Optimization of Spray Drying Conditions for Production of Barley Beer Powder

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Abstract: ***The aim of this study was to use response surface approach to optimize spray drying operating parameters for barley beer powder manufacture. As independent variables, the spray drying operating parameters were employed, which included inlet air temperature (100–150°C), outlet air temperature (60–90°C), and carrier agent ratio. Moisture content, water activity, solubility, pH, and product yield were among the responses assessed. The independent variables had a stronger impact on all of the responses studied, according to the statistical analysis. The quadratic model's validity was tested by using the obtained optimum conditions for powder manufacturing. The results showed that 150°C inlet air temperature, 81°C outlet temperature, and 1.7 (w/w) carrier agent were the best spray drying operating parameters for producing high-quality barley beer powder. Barley beer powder with desirable features such as low moisture content, low water activity, high solubility, and high yield might be generated under these ideal conditions.***

Keywords: Optimization, RSM, BBD, Spray drying, β--Cyclodextrin, Beer powder

1. Introduction

Beer is a popular beverage and has its history stretching way back to the earliest of centuries. The word beer has an interesting history. It is sourced from a Latin word '*bibere*' which means to drink. The Latin root is also the foundation of the English word 'beverage' [1]. The quantities of volumes of beer produced makes it one of the most important alcoholic beverages in the world. In 2020 about 1.78 billion hectoliters (one hundred and seventy eight billion of hectoliters) were consumed while in the same year, 234m hl (two hundred and thirty four million hectoliters) of wine were consumed [2, 3]. China is the world's highest producer of beer as well as in consumption [4]. A total of 360 m hl (three hundred and sixty million of hectoliters) were consumed in China representing 20.3% of the world's consumption average [2]. By the year 2017, Tsingtao brewery of China held a market share in terms of beer beverage sales in China by 17.5 %. The others are shared amongst the leading beer beverage companies in China as well as the imported brands [5]. Beer, therefore is a commodity of immense interest in the world.

Beer is made from barley malt, hops, water and fermented by yeast [6]. Adjuncts such as rice, wheat, millet, sorghum are also added for various purposes [7]. Water forms about 90% of the total volume of beer beverage. The complex flavor is made up of different compounds emanating from the raw materials. There are approximately 400 compounds found in beer and all these contribute to the flavor and other properties of the beer [8, 9]. The product is known for its usage both in celebratory and mournful moods. It's also endowed with various health benefits to the moderate consumer. Beer is a polyphenol-rich drink that has lately been linked to a lower risk of cardiovascular disease and cancer. The antioxidant, anti-inflammatory and antifungal properties of its phenolic components are among the ways by which this effect occurs. This is generally as a result of the antioxidant capacity of hop polyphenols [10–17]. Beer is also considered for its prebiotic effects [18–21]. It's rather a different story for some others who by virtue of religion, medical, ethnic and social reasons do not consume beer. The alcohol content, thus has been an impediment to such people. A low or alcohol free beer beverage is therefore encouraged.

Drying has been used to produce food products with lower moisture content and water activities. This process therefore has been explored in producing products such as wine [22], pomegranate [23], milk [24], fruits and vegetables [25]. In these products, their quality properties were analysed. Spray and freeze drying are two drying technologies mostly used. Powders with good quality, low water activity, longer shelf life, and simplicity of transport are produced by drying. The presence of low-molecular-weight sugars and acids with low glass transition temperatures, spray and freeze dried powders have several intrinsic difficulties, such as stickiness. The use of high-molecular-weight carrier molecules such as cyclodextrin can help overcome these issues [26, 27].

RSM (response surface methodology) is a tool for analyzing and optimizing complex food operations. It entails the application of a set of mathematical and statistical processes to investigate the relationship between one or more factors (independent variables) and one or more answers (dependent variables). Some process variables, such as inlet air temperature, outlet temperature, feed concentration ratio and so on, influence the physical qualities of powders produced by spray and freeze drying [28]. As a result, it's important to figure out what the best conditions are for producing high-quality barley beer powder with high physical properties

2. Materials and Methods

2.1 Materials

The barley beer, Tsingtao Beer Light (Tsingtao Brewery Company Limited, Qingdao, China) was bought from Auchan supermarket (Wuxi city, Jiangsu, China). The solid matter content was measured to be 3°Brix by a Brix temperature compensating refractometer (PAL-1 0–32, Atago Co., Japan). The β-Cyclodextrin was purchased from Xi'an Yuhua Biotechnology Co., Ltd. Other chemicals and solvents (HPLC or analytical grades) were obtained from Shanghai Chemical Reagent Co. (Shanghai, China).

2.2 Preparation of the barley beer powders

The microencapsulated samples were made by combining liquid beer (LB) samples with β--Cyclodextrin (β--CD) for encapsulation potential. The treated mixture was mixed and then constantly agitated with a Minimag magnetic stirrer (Benchmark Scientific, New Jersey, USA) at 800 rpm.

2.3 Spray drying of the barley beer powder

For the spray drying process, a pilot plant-scale spray dryer QZR-5 (Linzhou Drying Equipment Co., Ltd., Wuxi, China). The feed mixture in a beaker placed under magnetic agitation at room temperature (25°C) was fed into the drying chamber through a peristaltic pump with a drying airflow rate of 0.35 m^3/min. The spray drying process was carried out using the rotary disc atomizer at a speed of 32000 rpm. The drying air inlet flow rate, feeding speed and feed temperature were kept in conformity with the inlet and outlet air temperatures throughout the experiment. The obtained powders were collected in an insulated glass bottle connected at the end of the cyclone after drying while the powders that remained in the chambers were discarded

2.4 Determination of the physical properties of the powder

2.4.1 The moisture content

The moisture content of beer powder samples was ascertained using an oven-dry method. One gram of sample was carefully measured and dried in a vacuum oven (Binder Vacuum Oven, VDL 53; Binder GmbH, Tuttlingen, Germany) at 70 °C until constant weight was obtained and the analysis was performed in triplicate. The final moisture content was calculated as the ratio of the total weight of moisture loss to the total weight of the powder sample.

2.4.2 The water activity (a_w)

The water activity (a_w) was measured using the water activity meter (AquaLab, Decagon Devices, USA).

2.4.3 The pH

The pH was determined using a pH meter (PHS-3C, INESA, Shanghai, China).

2.4.4 The water solubility index (WSI)

The water solubility index (WSI) was determined. Approximately 1 g of the beer powder was mixed with distilled water (30 mL) and stirred for 30 min using the Minimag magnetic stirrer (Benchmark Scientific, New Jersey, USA). The mixture was centrifuged at 8600 g for 30min (25°C). The supernatants were collected and oven-dried (103 ± 2°C). The WSI was determined as a percentage (%) of the dried supernatant per gram of sample.

2.4.5 The powder yield

The powder yield was evaluated as the percentage of the total solids collected to the total solids provided in the feed suspension.

$$Yield = \frac{beer\ powder(g)}{beer\ powder(g) + carrier\ agent(g)} \times 100 \qquad (1)$$

2.5 Experimental Design

The design of experiments was made using the Box-Behnken Design (BDD) Method (Design-Expert 11) of Response Surface Method (RSM). RSM is a vital tool employed in the analysis and evaluation of experimental data for optimizing conditions in product developments. Its ability to combine multiple variables to achieve favorable responses makes it a good methodology in this research. A 3 factor and 5 responses giving 17 experimental runs were used. The inlet air temperature, output temperature, and carrier agent were the independent variables that influenced the end product's quality. A range of variables must be chosen before an experiment can be designed using RSM. The pre-trials were used to determine the variables' maximum and minimum values. As a result, the inlet air temperature range was set to 100-150°C, the outlet temperature was set to 60-90°C, and the carrier agent ratio was set at 0-2 (w/w) as shown in Table 1. Physical parameters of barley beer powder were measured using response factors such as water activity, moisture content, pH, solubility, and product yield.

A second-order polynomial equation was fitted to correlate each factor to the response. The equation was:

$$Y = \beta_0 + \sum_{(i=1)}^{5} \beta_1 X_i + \sum_{i=1}^{4} \sum_{(j=i+1)}^{5} \beta_{ij} X_i X_j + \sum_{(i=1)}^{5} \beta_{ii} X_i^2 \qquad (2)$$

Where Y = predicted response variable, β_0 = intercepts, β_i = linear regression coefficients, β_{ii} = second-order regression coefficients and β_{ij} = interaction regression coefficients, all estimated by the model and X_i and X_j = values of the independent variables.

The overall Desirability Index (DI) was the basis for selection of the optimized parameters ac-cording to the relation:

$$DI = \left[\prod_{i=1}^{4} d_i\,(y_i)\right]^{1/4} \qquad (3)$$

Where d_i = DI (0 to 1) for the dependent variable and y_i = response.

Table 1: BBD matrix with experimental design and data for the barley beer powder

Run	Drying Factors (Actual and Coded Values)			Responses				
	Inlet Temp (^{o}C)	Outlet Temp (^{o}C)	Carrier Agent (w/w)	Moisture Content (%)	Water Activity	Solubility (%)	pH	Yield (%)
	X_1	X_2	X_3					
1	100 (-1)	60 (-1)	1 (0)	6.23	6.23	91.9	4.13	62.73
2	150 (+1)	75 (0)	2 (+1)	4.91	4.91	92.1	4.12	53.61
3	125 (0)	75 (0)	1 (0)	5.61	5.61	92.9	4.21	60.84
4	100 (-1)	75 (0)	2 (+1)	4.53	4.53	93.2	4.13	55.15
5	125 (0)	75 (0)	1 (0)	5.68	5.68	92.6	4.21	61.28
6	100 (-1)	90 (+1)	1 (0)	3.98	3.98	90.3	4.15	59.06
7	125 (0)	60 (-1)	0 (-1)	7.58	7.58	90.6	4.3	53.79
8	125 (0)	90 (+1)	2 (+1)	3.47	3.47	93.5	4.27	55.62
9	125 (0)	90 (+1)	0 (-1)	5.46	5.46	89.4	4.26	55.23
10	125 (0)	75 (0)	1 (0)	5.54	5.54	92.7	4.25	60.96
11	150 (+1)	90 (+1)	1 (0)	4.76	4.76	91	4.2	61.53
12	100 (-1)	75 (0)	0 (-1)	6.47	6.47	91.9	4.08	57.78
13	125 (0)	60 (-1)	2 (+1)	5.76	5.76	90.4	4.24	52.22
14	150 (+1)	60 (-1)	1 (0)	7.15	7.15	87.7	4.2	52.85
15	125 (0)	75 (0)	1 (0)	5.75	5.75	92.8	4.27	61.19
16	125 (0)	75 (0)	1 (0)	6.04	6.04	92.3	4.25	60.91
17	150 (+1)	75 (0)	0 (-1)	7.13	7.13	89.5	4.2	52.37

Note: All the responses are mean values of three replicates.

2.6 Statistical Analysis

Design Expert Software was used to create the experimental designs as well as the statistical analysis for the optimization (version 11.0.5.0, STAT-EASE, Inc., Minneapolis, USA). MINITAB v18.1 software was used to screen the variables (Minitab Inc., Pennsylvania, USA). The P-test, the lack of fit test, and the coefficient of determination (R^2) were used to assess model accuracy at $p < 0.05$, 0.01, and 0.001. All of the experiments were done in triplicate, and the data was analysed using Microsoft Excel 2016. (Microsoft Corporation, Redmond, WA, USA). Tukeys' test was performed to compare the means at $p < 0.05$, and the values were reported as mean standard deviation.

3. Results and Discussions

Table 1 presents the numerical values of the responses for each experimental run. Table 2 shows the ANOVA findings for each response variable, as well as its significance at the 95 percent confidence level and correlation coefficient. The fitted models were found to be suitable based on the ANOVA data, with significant regression, low residual values, no lack of fit, and satisfactory determination coefficients (R^2) of 0.9884, 0.9926, 0.9947, 0.9515, and 0.9992 for moisture content, water activity, solubility, pH, and yield, respectively. Each response's graphical representation was created as a simultaneous function of the independent factors' significance to the response

Table 2: ANOVA, regression analysis and optimal conditions for barley beer powder

Source	Moisture Content (%)		Water Activity		Solubility (%)		pH		Yield (%)	
	F-value	*p*-value	*F*-value	*p*-value	*F*-value	*p*-value	*F*-value	*p*-value	*F*-value	*p*-value
Model	66.54	<0.0001***	103.65	<0.0001***	146.32	<0.0001***	15.26	0.0008**	989.11	<0.0001***
Linear										
X_1	29.11	0.0010*	8.91	0.0204*	197.58	<0.0001***	15.15	0.0060*	1008.60	<0.0001***
X_2	317.53	<0.0001***	248.54	<0.0001***	52.26	0.0002**	0.0286	0.8704^{NS}	474.55	<0.0001***
X_3	246.27	<0.0001***	140.36	<0.0001***	245.32	<0.0001***	1.83	0.2178^{NS}	32.31	0.0007**
Interactions										
AB	0.1520	0.7082^{NS}	0.4351	0.5306^{NS}	193.63	<0.0001***	0.2291	0.6468^{NS}	1492.02	<0.0001***
AC	0.6079	0.4611^{NS}	48.89	0.0002**	13.63	0.0077*	9.68	0.0170*	146.51	<0.0001***
BC	0.2241	0.6504^{NS}	128.72	<0.0001***	149.11	<0.0001***	2.81	0.1378^{NS}	37.58	0.0005**
Quadratic										
A^2	0.0001	0.9934^{NS}	0.3547	0.5702^{NS}	102.21	<0.0001***	99.39	<0.0001***	90.34	<0.0001***
B^2	4.88	0.629^{NS}	137.39	<0.0001***	333.73	<0.0001***	10.83	0.0133*	258.67	<0.0001***
C^2	0.1764	0.6871^{NS}	200.45	<0.0001***	1.88	0.2132^{NS}	0.1544	0.7061^{NS}	5107.81	<0.0001***
Fitting statistics										
Lack of fit	0.6820	0.6078 ^{NS}	0.2476	0.8596 ^{NS}	0.0314	0.9914 ^{NS}	0.0810	0.9669^{NS}	0.3310	0.8048^{NS}
R^2	0.9884		0.9926		0.9947		0.9515		0.9992	
Adjusted R^2	0.9736		0.9830		0.9879		0.8892		0.9982	
Predicted R	0.9255		0.9715		0.9900		0.8841		0.9965	
Adeq. Precision	30.8967		39.3472		42.9508		13.2625		85.8108	
C. V. %	3.18		1.47		0.1925		0.4969		0.2751	
Standard Dev.	0.1796		0.0038		0.1761		0.0209		0.1599	
Mean	5.65		0.2572		91.46		4.20		57.48	

Optimization equations:

Moisture Content (%) = $5.72 + 0.3425X_{1-}1.13X_{2-}0.9962X_3$

Water Activity = $0.2352-0.0040X_{1-}0.0211X_{2-}0.0159X_{3-}0.0132X_1X_3 + 0.0215X_2X_3 + 0.0216X_2^2 + 0.0262X_3^2$
Solubility = $92.66-0.8750X_1 + 0.4500X_2 + 0.9750X_3 + 1.22X_1X_2 + 0.3250X_1X_3 + 1.07X_2X_{3-}0.8675X_1^{2-}1.57X_2^2$
pH= $4.24 + 0.0288X_1-0.0325X_1X_{3-}0.1015X_1^2 + 0.0335X_2^2$
Yield= $61.04-1.79X_1 + 1.23X_{2-}0.3213X_3 + 3.09X_1X_2 + 0.9675X_1X_3 + 4900X_2X_{3-}0.7405X_1^{2-}1.25X_2^{2-}5.57X_3^2$

Abbreviations: * = $p < 0.05$; ** = $p < 0.01$; *** = $p < 0.001$ and NS = not significant.

3.1 Moisture content

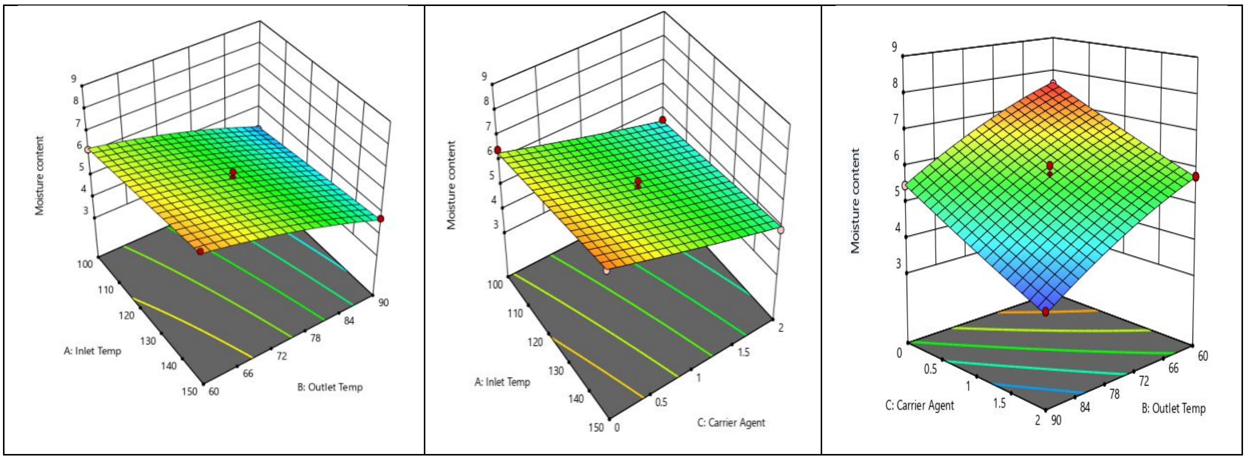

Figure 1: Response surface on the moisture content by inlet air temperature, outlet air temperature and carrier agent

Moisture content is a vital property associated with the overall quality and shelf life of the powder [31]. The flavour, aroma, and colour of very low moisture food product may disappear, while a high moisture product can become sticky and may not be suitable for long-term storage. The moisture content of barley beer powder prepared under different experimental conditions was in the range of 3.47 to 7.58% dry basis (Table 1). Figure 1 represents the effect of inlet air temperature, outlet air temperature and carrier agent ratio on the moisture content of barley beer powder. The moisture content decreased with an increase in inlet air temperature and outlet air temperature as evident from Figure 1A. The coefficients of the second order terms variables indicated that the moisture content decreased with the increase in outlet air temperature as well as carrier agent ratio (figure 1B and 1C). The moisture content of powder decreased from 6.28% to 4.77% (d. b.) when inlet air temperature increased from 101.14°C to 149.47°C at constant carrier agent ratio of 1 (w/w) (Fig 1a). The moisture content (4.8% d. b.) of the powder was found lowest at 150°C inlet air temperature, 81°C and 1.7 (w/w) carrier agent ratio whereas, highest moisture content (7.58% d. b.) was observed at 125°C inlet air temperature, 60°C outlet air temperature and 0 carrier agent. Outlet air temperature and carrier agent ratio negatively influenced the moisture content of powder (Eq.4). It can also be observed from the Table 2 that the inlet air temperature, outlet air temperature and carrier agent ratio significantly ($p \leq 0.01$) affected the moisture content of the powder. Higher inlet air temperature creates a greater temperature gradient between atomized feed and drying air, thereby providing greater driving force for removal of moisture. In the past also, some researchers have reported lower moisture content in the product at high inlet air temperature of the spray dryer [32–34]. This may be due to the generation of high hot air during drying, which might have trapped the moist air that was found in the fed product and it might have reduced the moisture content to a greater extent [28]. Further, the higher carrier agent ratio helps in microencapsulation with increased surface area and thereby resulting in lower moisture content in the final product. It is evident that by regulating inlet air temperature and carrier agent as well as outlet air temperature during spray drying, the moisture content in the powder can be controlled. The relationship between moisture content and independent variables are mentioned as below through Equation (4).

Moisture Content (% db) = $+5.72 + 0.3425X_{1-}1.13X_{2-}0.9962X_3$ (4)
where, X_1= inlet air temperature (^{0}C), X_2 = outlet air temperature (^{0}C),, X_3 = carrier agent ratio (w/w)

3.2 Water activity

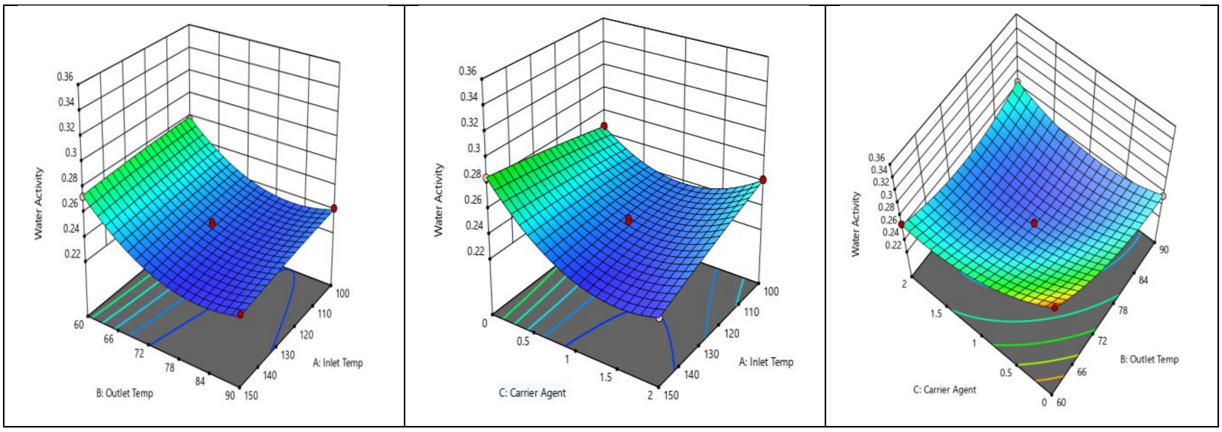

Figure 2: Response surface on the water activity by inlet air temperature, outlet air temperature and carrier agent

The minimum water activity value of barley beer powder were determined as 0.227 at 150 ^{o}C inlet air temperature, 75 ^{o}C outlet temperature and 2 (w/w) carrier agent ratio. According to [35]the minimum value of water activity of spray dried water melon powder was 0.20 at inlet air temperature of 145 ^{o}C and outlet temperature of 95.4 ^{o}C. The dried products with water activity values under 0.60 considered as stable for browning and hydrolytical reactions, lipid oxidation and enzymatic reactions [36, 37]. According to results we can say barley beer powder is a safety product against detrimental chemical and microbiological reactions. Water activity values of barley beer powders were significantly affected by linear effect of the outlet air temperature and carrier agent ratio as well as the interaction between the outlet air temperature and carrier agent ratio. Water activity increases with decreasing of the inlet temperature (Table 1). Figure 2c shows elliptical contour plot that indicate there is an interaction between outlet air temperature and carrier agent ratio. It can be observed from figure that an increase in outlet air temperature cause decrease in water activity of barley beer powder samples.

The relationship between water activity and independent variables are mentioned as below through Equation (5).
Water Activity = $0.2352\text{-}0.0040X_{1\text{-}}0.0211X_{2\text{-}}0.0159X_{3\text{-}}0.0132X_1X_3 + 0.0215X_2X_3 + 0.0216X_2^2 + 0.0262X_3^2$ ……. (5)
where, X_1= inlet air temperature (^{0}C), X_2 = outlet air temperature (^{0}C),, X_3 = carrier agent ratio (w/w)

3.3 Solubility

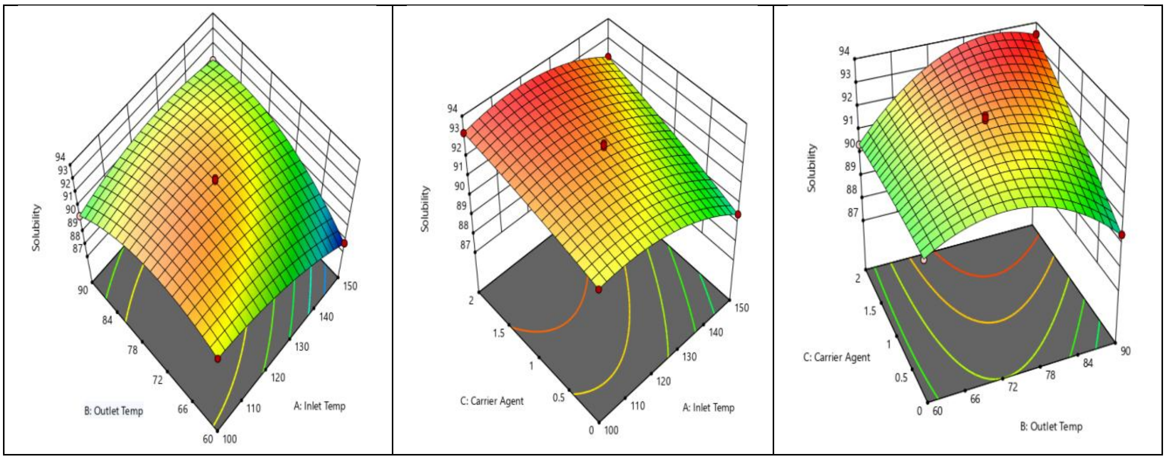

Figure 3: Response surface on the solubility by inlet air temperature, outlet air temperature and carrier agent

From Equation (6), it is clear that a decrease in the carrier agent ratio as well as a marginal increase in the inlet air temperature and outllet air temperature increased the solubility of the barley beer powder. This may be due to the presence of less amount of insoluble residue and formation of very few lumps as a result of the use of the drying process. Increase in solubility was reported during drying of persimmon pulp [38] and soluble sage [39].

The relationship between moisture content and independent variables are mentioned as below through Equation (6).
Solubility = $92.66\text{-}0.8750X_1 + 0.4500X_2 + 0.9750X_3 + 1.22X_1X_2 + 0.3250X_1X_3 + 1.07X_2X_{3\text{-}}0.8675X_1^{2\text{-}}1.57X_2^2$ ……. . (6)
where, X_1= inlet air temperature (^{0}C), X_2 = outlet air temperature (^{0}C),, X_3 = carrier agent ratio (w/w)

3.4 pH

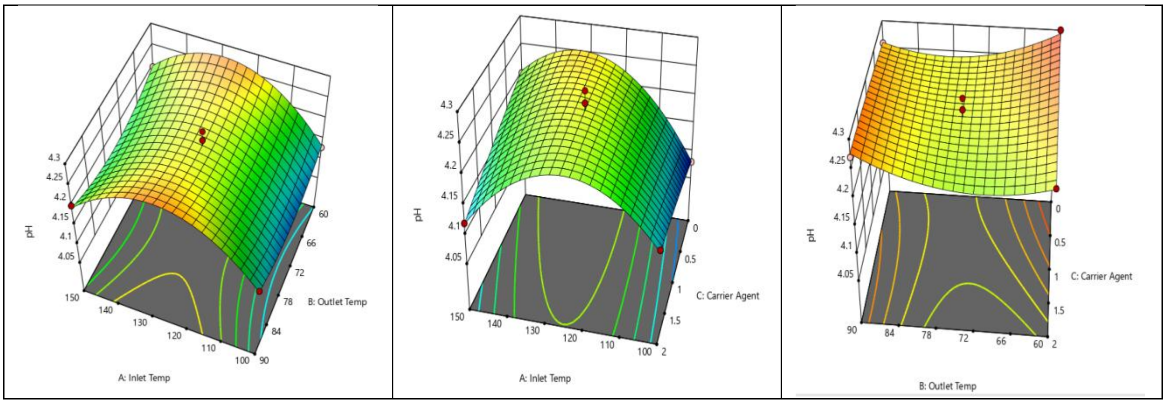

Figure 4: Response surface on the pH by inlet air temperature, outlet air temperature and carrier agent

The lowest pH value of barley beer powder was determined as 4.08 at the 100 °C inlet air temperature, 75 °C outlet air temperature and 0 carrier agent ratio. From the table 2, the inlet air temperature had significant (*p*>0.01) effect on the pH of the barley beer powder. Results of figure 4 a-c suggest that conditions have little effect on pH values of barley beer powder

$pH = 4.24 + 0.0288X_1 - 0.0325X_1X_3 - 0.1015X_1^2 + 0.0335X_2^2$ ……. (7)
where, X_1 = inlet air temperature (^0C), X_2 = outlet air temperature (^0C),, X_3 = carrier agent ratio (w/w)

3.5 Product yield

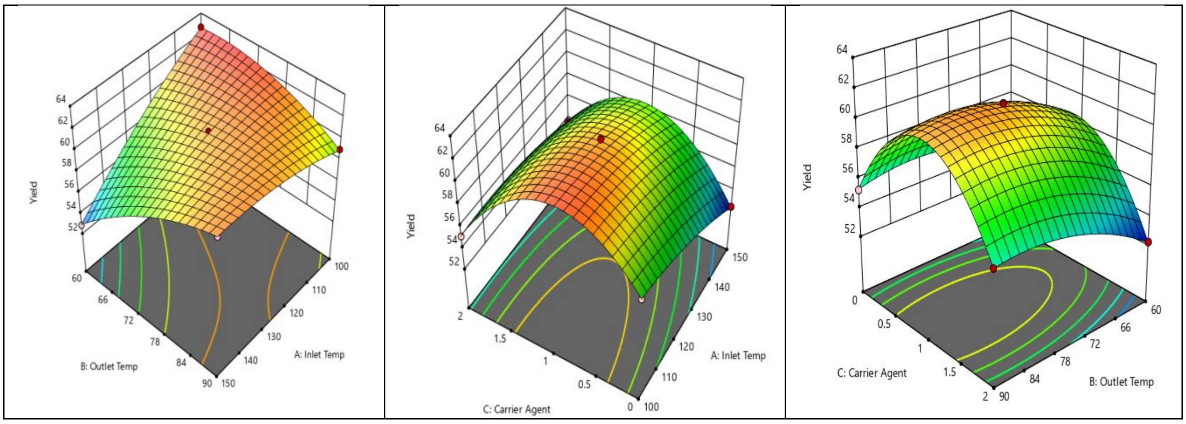

Figure 5: Response surface on the product yield by inlet air temperature, outlet air temperature and carrier agent

In the literature it has been described how the product yield is affected by the inlet air temperature and carrier agent ratio. It has been reported that higher product yields resulted from higher drying temperatures in the case of spray dried pomegranate powder [33]. These variations could be related to different feed compositions and process conditions during spray drying. A low inlet temperature will not provide enough heat to dry the product, and wet powders will easily stick to the chamber wall (generally, only those powders collected in the container are regarded as effective), so the drying yield will decrease [40]. The drying yield values ranged between 44.4 and 57.3%. These results are similar to those obtained by [41–44].

$Yield = 61.04 - 1.79X_1 + 1.23X_2 - 0.3213X_3 + 3.09X_1X_2 + 0.9675X_1X_3 + 4900X_2X_3 - 0.7405X_1^2 - 1.25X_2^2 - 5.57X_3^2$ ……… (8)
where, X_1 = inlet air temperature (^0C), X_2 = outlet air temperature (^0C),, X_3 = carrier agent ratio (w/w)

3.6 Optimization of beer powder processing

Table 3: Criteria and outputs of the numerical optimization of the responses for barley beer

Variables	Goal	Lower Limit	Upper Limit	Importance
Inlet Temp	maximize	100	150	4
Outlet Temp	minimize	60	90	4
Carrier Agent	is in range	0	2	4
Solubility	maximize	87.7	93.5	4
Moisture content	minimize	3.47	7.58	4
pH	minimize	4.08	4.3	4
Yield	maximize	52.22	62.73	4
Water Activity	minimize	0.227	0.343	4

Numeric and graphic optimizations were carried out for the process parameters of the barley beer powder. The desired goals for each variable and response were chosen as summarized in Table 3. The limit for each variable was narrowed down to obtain an optimal region. Each goal was chosen to be as follows: to minimize and also to maximize based on the moisture content, water activity, solubility, pH and product yield of the developed product, because at this desired level only the free flowing characteristics of the barley beer powder can be obtained i. e. by using low moisture content level; improved physical properties can be achieved by maximizing the levels of solubility.

The software generated optimum conditions of independent variables with the predicted values of responses; in the inlet air temperature at maximum level 150 °C, minimum outlet air temperature of 81°C and minimum carrier agent ratio of 1.7 (w/w) for achieving the minimum moisture content 4.80%, minimum water activity 0.224, minimum pH 4.14, maximum solubility 92.53%, maximum product yield 57.698 %. For validation of results, the barley beer was spray dried using the optimized conditions and analyzed for various responses.

4. Conclusions

Seventeen different runs according to the BBD were used to study the physical parameters of barley beer powder at various levels of inlet air temperature, outlet air temperature and carrier agent ratio. The response surface methodology was used to optimize the processing conditions using moisture content, water activity, solubility, pH and product yield as responses. The models for moisture content, moisture content, water activity, solubility, pH and product yield were statistically significant. By superimposing the graphs, an optimum spray-drying process i. e. inlet air temperature level of 150 °C, outlet air temperature 81 °C and carrier agent ratio 1.7 (w/w) for drying barley beer was recommended with predicted responses close to experimental values. The barley beer powder sample developed using the optimized spray-drying process were stored further tests.

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