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INFLUENCE OF SPRAY DRYING CONDITIONS ON BEETROOT PIGMENTS RETENTION AFTER MICROENCAPSULATION PROCESS

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Abstract. In food colorants microencapsulation process, apart from appropriate carrier selection, the determination of the spray drying parameters which can affect the retention of active ingredients is essential. The aim of this study was to investigate the effect of drying parameters on beetroot pigments retention after microencapsulation. Raw material used in the study was the 100% beetroot juice. Low-crystallised maltodextrin DE=11 (MD) was used as the carrier. To obtain 30% dry matter concentration in the solution, the proper amount of maltodextrin was added to beet root juice with 15% of dry matter. Drying was carried out in a spray-drier at disc speed of 39,000 rpm and solution flux rate of $0.3 \cdot 10^{-6}$ and $0.8 \cdot 10^{-6}$ m^3 s^{-1}. The inlet air temperature was 120, 140 and 160^{o}C, at a constant air flow rate of 0.0055 m^3 s^{-1}. Before drying, viscosity and density of the solutions were measured. Dry matter content, apparent density, loose bulk density of the powder, and porosity were determined. The particle morphology was tested as well. Pigment content was measured by Nillson (1970) and Von Elbe (2001) methods to determine the efficiency of encapsulation. The viscosity and density of solutions of beet juice with maltodextrin was 3.86 mPa s and 1100 kg m^{-3}, respectively. In both cases, the values of viscosity and density were higher compared to the raw juice. Increase of solution flux rate caused a decrease of dry matter content, apparent particle density and loose bulk density. Increase of inlet air temperature caused an increase of dry matter content, average diameter and a decrease of both densities. It was observed that the increase of inlet air temperature caused a decrease in the yellow pigment to a higher degree (47%) than in the violet pigment (17%). However, no clear correlation was observed for violet pigment. There were no changes in porosity and shape factor. The obtained microcapsules were sphere-like in shape, with numerous deep cavities. In the whole experiment the retention of beet root pigments was in the range of 26.7-29.3%.

Keywords: microencapsulation, morphology of powders, betalain pigments, density of powders

INTRODUCTION

Every year the food industry introduces a wide range of products onto the market. Each with different structure, new shape, desired characteristics of flavour and colour. Dried vegetable and fruit juices can be considered as a one of new forms of product introduction onto the market. Fruit and vegetable powders are used in many kinds of food such as beverages, soups, cakes (Khalil *et al.* 2002), pastry fillings, ice cream and yogurt (Komes *et al.* 2007). Nutritional values of fruit and vegetable powders make them a very important ingredient of sauces, snacks, baby food, or extruded cereal products (Grabowski *et al.* 2008). During the last decade there has been a significant increase of consumer interest in natural foods without any artificial additives. As a result, companies started to use native components such as natural plant or animal origin pigments (Henry 1995). However, natural colorants can be sensitive to environmental or technological process conditions. Therefore, the industry should pay more attention to the microencapsulation process, for instance of plant-based material like beetroot.

In Poland beetroot is the second most important vegetable in terms of production and consumption. Beetroots show ability for long-term storage which makes them available all year. Red beetroots contain 65.7% of water, 1.4% protein, 4-8% sugar, 0.3% fat, 1% fibre and mineral salts of calcium, phosphorus, magnesium and iron. Beetroots colour is caused by the presence of water-soluble betalain colorants: purple to violet betacyanins and yellow to orange betaxanthins (Ravichandran *et al.* 2011, Gokhale and Lele 2011). In recent years the interest in beetroots increased, mainly because of the betalain antioxidant activity and preventive potential against selected degenerative diseases (Georgiev *et al.* 2010, Azeredo *et al.* 2009). The total betacyanin and betaxanthin content of red beetroots varies within the ranges of 47-58 mg (100 g d.m.)$^{-1}$ for betaxanthin (Gaertner and Goldman 2005) and 525-875 mg (100 g d.m.)$^{-1}$ for betacyanin (Nowak and Syta 2009), depending on the cultivar. Like the other pigments, betalains are sensitive to external factors, mainly temperature and oxygen, and their stability depends on pH (from 3 to 7) (Henry 1995).

Microencapsulation can be considered as one of many methods used to preserve compounds of natural origin. What is more, it allows to obtain a product in powder form. Enclosed material is protected from the environmental factors and thus the final product is stable. The main objective of the encapsulation process is to create a barrier between the core material and the environment. The barrier is created by the carrier material which can create an envelope or a matrix. One of the most widely used techniques of microencapsulation is spray drying. Spray drying of beetroot extract is reported on by several research groups (Roy *et al.* 2004, Desai and Park 2005, Pitalua *et al.* 2010). In this process, microencapsulated vegetable juice is mixed with a carrier like maltodextrin, Arabic gum or starch. Since the final product is free of water, a significant concentration of other ingredients such as betalains is observed, even if during the drying their partial degradation has taken place. Spray drying as a microencapsulation method allows to obtain a product characterised by various physical properties which depend on drying parameters: temperature, solution flux rate or the carrier concentration (Ersus and Yurdagel 2007, Loksuwan, 2007).

Therefore, the aim of the study was to investigate the effect of spray drying parameters on the physical properties and microencapsulation efficiency of betalain from red beetroot juice.

MATERIALS AND METHODS

Raw material for the study was the 100% beetroot juice (SVZ, Tomaszów Lubelski, Poland). Low-crystallised maltodextrin DE=11 (PPS "PEEPES" S.A., Łomża, Poland) was used as the carrier. To obtain 30% dry matter concentration in the solution, the proper amount of maltodextrin was added to beet root juice with 15% of dry matter.

Apparent viscosity of the solutions (η) was tested in a viscometer (Brookfield, model RVDV-III, Middleboro, MA, USA), as a spindle an Ultra Low Adapter (ULA) was used, with the range of shear rate from 20 to 100 rpm. Sample volume for the tests was 16 mL.

The density of the solutions (kg m^{-3}) was determined by the pycnometric method and the calculations were made according to the formula presented by Janiszewska *et al.* (2010).

Drying was carried out in the semi-industrial spray drier LAB S1 (drying tower dimensions: diameter of approximately 1000 mm, cylindrical height approx. 820 mm, conical height 1020 mm) (Anhydro, Copenhagen, Denmark) at spray disk speed of 39,000 rpm and solution flux of $0.3\cdot10^{-6}$ and $0.8\cdot10^{-6}$ m^3 s^{-1}. Drying was carried out with the co-current method. The inlet air temperature was 120, 140 and 160°C at a constant air flow of 0.055 m^3 s^{-1}.

The powders obtained were described by dry matter content, the apparent particle density, the loose bulk density, the porosity of the powder, size and structure of the microcapsules. The content of pigments (Nillson 1970, Von Elbe 2001) needed to determine the microencapsulation efficiency of pigments in the powders obtained was examined as well.

The dry matter of powder (DM) was determined according to PN-A-790011/3. The apparent density was measured in a helium pycnometer, Quantachrome's Stereo-pycnometr (Boyton Beach, USA). Loose bulk density was determined using a vibrating volumeter (STAV2003 Engelsmann AG Ludwigshafen, Germany) (Domian and Bialik, 2006). Porosity was calculated from the equation presented by Janiszewska *et al.* (2010).

The analysis of the structure and size of powder particles was based on images taken by a scanning electron microscope, Hitachi TM3000 (Hitachi High-Technologies Corporation, Tokyo, Japan). Projected diameter of powder particles was determined by the MultiScan v 18.03 software (Warsaw, Poland) (Janiszewska *et al.* 2011). Additionally, on the basis of the area (A) and perimeter (L) of the powder particles, the aspect ratio (W) was calculated from the formula presented by Dajnowiec *et al.* (2011):

$$W = 2\frac{\sqrt{\pi A}}{L} \quad (1)$$

Quantification of betalains was performed by the spectrophotometric method of Nillson (1970) and Von Elbe (2001), using a spectrophotometer Helios Gamma (Thermo Spectronic, Cambridge, Great Britain). Identification of pigments was carried out on each stage of the technological process – in beetroot juice with maltodextrin and in the resulting powder. Pigments were extracted from the sample with a phosphate buffer at pH 6.5. The buffer was prepared as follows: to 1000 cm^3 of 0.05 M solution of potassium dihydrogen orthophosphate (KH_2PO_4) 450 cm^3 of a 0.05 M solution of sodium hydrogen orthophosphate (Na_2HPO_4) was added and thoroughly stirred. To this solution 0.1 g of disodium EDTA was added. Before testing the pH was controlled.

1 g of beetroot solution or powder was added into the 100 cm^3 volumetric flask and supplemented with buffer, thoroughly mixed and filtered through corrugated filters.

The determination of betalain concentration, i.e. violet and yellow pigments, was calculated in terms of betanin and vulgaxanthin-I, respectively. Total pigment content was expressed as the sum of violet and yellow components. Pigment content calculations were based upon the absorptivity values $A^{1\%}$ which were 1120 for betanin (at 538 nm) and 750 for vulgaxanthin-I (at 476 nm). According to the Nillson (1970) and Von Elbe (2001) methodology, absorbance at 600 nm was measured and used to correct the amounts of impurities.

Absorbance value for the violet colours (A_V) was calculated from the formula:

$$A_V = 1.095\left(A_{538} - A_{600}\right) \quad (2)$$

where: A_{538} – light absorption of the sample at 538 nm, A_{600} – light absorption of the sample at 600 nm, 1.095 – factor related to the increase in absorbance at 538 nm wavelength length due to the presence of impurities.

Content of violet colours in tested sample (*BV*) (mg (100g d.m)$^{-1}$) was calculated from the equation:

$$BV = \frac{1000 \cdot A_V}{(1120 \cdot a \cdot DM)} \quad (3)$$

where: 1000 – conversion of grams to milligrams, *a* – sample weight (g).

Absorbance for the yellow pigment (A_Y) was calculated from the formula:

$$A_Y = A_{476} - A_{538} + 0.677 \cdot A_V \quad (4)$$

where: A_{476} - light absorption of the sample at 476 nm.

Yellow pigment content (*BY*) (mg (100g d.m.)$^{-1}$) was calculated from the formula:

$$BY = \frac{1000 \cdot A_Y}{(750 \cdot a \cdot DM)} \quad (5)$$

where: 1000 – conversion of grams to milligrams, *a* – sample weight (g).

Betalain retention (BR, %) was determined from the formula:

$$BR = \frac{B_p \cdot DM_j}{0.41\, B_j \cdot DM_p} 100 \quad (6)$$

where: *B* – sum of betanin (BV) and vulgaxanthin-I (BY) in solution of beetroot juice with maltodextrin and in powder (mg (100 g d.m)$^{-1}$), 0.41 – share of dry matter of juice in the dry matter of microcapsules, Indexes: *j* – juice, *p* – powder.

Drying trials and analyses were repeated in triplicate. Data are presented as mean ± standard deviation. Significance of inter-group differences was determined by one-way analysis of variance (ANOVA) also interaction between parameters (T and feed flux) was tested by multifactor ANOVA, with Statgraphics Plus 5.1 Software. Individual group differences were identified using the Tukey multiple range at a significance level of 0.05.

RESULTS AND DISCUSSION

Beetroot juice and the solutions with maltodextrin were Newtonian fluids over the entire range of shear rates. The viscosity of the raw juice was 1.13±0.13 mPa s. The addition of maltodextrin caused a statistically significant increase in viscosity up to 3.72±0.55 mPa s, which was associated with increase of dry matter content in the solution.

For all tested physical properties of the powders (Tab. 1) interactions temperature-feed flux were statistically significant (p less than 0.05).

The increase of dry matter content from 15 (in juice) up to 30% (juice solution with maltodextrin) caused an increase of density from 1048±1 to 1097±1 kg m^{-3}.

Changing the inlet air temperature from 120 to 160°C resulted in a significant decrease of apparent particles density, loose bulk density of powders, and an increase of the average diameter (Tab. 1). Increase of dry matter content was significant only at higher solution flux rate. Ersus and Yurdagel (2007) in the purple carrot powders based on maltodextrin, Kha *et al.* (2010) in the *Momordicaco chinchinensis* fruit powders, and Cai and Cork (2000) in amaranth powder, based also on maltodextrin as the carrier, observed analogous positive correlations between the inlet air temperature and dry matter content of powders. It could be explained by higher heat transfer coefficient at higher processing temperatures, which could result in faster evaporation of water from the droplet during drying. This fact was also noticed by Kha *et al.* (2010) and Fazaeli *et al.* (2012).

The change of raw material feed stream from 0.3 to 0.8 mL s^{-1} resulted in reverse dependence in the case of the dry matter content as when the temperature was increased. Evaporation occurred most intensely in powders obtained at a lower feed flux of raw material, regardless of the inlet air temperature. Moreover, this resulted in obtaining powders with the highest dry matter content (Tab. 1). Increase of raw material feed flow resulted in a decrease of dry matter also in experiments of Cai and Cork (2000) and Jimenez-Aguilar *et al.* (2011).

Table 1. Selected physical properties of microcapsules

Inlet temperature (°C)	Feed rate (10^{-6} m^3 s^{-1})	Dry matter content (%)	Apparent particles density (kg m^{-3})	Loose bulk density (kg m^{-3})	Porosity (–)	Average diameter (µm)	Particles aspect ratio (–)
120	0.3	96.99^{c}±0.29	1389^{d}±9	615^{e}±20	0.56^{a}±0.03	9.36^{a}±0.69	0.87^{a}±0.02
	0.8	95.02^{a}±0.10	1220^{b}±30	466^{b}±17	0.62^{bc}±0.01	9.89^{a}±0.23	0.87^{a}±0.03
140	0.3	97.23^{c}±0.31	1334^{cd}±29	583^{d}±15	0.56^{a}±0.01	10.03^{a}±0.01	0.86^{a}±0.03
	0.8	95.25^{a}±0.8	1218^{b}±12	412^{a}±11	0.66^{c}±0.01	10.28^{b}±0.07	0.86^{a}±0.03
160	0.3	97.17^{c}±0.28	1272^{bc}±28	507^{c}±20	0.60^{ab}±0.02	11.39^{c}±0.06	0.86^{a}±0.03
	0.8	96.21^{b}±0.79	1112^{a}±3	410^{a}±14	0.63^{c}±0.01	12.81^{d}±0.03	0.87^{a}±0.03

Mean values in the same column denoted with different letters: a, b, c, differ statistically at p = 0.05.

It was shown that increase of inlet air temperature caused a decrease in apparent particles density, but only for the higher feed flux rate at the temperature of 140-160°C (Tab. 1). The same relationship was observed by Finney *et al.* (2002) who examined the effect of spray drying parameters on physical properties of microencapsulated flavours. Correlation between inlet air temperature and apparent particles density during spraying of liquid droplets is related to the fact that a lower amount of hot air enters the particle and during the evaporation process it stays enclosed inside the particle. Similarly, the coating material itself, maltodextrin, results in a smaller apparent particles density at higher temperatures.

Loose bulk density decreased with the increase of temperature and solution flux rate. Decrease of loose bulk density with the temperature increase from 120 to 160°C was also observed by Chegini and Ghobadian (2005) in the case of orange juice microencapsulation, and by Fazaeli *et al.* (2012) for black mulberry juice. According to those authors, decrease in loose bulk density is due to the higher evaporation rates which can produce more porous powders with lower shrinkage of the droplets during drying. This phenomenon is confirmed also in the presented research (Tab. 1).

The porosity and particles aspect ratio did not differ within themselves when temperature was changed.

The increase of temperature and solution flux rate caused an increase in the diameter of beetroot juice microcapsules. This can be caused by faster microcapsules formation with the higher temperature, which is connected to faster water evaporation. On the other hand, the increase in mean diameter with increasing solution flux rate is associated with higher supply of the solution per second to the spray disc, which creates larger droplets and thus larger particles after the drying. The same relationship between the diameter growth and increasing both the temperature and feeding flux was obtained by Chegini and Ghobadian (2005, 2007) who spray-dried solutions of orange juice with maltodextrin as the carrier.

All powders, regardless of the drying parameters, had spherical shape (Photo. 1, Tab. 1). Analysing the particles in the images, two types of morphology can be distinguished: powders with smooth surface, but with smaller semicircular diameter, and larger particles with very strongly folded surface. Fruit or vegetable powders obtained by spray drying of juice solution containing carriers like maltodextrins were close to spherical shape with numerous dents (Cai and Cork 2000, Ersus and Yurdagel 2007, Obón *et al.* 2009, Robert *et al.* 2010, Fazaeli *et al.* 2012).

In the solution of beetroot juice and maltodextrin, betalain content was 248.2 ±1.4 (mg (100 g d.m.)$^{-1}$), including violet pigment in the range of 141.6±0.5 (mg (100 g d. m.)$^{-1}$) and yellow pigment as vulgaxanthin-I 106.6±0.9 (mg (100 g d.m.)$^{-1}$). It turn, in powders, violet pigment as betanin content ranged from 117 to 123 (mg (100 g d.m.)$^{-1}$) (Fig. 1.a), while the value of yellow pigment ranged from

57 to 67 (mg (100 g d.m.)$^{-1}$) (Fig. 1.b). Similar values for beetroot juice powder based on maltodextrin spray dried at 150^{o}C were obtained by Nemzer *et al.* (2011) for a Polish variety of beetroot.

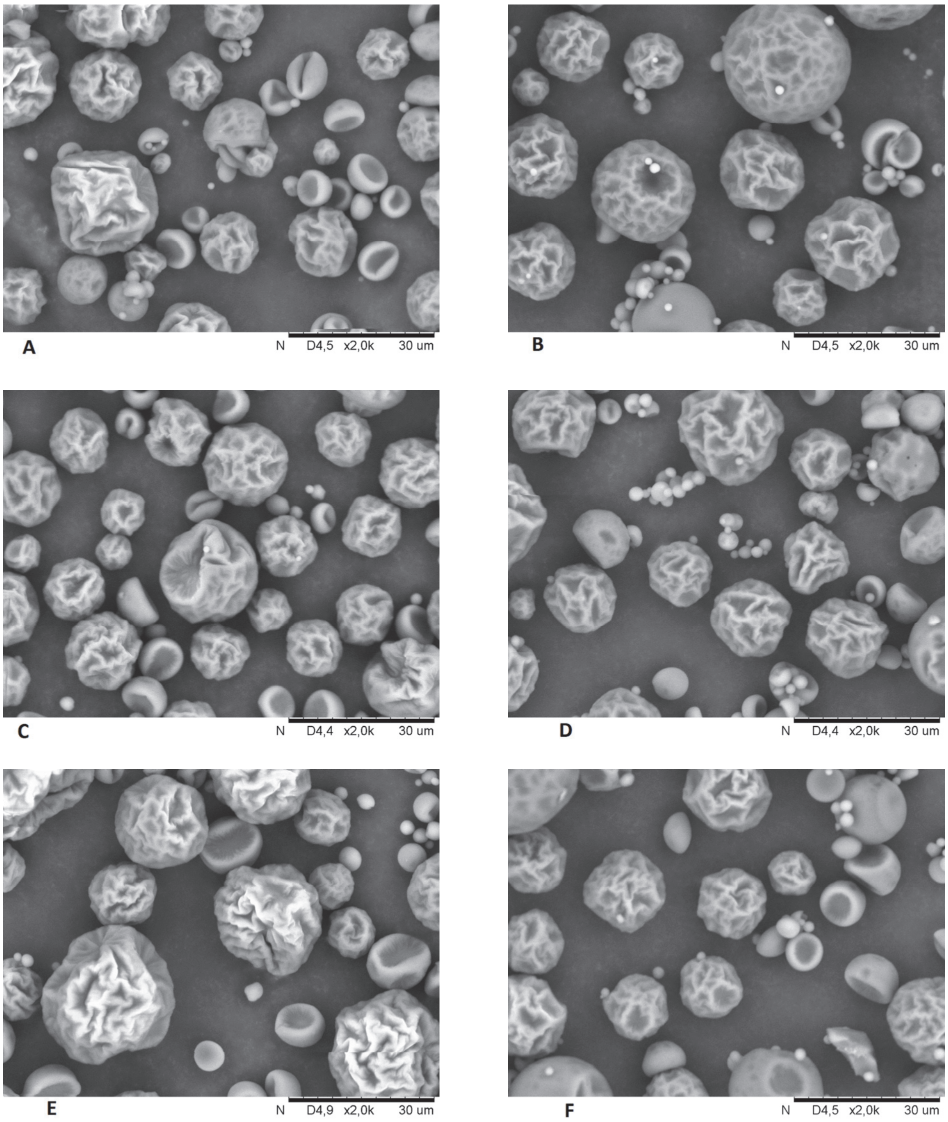

Photo. 1. Morphology of microcapsules of beetroot juice with maltodextrin as carrier (magnification 2000 x). A) 120°C, 0.3 (mL s^{-1}), B) 120°C, 0.8 (mL s^{-1}), C) 140°C, 0.3 (mL s^{-1}), D) 140°C, 0.8 (mL s^{-1}), E)160°C, 0.3 (mL s^{-1}), F) 160°C, 0.8 (mL s^{-1})

The decrease is acceptable and the obtained powder could be used in food industry as a colorant.

A significant negative effect of air temperature on the violet and yellow beetroot pigments content was observed. A similar tendency was observed by Quek *et al.* (2007) and Solval *et al.* (2012) who examined the spray drying of melon and watermelon juice based on maltodextrin. The β-carotene content in the obtained powders decreased with increasing inlet air temperature

No clear tendency between increasing the solution flux rate and the violet betanin pigment content was observed (Fig. 1.a). For the yellow pigment vulgaxanthin-I content an upward trend with increasing the solution flux rate was noted. However, this increase was statistically significant only for temperature of 160^{o}C (Fig. 1.b).

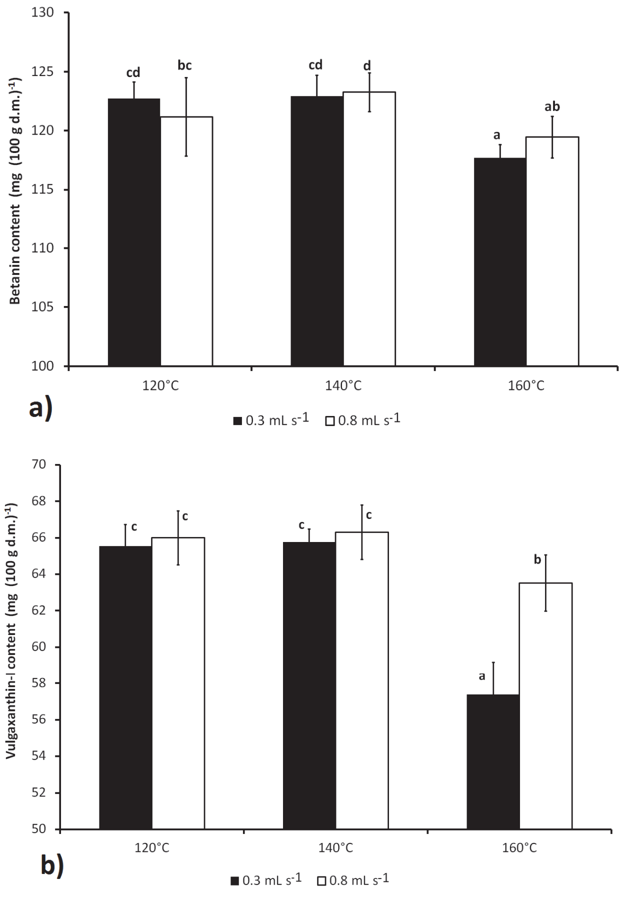

Fig. 1. Content of betalain a) violet pigment – betanin (BV), b) yellow pigment – vulgaxanthin-I (BY). Mean values denoted with different letters: a, b, c, differ statistically at p = 0.05

Such a different betanin colours behaviour during drying may be due to their chemical structure (Neelwarne and Halagur 2013). Vulgaxanthin-I has less molecular mass and its chemical structure is not so complicated as that of betalain, which may result in greater sensitivity to high temperature and rapid degradation.

The betalain retention (BR) after the spray drying process ranged from 26.7 to 29.3% (Fig. 2). Increase of the solution flux rate caused an increasing tendency in betalain retention. Robert *et al.* (2010) and Saénz *et al.* (2009) obtained retention of polyphenols at the level of 51-83% and 92%, respectively.

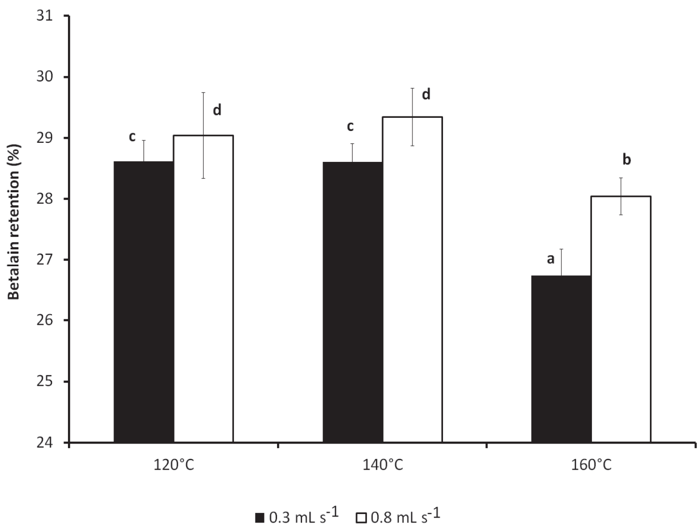

Fig. 2. Betalain retention (BR). Mean values denoted with different letters: a, b, c differ statistically at p = 0.05

The increase in inlet air temperature decreased betalain retention. Similar results were observed by Chegini and Ghobadian (2007) for orange juice, and by Saénz *et al*. (2009) for cactus pear juice, both with maltodextrin as the carrier.

The powder was characterised by a high content of pigments, although it decreased compared to the starting juice. The inlet air temperature of 140°C could be recommended as a good condition because of high content of betanin pigments and also because of good physical properties of microcapsules (particle size, porosity, apparent particles density, loose density and dry matter content).

CONCLUSIONS

1. Increase of solution flux rate caused an decrease of dry matter content of powders, apparent particles density, loose bulk density and particles diameter. The increase of inlet air temperature caused an increase of dry matter content, and decrease in both treatment densities.

2. The porosity and particles aspect ratio did not differ within themselves when temperature was changed.

3. Obtained microcapsules were sphere-like shaped with numerous deep indentations.

4. It was observed that increase of inlet air temperature caused higher decrease in yellow colour (about 46%) than in violet colour of beetroot juice (almost 17%).

5. In the whole experiment the retention of beet root pigments was in the range of 26.7-29.3%.

6. The inlet air temperature of 140^{o}C could be recommended as a good drying condition because of the higher content of betanin pigments and also because of good physical properties of microcapsules, which is important for storage.

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WPŁYW PARAMETRÓW SUSZENIA ROZPYŁOWEGO NA STOPIEŃ ZAMKNIĘCIA BARWNIKÓW BURAKA ĆWIKŁOWEGO PO PROCESIE MIKROKAPSUŁKOWANIA

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Streszczenie. W procesie mikrokapsułkowania barwników, poza dobraniem odpowiedniego nośnika, istotne jest ustalenie parametrów suszenia rozpyłowego, mających wpływ na stopień zatrzymania rdzenia. Celem pracy było zbadanie wpływu zmiennych parametrów suszenia na stopień zatrzymania barwników buraka ćwikłowego po procesie mikrokapsułkowania. Surowiec do badań stanowił 100%-owy sok z buraka ćwikłowego. Jako nośnik używano maltodekstryny niskoscukrzonej DE=11 (MD). Do soku o 15% zawartości suchej substancji dodawano proszek maltodekstryny w celu uzyskania roztworu o 30% stężeniu suchej masy. Suszenie sporządzonych roztworów prowadzono w suszarce rozpyłowej LAB S1 firmy Anhydro, przy prędkości dysku rozpyłowego 39000 obr·min^{-1} i strumieniu surowca $0{,}3\cdot10^{-6}$ oraz $0{,}8\cdot10^{-6}$ m^3·s^{-1}. Suszenie odbywało się współprądowo, a temperatura powietrza wlotowego wynosiła 120, 140 i 160°C, Natężenie przepływu powietrza było stałe i wynosiło 0,0055 m^3·s^{-1}. Przed suszeniem oznaczano lepkość i gęstość roztworów. W otrzymanych proszkach oznaczano zawartość suchej substancji, gęstość pozorną proszku, gęstość luźną złoża proszku, porowatość złoża, określano morfologię cząstek oraz zawartość barwników metodą Nillson'a (1970) i Von Elbe (2001) w celu wyznaczenia efektywności procesu kapsułkowania. Lepkość roztworów soku buraczanego z maltodekstryną wynosiła 3,86 mPa·s, gęstość natomiast 1100 kg·m^{-3}, w obu przypadkach uzyskane wartości były wyższe w porównaniu do surowego soku. Wzrost prędkości podawania surowca spowodował spadek zawartości suchej substancji w proszku, gęstości pozornej, gęstości luźnej złoża proszku. Wzrost temperatury powietrza wlotowego spowodował wzrost zawartości suchej substancji i średniej średnicy cząstek oraz spadek obu badanych gęstości. Zaobserwowano, iż wzrost temperatury powietrza wlotowego spowodował większy spadek zawartości barwnika żółtego (ok. 47%) w porównaniu do obniżenia zawartości barwnika czerwonego (ok. 17%). Nie zaobserwowano zmian w wartościach porowatości oraz współczynnika kształtu. Otrzymane mikrokapsułki były zbliżone kształtem do kuli z licznymi głębokimi wklęśnięciami. W całym eksperymencie efektywności kapsułkowania mieściły się w zakresie od 26,7 do 29,3%.

Słowa kluczowe: mikrokapsułkowanie, morfologia cząstek, barwniki betalainowe, gęstość proszku