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Optimization of spray drying process parameters for kefir powder using response surface methodology

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ABSTRACT

In this study response surface methodology (RSM) was used to optimize the spray drying process conditions for production of kefir powder. Influence of inlet air temperature (120–180 °C), feed temperature (4–30 °C) and pump rate (20–40%) on the survival rates of microorganisms, outlet temperature, moisture content and water activity were assessed after drying and modeled by RSM. A lab-scale spray dryer (Mini Spray Dryer B–290, Switzerland) was used to carry out the drying experiments which are planned according to Central Composite Rotatable Design (CCRD). Inlet temperature was found as the main factor that effects the all responses statistically significant (p < 0.05). Effect of pump rate on the responses was found significant for some responses. Feed temperature has no significant effect for any responses. The optimum conditions were found as 135 °C inlet air temperature and 35% pump rate with using Desirability Function. Desired parameters were determined in regard to model fit and lack of fit test analysis results. At the end of this study, optimum conditions of spray drying were matched with freeze drying results. Results showed that at the optimum point, good quality powder can be obtained as freeze dried powder quality.

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1. Introduction

Kefir is a fermented dairy product that fermented with kefir grains or kefir starter cultures with various strains of *Lactobacillus*, *Leuconostoc*, *Lactococcus*, *Acetobacter* and *Saccharomyces unisporus*, *Saccharomyces cerevisiae*, *Saccharomyces exiguus* and *Kluyveromyces marxianus* (Anonymous, 2009). Symbiotic relations between lactic acid bacteria, yeasts and acetic acid bacteria are responsible for production of taste and flavor of kefir product. Major end products of the lactic acid and alcohol fermentation are lactic acid, CO₂, ethanol and other flavoring materials that gave the specific kefir flavor (Beskhova, Simova, Frengova, Simov, & Dimitrov, 2003).

Since kefir is a probiotic product and beneficial to health, consumer's interests are increasing about consuming kefir. There was only plain kefir at first time and then various kefir types as fruit kefir, nonfat kefir starts to put on the market. In this concept, kefir powder is an alternative product to kefir due to long shelf life, easy of use when it desired. Storage and packaging costs of kefir powder less than kefir beverage and there is no refrigeration requirement. There are no reports concerning the characterization and optimization of kefir powder in the literature. In this study, kefir were produced by kefir grains and drying processes were carried with spray and freeze dryers. The effect of spray drying process conditions on survival rates of microorganisms, water activity, dry matter, outlet temperature, pH values, titratable acidity, color values (L, a, b) were investigated and results are compared with freeze drying process results.

Spray drying is the most common technology in the dairy powder industry due to reduced cost, short drying time, effective drying and high moisture removal rate (Tamime, 2009). However, spray drying is a thermal process, there would be some decreases on the viability of microorganisms. Survival of kefir bacteria after drying was affected by many factors, such as spray drying inlet and outlet temperatures, atomization type, direction of airflow and initial microorganism counts (Bielecka & Majkowska, 2000). As well as reduction of the bacteria counts, losses of volatile compounds also occur during drying process (Figueroa, Cervantes, Rodriguez, & Garcia, 2002). Freeze drying is known as the best drying method when sensory properties of powders and viability of bacteria taken into consideration. Nutritional and flavor properties of freeze dried powders are higher than other drying methods. However, high cost of freeze drying application constricts the use of this technology in the industry (Kumar & Mishra, 2004). In this study spray drying and freeze drying techniques were chosen







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among the drying methods due to applicable to the drying of dairy powders and their hygienically process conditions.

During the spray drying of dairy powders it is important to improve survival rate and powder properties with decreasing the inlet temperature. Using optimization methodologies lead to determine of the best production conditions. RSM is one of the most common optimization method used in recent studies. RSM is applicable to design, improvement and formulation of new products and also development of existing products properties (Baş & Boyacı, 2007). For this reason RSM was used to determine the best production conditions of Kefir powder which is a novel dairy powder.

2. Materials and methods

2.1. Materials

Kefir grains were added 3-5% rates to UHT milks and incubated at 24 °C. Incubation was ended at the end of 18 h incubation time. Kefir was packaged and refrigerated until drying.

2.2. Drying processes

Kefir samples were homogenized with Ultra Turrax blender before drying process. Drying process was made in a lab-scale spray dryer (Mini Spray Dryer B-290, Switzerland) equipped with pressure nozzle. Concentration of kefir samples are changes between % 12–13 dry matter contents. Inlet air temperature, feed temperature, and pump rate of spray drying were chosen in the range of 120–180 °C, 4–30 °C and 20–40% (240 L/h–473 L/h) respectively. Experiment design was planned according to the Central Composite Rotatable Design. Airflow rate was fixed 414 L/h and aspiration flow rate was constant at 100%. The dried kefir powders were collected in glass jars and then kept in the room temperature. For freeze drying, kefir was poured in to plastic plates as 1 cm thickness and then frozen at $-40\ ^\circ C$ for a night. Then frozen kefir was lyophilized in an lab-scale lyophilizer (VirTis BenchTop 'K' Manifolt Freeze Dryer, USA) for 48 h. Drying conditions were 50-60 mTorr condenser pressure and -85 °C drying temperature.

2.3. Moisture content

Moisture content of kefir and kefir powder were determined gravimetrically by oven at 103 $^{\circ}$ C until difference of consecutive weighing of samples was constant.

2.4. Titratable acidity and pH

Titratable acidity of kefir and kefir powder was determinated as lactic acid by titration with 0.1 N NaOH. pH values of kefir and kefir powder were measured by a pH meter (Eutech Cyberscan pH 2700, Singapore).

2.5. Water activity

Water activity values of kefir powders were measured by water activity measurement device (Aqualab dew point water activity mater 4 TE, USA).

2.6. Color

The color values (L, a, b) of kefir powder was measured by a colorimeter (Minolta Chroma Meter, CR-400, Osaka, Japan). L value tells how the color of product dark or light and changes between 0 and 100 from darkness to lightness. a values is a scale that

positive numbers indicate red and negative numbers indicate green. *b* values show yellow-blue colors that positive numbers indicate yellow and negative numbers indicates blue.

2.7. Ethanol analysis

Kefir sample (2.5 g) was put in to a beaker and diluted in to 20 mL deionized water and centrifugated for 30 min at 6000 rpm. The supernatant were filtered on Whatman 42 filter paper and kept in the deep freeze until the analysis time. After thawing, samples were filtered on 0.45 μ m membrane filter and injected in to gas chromotragraphy (Shimadzu GC MS-QP2010 Plus, Japan) equipped with ZB-WAX polyetilen glycol colon (30 mm \times 25 mm \times 0.25 μ m, 7HG–G007–17). Operating conditions: flame-ionization detector and injector 200 °C, oven temperature increased 70 °C–120 °C by 4 °C per minute, injection volume 1 μ L, carrier gas nitrogen 100 kPa (Erbas, 2003).

2.8. Microbiological analysis

Ten milliliters of kefir samples and 90 mL dilution solution (8.5 g NaCl₂/L distilled water) were homogenized for 1 min in Stomacher. Dried kefir samples were rehydrated to the initial solid content before homogenization process. Leuconostoc counts were performed on Vancomycin added MRS medium from Merck and incubated at 30 °C for 3 days (Manolopoulou et al., 2003). Lactococci counts were enumerated on M17 medium from Merck at 30 °C for 3 days incubation conditions (Zarate, Belda, Perez, & Cardell, 1997). Lactobacilli counts were carried out on Rogosa Agar from Merck (adjusted pH 5.5 with acetic acid) under anaerobic conditions at 30 °C for 3 days incubation conditions (Ferezza, Fresno, Ribeiro, Tornadijo, & Mansur Furtado, 2004). Yeast counts were enumerated on Yeast Extract Glucose Cloramphenicol agar from Merck and incubated at 30 °C for 3 days (Manolopoulou et al., 2003). Survival rates (N/N_0) were expressed as the quotient of colony forming units per milliliter of kefir before drying (N_0) and kefir powder after drying (N).

2.9. Experimental design and statistically analysis

Response surface method was used for modeling response variables (survival of microorganisms, water activity values, outlet temperature and dry matter content) with in regard to independent parameters (inlet temperature, feed temperature, pump rate).

Central Composite Rotatable Design (CCRD) was used for experimental design. Equally estimation to the all points was carried out by included α values to the design (Montrogomery, 2008). The design is comprised of 20 experiments (Table 1). The results were analyzed nonlinear regression that fit second-order equation.

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} x_{ij} + \varepsilon$$
(1)

$$Y = y - \varepsilon \tag{2}$$

 β_0 is the constant, β_i is the linear coefficient, β_{ii} is the quadratic coefficient and β_{ij} is the interaction coefficients, *y* is the response, *e* is the random error, *Y* is the expected value of response. Lack of fit test was used to evaluate the accordances of mathematical models. F-statistic value for lack of fit test shouldn't be significant for model fitting (Kahyaoglu & Kaya, 2006). The optimum levels of independent values were analyzed by using desirability function method. Design Expert trial version 7.0.0 (Statease Inc., Minneapolis, USA) was used for regression, correlation coefficients, the analysis of variance (ANOVA) and optimization.

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