

The effect of some spray drying parameters on the kinetics and microencapsulation of sunflower oil

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Introduction

Spray drying is a continuously developed technique that is applied practically in fundamental research and mathematical modeling, and in many cases of microencapsulation [1-2]. Its numerous advantages – it is a one-stage continuous process, easy to scale-up, appear to be even greater with the advances of studies on drying specific substances with high process and technological requirements [3-6]. Microencapsulation and drying have been studied and discussed in papers published recently which confirms that such a research should be continued [2,6,7].

The aim of this study is to continue the earlier presented investigations [8,9] on a model multicomponent system using a properly adopted dryer, i.e. a vertical tunnel 8 m high and 0.5 m in diameter with spraying of a multicomponent emulsion, equipped with a laser system for investigation of sprayed stream in many cross sections of the tunnel. It is worth noting that this study contains determinations of both final product and “instantaneous” product to investigate the kinetics of microencapsulation and drying inside a drying chamber which will enable description of the process mechanisms and scaling-up.

Materials and Methods

The model systems to study microencapsulation and drying kinetics were water solutions of maltodextrin (dextrin equivalent 16) as a matrix material and sunflower oil as a core substance. 30%wt. concentrations of maltodextrin and 1.5, 3 and 4.5 wt.% oil content in solution were investigated. The emulsification procedure covered a two-stage method – a mechanical flow method using an Ultra-Turrax T25 (IKA-Werke) system at rotations frequency about 20000 min⁻¹ and ultrasonic flow method using an ultrasonic homogenizer UP 200S (Dr. Hielscher GmbH), a multiple flow was used for ca. 12 hours (power consumption – 100% homogenizer efficiency).

The spray drying process was carried out in an experimental vertical drying tunnel about 8 m high and 0.5 m in diameter with a rich equipment for data acquisition and process control [8]. The tunnel is equipped with a 60 kW heating system, waste air cooling system, dedusting system and optical glass control windows (10 measuring levels along 5.5 m tunnel length) to perform measurements using laser technique (LDA, PDA, FLOWLITE system by DANTEC). The construction enables taking samples at subsequent time intervals and making laboratory determination of moisture content, size distribution, etc. as well as quality index specific for a given product at a different distance from the atomizer.

The process was carried out in the concurrent system at the following parameters: air (drying agent) flow rate 200 Nm³/h, 300 Nm³/h, and 400 Nm³/h, inlet gas temperature 150°C, 175°C and 200°C, outlet gas temperature 90°C to 105°C. Liquid mixture was dispersed by two types (NL-0.6 and NL-1) of a pneumatic nozzle at the pressure 2 MPa and feed intensity 16 kg/h.

Dry particles geometry was determined by microscopic methods (Olympus BX 40) for visual assessment of particles and for a computer particle size analysis (program MicroImage v. 4.0).

The degree of encapsulation was calculated on the basis of spectrophotometric determinations of the amount of surface and total oil in powder with a Lambda 11 spectrophotometer (Perkin Elmer

UV/Vis, USA). The measurement was carried out at the wavelength 272 nm. A solvent that eluted oil from the powder was hexane.

For the determination of absolute material density an AccuPyc 1330 Helium Pycnometer (Micromeritics, USA) was used. Equilibrium moisture content was determined as sorption of water vapor in a NOVASINA (Axair Ltd., Pfaffikon, Switzerland) instrument type A_w SPRINT.

Results and Discussion

Figure 1 shows the impact of initial content of sunflower oil in the emulsion supplied to the nozzle. The most intensive change of moisture content in the disperse phase was observed at medium oil concentration, i.e. at 1.5 and 3.0 wt.%. The least intensive drying was in the case of maltodextrin solution with no oil added. A similar observation refers to the highest oil concentration 4.5 wt.%. This can be explained by the degree of dispersion of the emulsion in the spraying zone, which is confirmed by the change of particle diameter in this zone vs. oil content.

Figure 1 shows a diagram of changes in relative moisture content of disperse phase depending on the distance from the nozzle. The curves show that the process is very intensive, because from the initial suspension containing ca. 70 wt.% water, microcapsuled powder with moisture content close to final one (i.e. about 3 wt.%) is obtained already at the distance of ca. 1.5 m (and less) from the atomizer, i.e. at about 25% of the total measuring height of the tunnel.

Analysis of the impact of drying parameters shows that worse results (higher final moisture content) are obtained at the lowest values of drying parameters, i.e. air temperature 150°C and air flow rate 200 Nm³/h. Dried products (microcapsules) were also less stable and more difficult for microscopic and spectrophotometric analysis.

Figure 2 shows drying curves obtained at two different nozzles of diameter 0.6 and 1.0 mm. In the case of the smaller nozzle, the process of drying was more intensive; already at the distance smaller than 1 m from the nozzle moisture content in the powder was below 2 wt.%.

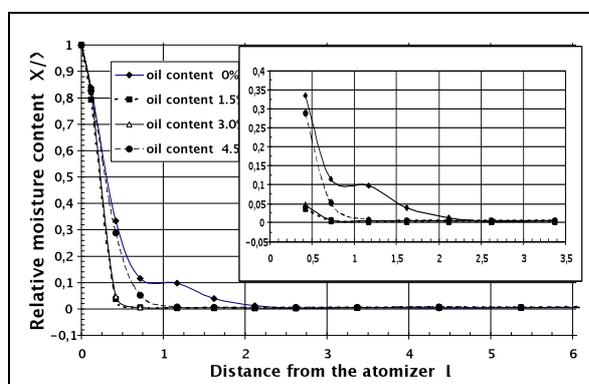


Figure 1. Relative material moisture content vs. distance from the nozzle at different oil content in the emulsion

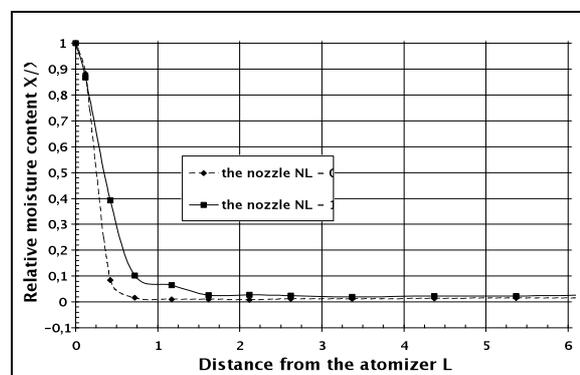


Figure 2. Relative material moisture content vs. distance from the nozzle at different type of the nozzle

The process efficiency (E) was estimated by calculating the ratio of the amount of oil encapsulated in microcapsules to the initial quantity of oil in the emulsion. Figure 3 illustrates a change of these values vs. distance from the atomizer, i.e. the pathway of particles in the drying tunnel at different drying parameters. The diagram presents results obtained at 3 wt.% oil content in the emulsion sprayed with the 0.6 mm nozzle.

With the distance from the nozzle, the general efficiency of encapsulation decreases also in extreme conditions of the process, i.e. at the highest air flow rate 400 Nm³/h and the lowest drying

temperature 150°C. A decrease of encapsulation efficiency along the tunnel can be explained by a destruction of material walls and breakage of part of the microcapsules.

A relatively high efficiency of microencapsulation reaching over 80% especially in the initial drying zone, with powder moisture content below 3 wt.%, is worth stressing. The results indicate that material residence time in the tunnel should be reduced to obtain high process efficiency.

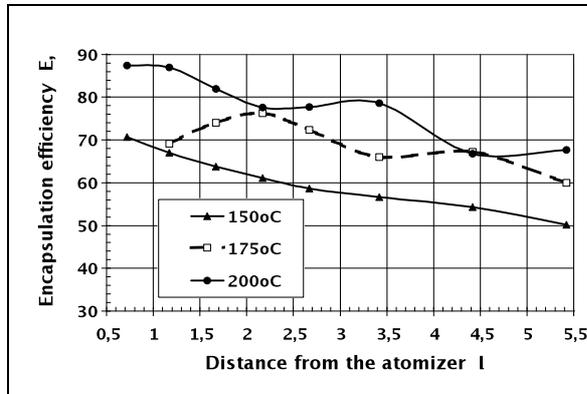


Figure 3. Encapsulation efficiency E_t vs. distance from the nozzle at different inlet air temperatures. Drying air flow $300 \text{ Nm}^3/\text{h}$

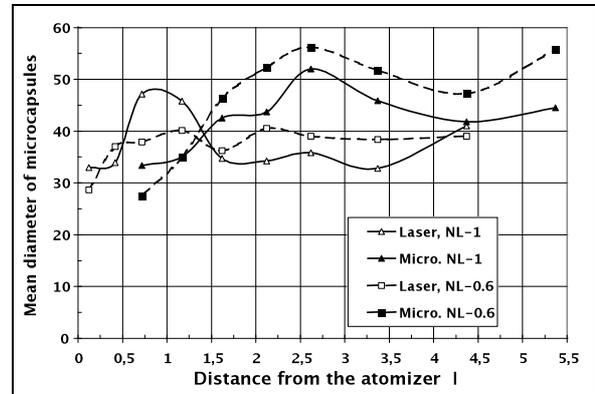


Figure 4. Mean diameter of microcapsules vs. distance from the nozzle at different types of nozzle and measuring methods. Air temperature 200°C and air flow $200 \text{ Nm}^3/\text{h}$

The structure of our drying tunnel equipped with a laser measuring device allows us to investigate the characteristic features of disperse phase transported in the tunnel. Examples of such measurements are given in Figure 4. They present averaged values of microcapsule diameters vs. distance from the atomizer calculated from detailed laser measurements taken in many points of the tunnel cross section and at its several levels.

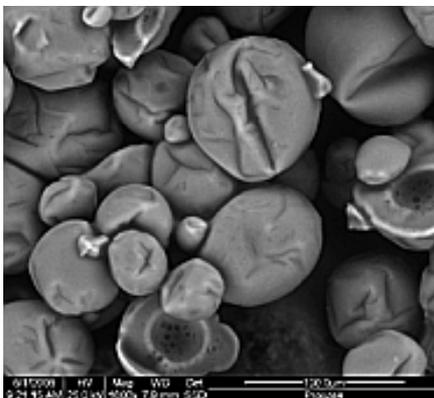


Photo 1. Microcapsules of sunflower oil in maltodextrin under scanning electron microscope, magnification $\times 1000$

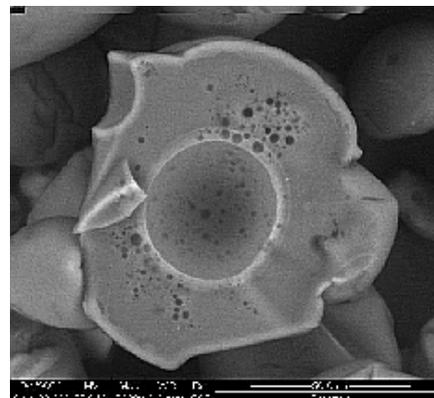


Photo 2. Microcapsules of sunflower oil in maltodextrin under scanning electron microscope, magnification $\times 2000$

It follows from the diagram that average diameters of the microcapsules containing oil range from 30 to 60 μm . Figure 4 shows a comparison of the values calculated from laser measurements with those calculated on the basis of microscopic image analysis. There is some regular discrepancy of results; in the initial zone of emulsion spraying and formation of microcapsules, laser measurements give slightly bigger diameters, while after exceeding the distance of ca. 1.5 m from the nozzle, the relation is reversed, i.e. particles measured by the laser meter have diameters of about 40 μm , while those observed and measured by the microscopic method have bigger particles reaching 50 μm . Additionally, results obtained while spraying with the smaller nozzle (0.6 mm diameter) provide

mean particle diameters slightly bigger (up to 5 μm) than those obtained when the bigger nozzle (1.0 mm) is used. The differences are not big (below 10%). Photos 1 and 2 show SEM images of microcapsules at the magnification of 1000 and 2000, respectively.

Conclusions

Investigations of the kinetics of spray drying and microencapsulation of disperse phase in an original drying tunnel provided new, valuable information on the process.

The highest efficiency of sunflower oil microencapsulation in maltodextrin was observed in the zone which is relatively close to the spraying nozzle (less than 1.5 m) where sufficiently dry powder (moisture content less than 3 wt.%) was obtained. The final result of the process was influenced by such process parameters as an optimum oil content – ca. 3 wt.%, drying temperature 200°C and drying air flow rate 200 to 300 Nm^3/h .

It can be successfully used for investigation of microencapsulation process of other multi-component emulsions with core substances such as food ingredients, pharmaceutical, biotechnological products, etc.

Acknowledgments

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References

- [1] Flores-Martinez H., et al., (2004), *Optimal spray-drier encapsulation process of orange oil*, Drying 2004, Proceedings of the 14th International Drying Symposium (IDS 2004), (eds. M.A. Silva, S.C.S. Rocha, State University of Campinas, Brazil), 22-25 August 2004, São Paulo, Brazil, vol. A, pp. 621-627.
- [2] Soottitantawat A., et al., (2005), *Microencapsulation of l-menthol by spray drying and its release characteristics*, Innovative Food Science and Emerging Technologies, Vol. 6, pp. 163-170.
- [3] Esposito E., et al., (2002), *Spray dried Eudragit microparticles as encapsulation devices for vitamin C*, International Journal of Pharmaceutics, No. 242, pp. 329-334.
- [4] Bruschi M.L., et al., (2003), *Gelatin microparticles containing propolis obtained by spray drying technique: preparation and characterization*, International Journal of Pharmaceutics, No. 264, pp. 45-55.
- [5] Jimenez M., et al., (2004), *Spray-drying microencapsulation and oxidative stability of conjugated linoleic acid*, European Food Research and Technology, vol. 219, pp. 588-592.
- [6] Bayram A., et al., (2005), *Spray drying of sumac flavour using sodium chloride, sucrose, glucose and starch as carriers*, Journal of Food Engineering, Vol. 69, pp. 253-260.
- [7] Tan L.H., et al., (2005), *Effect of oil loading on microspheres produced by spray drying*, Journal of Microencapsulation, Vol. 22, No. 3 (May), pp. 253-259.
- [8] Adamiec J., (2004), *Kinetics of fat encapsulation during spray-drying of multicomponent emulsions*, Proceedings of XII International Workshop on Bioencapsulation, (eds. J.L. Pedraz, G. Orive, D. Poncelet), 24-26 September 2004, Vitoria, Spain, pp. 311-314.
- [9] Adamiec J., et al. (2005), *The influence of spray-drying parameters on microencapsulation product quality*, Proceedings of XIII International Workshop on Bioencapsulation, (eds. R. Neufeld and D. Poncelet), 24-26 June 2005, Kingston, Ontario, Canada, pp. 11-12.