Optimization of production technology for encapsulated functional detoxicants

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Abstract. This study purpose was to optimize the production technology of encapsulated functional detoxicants based on combined dietary fibers (DF) obtained from beet pulp according to the developed technology. Production technology optimization for functional encapsulated food products was performed using mathematical programming methods based on Statistica v.10 and MathCAD v.15 software environments. An optimal technological production mode for encapsulated food products with high quality indicators, having an increased detoxification ability in relation to heavy metal ions, including combined DF, obtained from beet pulp.

1 Introduction

To protect human biochemical systems from technogenic factors, dietary fibers (DF), as physiologically active components of the human diet, should be included in the diet [1-3].

Secondary products of plant raw materials [4] processing, in particular, beet pulp, the most common by-product of sugar beet production seem to be perspective - about 20 million tons of this resource is produced in Europe every year [5, 6]. At present, highly efficient technologies have been developed to produce dietary fiber from beet pulp [7], they are distinguished by a high content of pectin substances and fiber, they may be used as a food additive in functional food production [8, 9].

At present, an urgent sector in food engineering, food industry and public catering industry is supported by innovative technologies, including natural structure-forming agents in the recipe formulation [10]. Alginates, a group of marine and bacterial structural exopolysaccharides, such as alginic acid, as well as its salts and derivatives, act as the most preferred and widespread materials for the preparation of bulk, macro- and microgels [11]. The use of sodium alginate is especially important in the encapsulation technology implementation, allowing for increased delivery stability and efficiency of encapsulated biologically active ingredients, including dietary fiber, with their more complete release in the human gastrointestinal tract [12].

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2 Materials and Methods

2.1 Study objects

Sodium alginate (viscosity of 1 wt% dissolved in water is 350-550 mPa s) according to GOST 33310-2015, anhydrous calcium chloride according to GOST R 55973, distilled and deionized water, potassium dichromate according to GOST 2652, black chromogen according to TU TU6-09-1760-72, lead (II) acetate according to GOST 1027. Combined DF were produced according to a previously developed technology from beet pulp using electrophysical and chemical exposure methods [13].

2.2 Preparation of model mixture samples to be encapsulated

The mixtures to be encapsulated were prepared as follows. A weighed portion of sodium alginate powder in an amount of 1% was dissolved in deionized water with constant stirring in Intllab MS-500 magnetic stirrer for 3-4 minutes [14]. To remove dissolved in the liquid phase, the solution was put in a VacModul vacuum module with VP1 vacuum pump for VD 53. Combined DF were added to the resulting structure former solution and stirred for 3-4 minutes until a homogeneous system was obtained.

The solution, forming a capsule shell containing polyvalent metal ions (required for the structure formation of a sodium alginate solution) was produced by dispersing calcium chloride in deionized water in an amount of 0.5%.

The mixture of biopolymers was extruded by axial feeding into the forming medium at a ratio of 1:10 through a device for encapsulated products with a given size of outlet nozzles, 2 mm in diameter. The resulting rounded-oval capsules were exposed in the interval from 1 to 3 minutes, washed with deionized water and dried by blowing air through a fine filter.

2.3 Determination of capsule syneresis

The syneresis value was calculated by gravimetric method to determine the amount separated from a 100 g sample of moisture over a regulated time interval.

2.4 Determination of capsule density

The capsule density was determined by hydrostatic weighing using VIBRA AFDK kit and SHINKO A.F.225 DRCE analytical balance.

2.5 Determination of binding capacity

The binding capacity of the biopolymer mixture to be encapsulated was determined by titration. For this, 25 ml of a model solution of polysaccharides was mixed with 30 ml of a 0.1M solution of lead acetate (Pb(CH3COO) 2). The mixture was kept with continuous shaking in SKO-180 Labtex laboratory rotation shaker and separated into fractions in Sartorius laboratory centrifuge, Sigma 2-6 10223. The precipitate was washed with distilled
water until a negative qualitative reaction for lead ions with potassium dichromate (K₂Cr₂O₇). The resulting fraction and washings were combined and made up to the mark with distilled water in a 250 ml flask. A 10 ml aliquot of the resulting solution was titrated with 0.01 mol/l solution of ethylenediamine tetraacetate (EDTA) at 9 to 10 pH in the presence of black chromogen until the color changed from violet to blue.

The binding capacity (BC) was estimated by the value of metal ions binding and calculated by the formula:

$$BC = \frac{A_1 - A_2}{A_1} \cdot 100\%,$$

where $A_1$ is the total mass of metal, g

$A_2$ is mass of metal remaining in solution, g

In equation (1), values $A_1$ and $A_2$ were calculated from the results of titrometric tests using the following formulas:

$$A_1 = \frac{C_{EDTA} \cdot V_1 \cdot M}{1000},$$

$$A_2 = \frac{C \cdot 250 \cdot V_{cp} \cdot M}{1000 \cdot V_2},$$

where $C$ is the concentration of initial metal solution, mol/l;

$V_1$ is initial volume of metal solution, ml;

$V_2$ is aliquot solution of the centrifugate taken for titration, ml;

$V_{cp}$ is average volume of EDTA used for titration, ml;

$M$ is metal molecular weight, Da.

### 2.6 Multicriteria optimization of study results

The multicriteria technology optimization for production of encapsulated food products with combined DF was performed using mathematical programming methods based on Statistica v.10 and MathCAD v.15 software.

### 3 Results and Discussion

To determine optimal technological parameters for encapsulated products production, a mathematical model of a two-factor experiment was developed.

Taking into account the previously obtained results [8, 14], the factors with the highest effect on target functions $Y_1$ (density of resulting capsules) ≤ 1.2 g/cm³, $Y_2$ (capsule binding capacity relative to Pb²⁺ ions) → max and $Y_3$ (capsule syneresis after 72 hours of storage) → min:

- $X_1$ is the amount of combined DF injected into a solution with a mass fraction of 1% sodium alginate, %;
- $X_2$ is the residence time of capsules in the forming solution with a mass fraction of 0.5% calcium chloride, s.

For two-factor optimization, the solution can be obtained graphically and coincides with experimentally calculated solution.

A graphical display of the capsule syneresis dependence after 72 h of storage is presented (FIG. 1), the binding capacity of capsules relative to lead ions Pb²⁺ (FIG. 2) and density of resulting capsules (FIG. 3) depending on the amount of combined DF introduced into the solution with a mass fraction of 1% sodium alginate, % and the residence time of
capsules in the forming solution with a mass fraction of 0.5% calcium chloride, s.

**Fig. 1.** Capsule syneresis after 72 hours of storage; $Y_3 \rightarrow \text{min}$. 

Fig. 1 shows that when the value is limited, target function $Y_3 \rightarrow \text{min}$ is achieved with the following possible values $X_1 = 9\%$, $X_2 = 60$ s.

Fig. 2 shows that the max. value of $Y_2$ is set with the following possible values $X_1 = 12\%$, $X_2 = 60$ s.

Fig. 3 shows that when target function value $Y_1 \leq 1.2 \text{ g/cm}^3$ is limited at $X_1 = 9\%$, $X_2 = 60$ s.

To determine optimal values $X_1, \%$ and $X_2$, using the mode by means of MathCAD v.15.0 package, an attainability set and an optimal solution for $Y_3$ and $Y_2$ are constructed (Fig. 4).

According to Fig. 4, the optimal technological parameters to determine encapsulated products $X_1 = 9\%$, $X_2 = 60$ s, when the values of target functions $Y_3 \rightarrow \text{min}$ and $V_2 \rightarrow \text{max}$ and $Y_1 \leq 1.2 \text{ g/cm}^3$ are achieved.

**Fig. 2.** Capsule binding capacity relative to ions Pb$^{2+}$; $Y_2 \rightarrow \text{max}$. 
Fig. 1. Capsule syneresis after 72 hours of storage; $Y_3 \rightarrow \min$.

Fig. 2 shows that when the value is limited, target function $Y_3 \rightarrow \min$ is achieved with the following possible values $X_1 = 9\%; X_2 = 60\,$s.

Fig. 3 shows that the max. value of $Y_2 \rightarrow \max$ is set with the following possible values $X_1 = 12\%; X_2 = 60\,$s.

Fig. 3. Density of resulting capsules; $Y_1 \leq 1.2\,$g/cm$^3$.

Fig. 4. Attainability set and an optimal solution for $Y_3$ and $Y_2$.

Thus, the optimal technological parameters for encapsulated products with combined DF were determined: the amount of combined DF introduced into the sodium alginate solution is 9%; exposure time of capsules in the forming solution of calcium chloride is 60 s. In this case, the capsule density is 1.18 g/cm$^3$, the binding capacity relative to lead ions Pb$^{2+}$ is 99.1%, the capsule syneresis is 1.95%.

4 Conclusions

The multicriteria optimization of technological modes of combined food products was performed using mathematical programming methods implemented by means of Statistica v.10 and MathCAD v.15 software environments.

The optimal technological modes for the production of functional encapsulated food products were determined, with increased sorption capacity relative to heavy metal ions, with combined DF obtained from beet pulp.

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