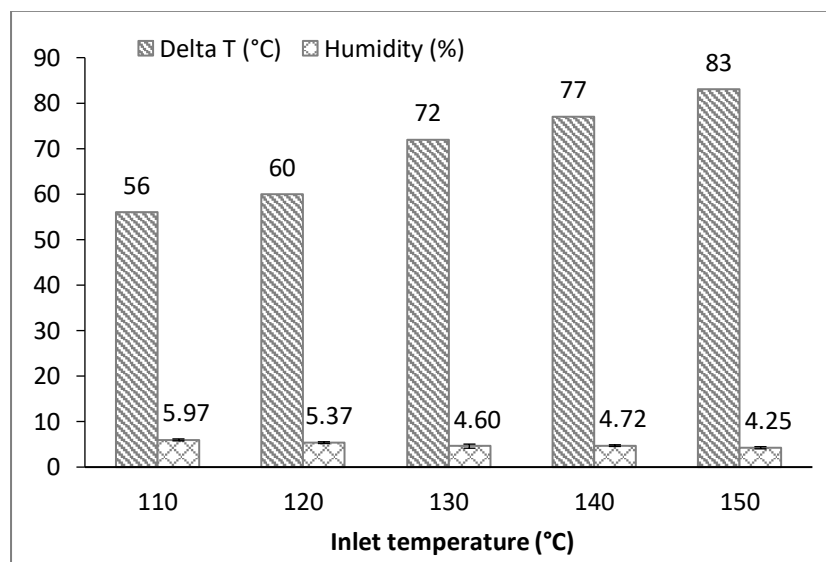


Figure 4. Temperature gradient (ΔT) in the spray dryer operation and average moisture of the powder as a function of temperature.

The data presented in Figure 5 allows evaluating the consumed energy for each assay, i.e. the amount of water evaporated per kg of hot air at the inlet of the dryer. It can be observed that the efficiency of the spray drying is more sensitive to the variation of the airflow than to the temperature. Unlike discontinuous drying processes, by increasing the inlet hot airflow, the amount of evaporated water decreases, that is, the process becomes less efficient, due to the effect of the residence time of the air. Thus, the lower the inlet airflow, the longer the time the particle takes to go through the drying chamber, allowing a greater evaporation with consequent greater transport of water in the form of vapor from the suspension to the air. These results confirm that the enthalpy of the inlet hot air provides twice as much the energy required for evaporation of the water contained in the emulsion.

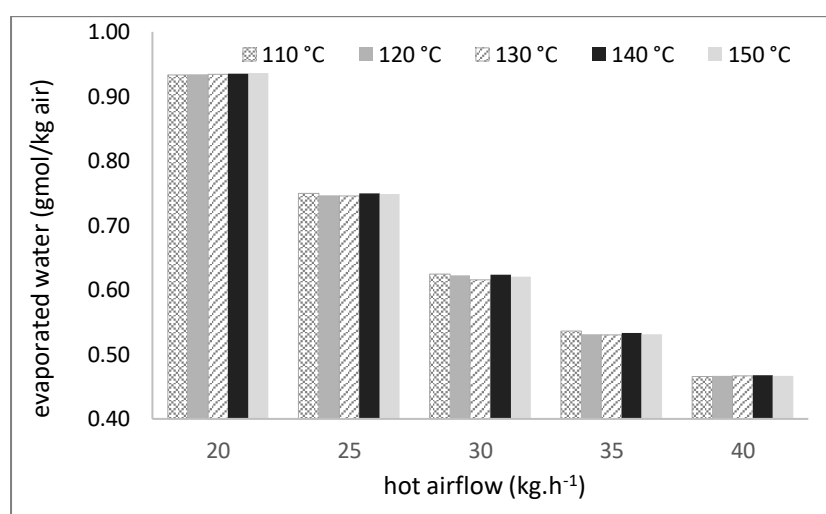


Figure 5. Process performance as a function of flow rate and air inlet temperature

4 ENCAPSULATION EFFICIENCY

Knowing the surface oil content on microparticles is very important to ensure its quality. The presence of lipids on the surface of microparticles reduces oxidative stability, wettability and flowability of the powder, important characteristics that will affect the final use of the product (Reineccius and Yan 2016).

It was observed that, regardless the temperature used, the surface of microparticles became more homogeneous as the airflow was reduced from 40 to 20 kg.h⁻¹. In this condition, the results of the encapsulation efficiency were in about 34; 64; 95; 79 and 79% for 110, 120, 130, 140 and 150 °C, respectively. Thus the highest efficiency was reached at T = 130 °C. As reported by Reineccius and Yan (2015), the lipid encapsulation efficiency does not increase linearly with the drying temperature.

Lower temperatures present lower encapsulation efficiency due to the high moisture in the system air, very close to the saturation point of the air. This inhibits water evaporation and disturbs the formation of the outer crust, allowing the oil to migrate through the particle instead of stay in the core. This also interferes in the particles sizes since, due to the high humidity in the system, particles stay wet for longer time and tend to merge and give rise to larger particles. Alternatively, higher temperatures have the crust formation too fast, as stated before, that impacts negatively the efficiency of the encapsulation. So an equilibrium point must be found to assure both process and cost efficiency.

Morphology of microparticles

To evaluate microparticles morphology, 10 experimental conditions selected between the 25 initial tests were chosen based at the condition of highest encapsulation efficiency ($T = 130\text{ }^{\circ}\text{C}$, all five airflows) and at the condition of best energy efficiency of the process ($AF = 20\text{ kg}\cdot\text{h}^{-1}$, all five temperatures).

In Figure 6, showing the constant airflow micrographs ($20\text{ kg}\cdot\text{h}^{-1}$), the first visible change is the difference in the microparticles size. The size distribution range increased slightly to $130\text{ }^{\circ}\text{C}$ (5.0 to $12.5\text{ }\mu\text{m}$) and thereafter decreased, reaching a smaller diameter range on microparticles set up at $150\text{ }^{\circ}\text{C}$ (2.5 to $10.0\text{ }\mu\text{m}$). These differences are best understood by simultaneously observing the data in Figures 6 and 7. The microparticles at $T = 150\text{ }^{\circ}\text{C}$ are smaller, possibly because the extra energy caused the fast formation of crust, with lower probability of collapse among particles. Figure 7 confirms that at higher temperatures (140 and $150\text{ }^{\circ}\text{C}$) the particles are smaller, while at lower temperature they present about the same size range.

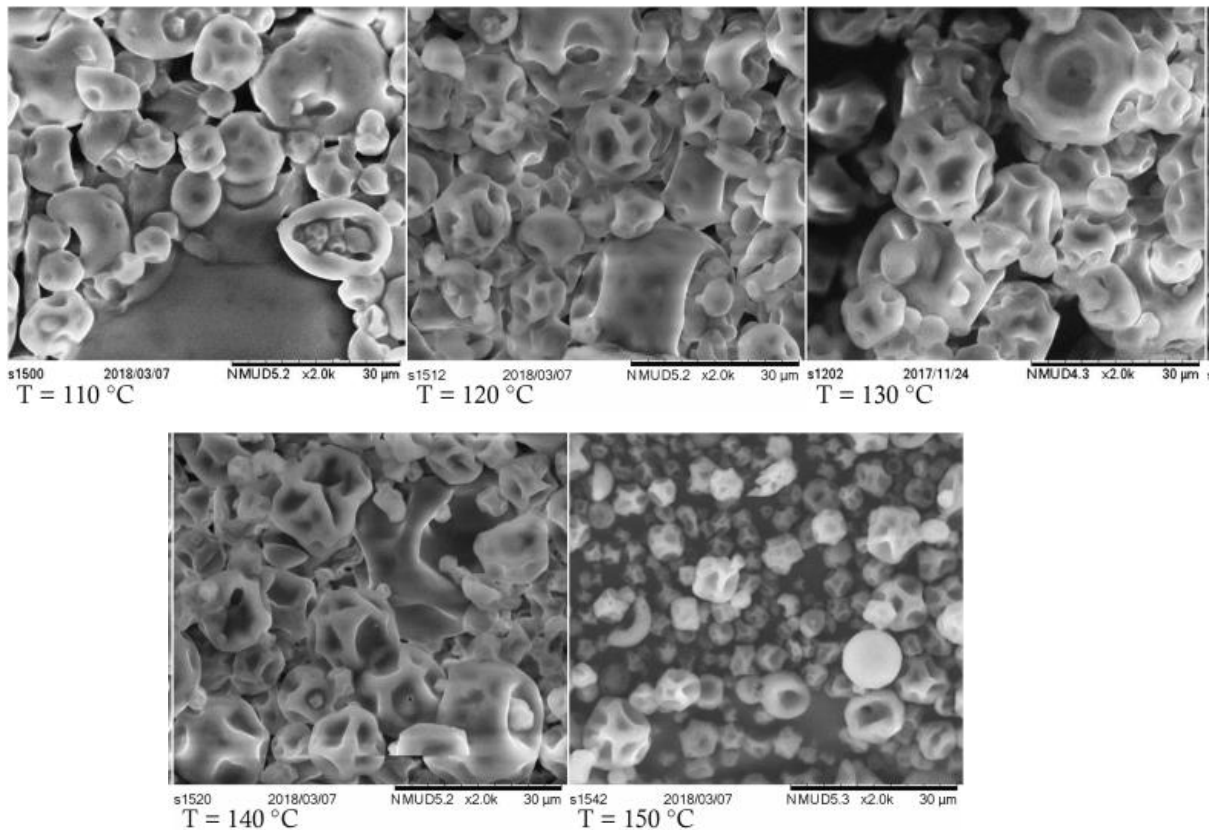


Figure 6. Micrographs of the formed microparticles. Inlet airflow = $20 \text{ kg}\cdot\text{h}^{-1}$ and temperature from 110 to 150 °C. 2.0 k magnification.

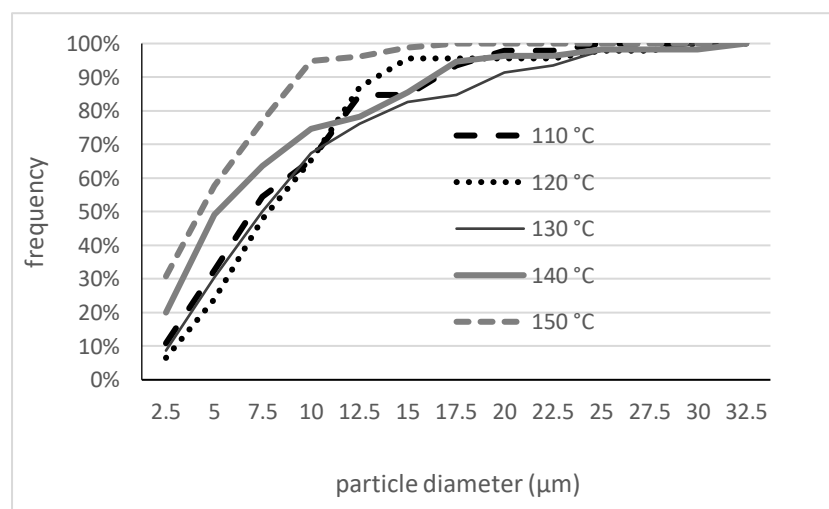


Figure 7. Cumulative frequency of particle size as a function of temperature, for inlet airflow = $20 \text{ kg}\cdot\text{h}^{-1}$.

Concerning the experiments carried out at constant temperature (130 °C) it is remarked that the increase of inlet airflow favored the heterogeneity of the size distribution (Fig. 8), contrary to the observed in the lowest airflow ($20 \text{ kg}\cdot\text{h}^{-1}$), the one that presented higher energy efficiency (Fig. 6). For higher airflows, the distributions were mostly bimodal. Because of this

heterogeneous distribution, the particle size cannot be correlated with the variation of airflow rates, as shown in Figure 9. In addition, the higher airflow caused the formation of microparticles with unconventional, deformed formats. At the last micrograph ($40 \text{ kg}\cdot\text{h}^{-1}$), microparticles with very irregular surface were observed. These atypical conformations can expose the core to ambient air, reducing the efficiency of the encapsulation, thus are not desired.

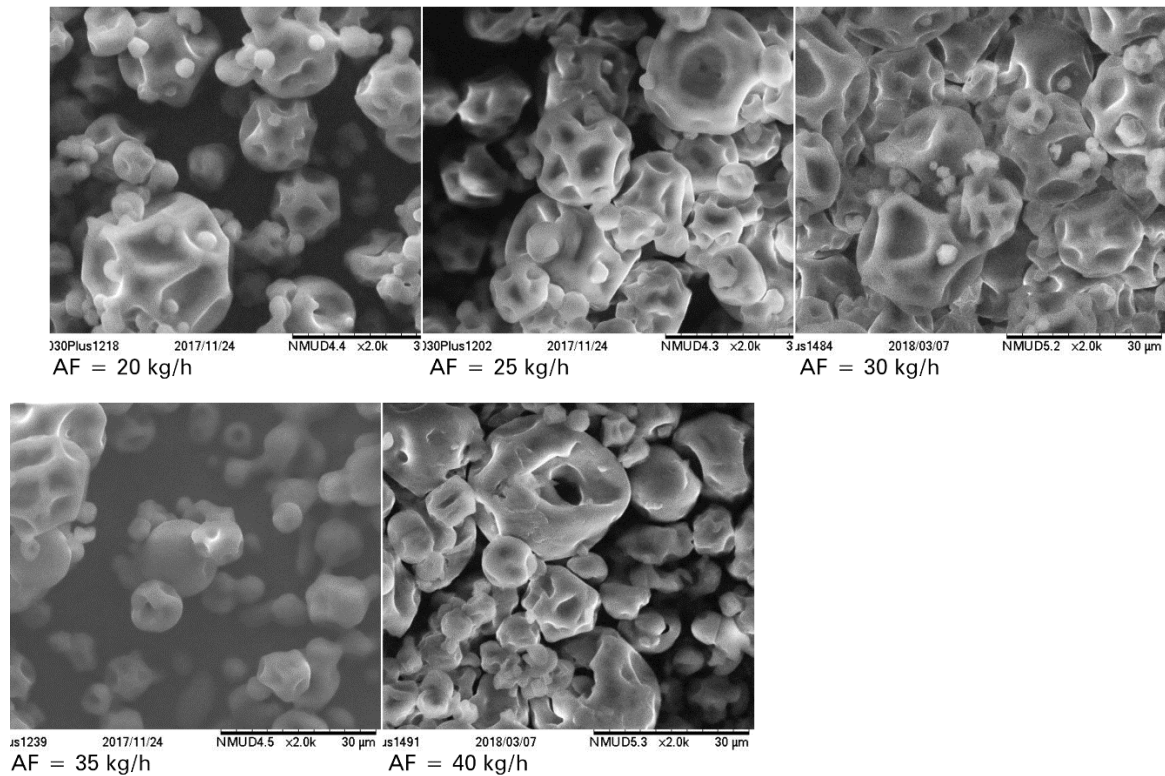


Figure 8. Micrographs of the microparticles formed at 130°C and inlet airflow (AF) from 20 to $40 \text{ kg}\cdot\text{h}^{-1}$. 2.0 k magnification.

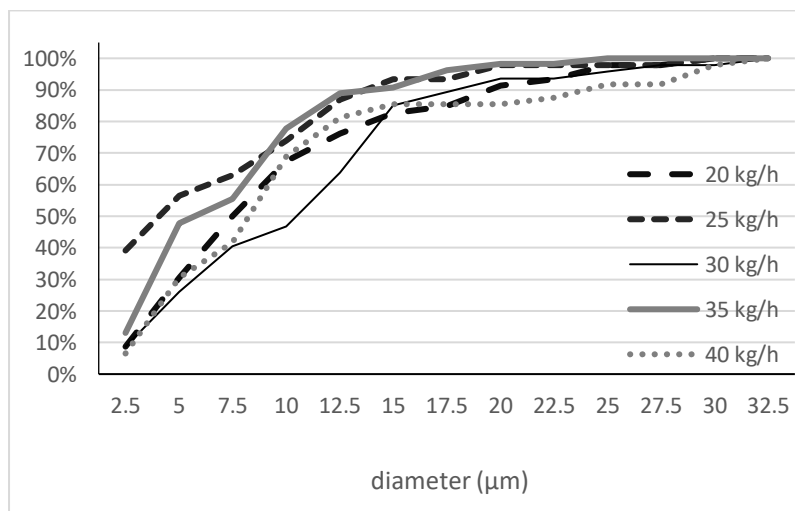


Figure 9. Cumulative frequency of particle size as a function of airflow at $T = 130^\circ\text{C}$.

5 DISCUSSION

In this study two operational parameters were selected to evaluate the performance of the spray dryer. The first was the inlet hot air temperature and the second was the airflow. Aiming to protect thermo-sensitive compounds in vegetable material, particularly polyunsaturated oils, the applied temperatures (110 to 150 °C) were lower than the commonly recommended in the literature (160 to 200 °C). In this work, different inlet airflows (20 to 40 kg.h⁻¹) were proposed to evaluate the influence of this parameters on the morphology of microparticles and on the energy consumption.

Results have shown that increasing airflow, more heat is carried out on the system, but at the same time diminishes the residence time (Fig. 2 and Fig.4). Overall, at T = 120 °C/AF = 25 kg.h⁻¹ presented energy enough to evaporate 99.5% of the water in the emulsion, but this condition presented low encapsulation efficiency, so T equal to 130 °C was selected. At this temperature, since the lower AF was enough to dry the emulsion, there is no need to go further. This selected condition presented outlet temperature of about 58 °C, low enough to avoid damages in thermo-sensitive compounds during transportation up to the cyclone.

6 CONCLUSIONS

At the selected temperature range (110 to 150 °C), the residence time of the gas in the drying chamber was the most relevant factor in the energy efficiency of heat and mass transport. Simultaneous increase in temperature and inlet airflow increased the heat loss to the environment without increasing the energy efficiency of the process. The lower airflow (20 kg.h⁻¹) was enough to obtain good homogeneity in the surface and size distribution of microparticles with less heat loss. The efficiency of microencapsulation was higher (95%) at 130 °C, indicating that it is possible to achieve good results at temperatures lower than those conventionally reported in literature, avoiding damage in thermo-sensitive compounds.

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