Original Article

Process optimization of spray-dried buffalo milk *Bubalus bubalis bubalis* (Linnaeus, 1758) using response surface methodology

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ABSTRACT

Buffalo milk has higher fat and solids-not-fat contents, consisting mainly of proteins, carbohydrates, and minerals, making it more nutritious than cow's milk. However, its composition also makes the product more perishable. Thus, preservation techniques like spray drying must be employed to extend its shelf life and facilitate efficient transportation and distribution. Spray-drying conditions for cow's milk have already been established. However, because of differences in physicochemical properties and composition, these conditions may not be applicable to buffalo milk. Therefore, optimizing the processing conditions for buffalo milk is necessary. This study aimed to optimize the spray-drying conditions for buffalo milk to produce an acceptable milk powder. Response surface methodology was used to evaluate the effects of three key variables: maltodextrin (10-20%w/w), feed pressure (8-12Hz), and inlet temperature (190-210°C). The goal was to determine their effects on the response variables, including moisture content, water activity, hygroscopic moisture, and insolubility index. The optimal spray-drying conditions were obtained at a maltodextrin concentration of 20% (w/w), a feed pressure of 12 Hz, and an inlet temperature of 210°C. These conditions produced a milk powder with a moisture content of 1.98%(w/w), a water activity of 0.227a_w, a hygroscopic moisture content of 16.04g/100 g solids, and an insolubility index of 0.67. The model used to predict the responses was reliable because the experimental outcomes closely matched the predicted values. The spray-dried buffalo milk produced met the European Pharmacopoeia classification criteria for powdered milk and may serve as an ingredient in various milk-based products, such as yogurt.

Keywords: maltodextrin, reconstitution, milk powder, milk preservation, milk processing, milk quality and safety.

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INTRODUCTION

Milk is the lacteal secretion of the mammary glands of female mammals, which serves as the primary source of nutrition for the young (Walstra et al. 2005). Cow's milk is the major source of milk and dairy products in developed countries, especially in the Western world (Park 2009). However, the milk of some animals, such as buffaloes, goats, and sheep, is also used for human consumption, either as such or in the form of a range of dairy products (Walstra et al. 2005). Buffalo *Bubalus bubalis bubalis* (Linnaeus, 1758) milk is more nutritious, as it is richer in fat, lactose, protein, total solids, calcium, magnesium, inorganic phosphate, and vitamins than cow's milk (Fundora et al. 2001; Ahmad et al. 2008). Its rich composition makes it an excellent ingredient for the development of a wide variety of dairy food products.

In tropical countries like the Philippines, the warm climate favors buffalo rearing over cattle rearing. However, the Philippines imports 99% of its milk supply requirements. To boost the country's milk supply, the Philippine Carabao Center (PCC) and the National Dairy Authority of the Philippine government have developed programs to assist small dairy farmers in raising buffaloes intended for milk production. As a result, buffalo milk production has significantly increased, which could help alleviate the malnutrition and address the country's milk supply shortage. However, due to the milk's perishable nature, transporting locally produced buffalo milk to nearby regions poses a significant challenge for farmers, considering the cost of maintaining it at low temperatures. Therefore, preservation techniques must be developed to help local farmers maintain milk quality and encourage greater engagement in buffalo milk production, thereby increasing the shelf life, value, and marketability of locally produced buffalo milk.

Spray drying is the most preferred commercial method for producing milk powder due to its ability to preserve the product quality at a relatively low cost (Liu 1997). The resulting dried milk is more stable, has a longer shelf life, requires less storage space, and incurs lower transportation costs, thereby allowing for wider distribution. Although spray drying is a well-established method for milk powder production, research has primarily focused on cows' milk and specific buffalo milk derivatives, such as protein co-precipitates (Gawande et al. 2022). Consequently, there is a significant data gap concerning spray-dried whole buffalo milk in the Philippine context. The high fat content of buffalo milk often leads to particle stickiness and wall deposition during the drying process, reducing product recovery and creates operational challenges (Schuck et al. 2008; Bhandari et al. 2023). To address these challenges, a carrier agent is commonly used. Maltodextrin is considered by many researchers to be an effective carrier agent for spray drying various food products because of its low viscosity at high solids content, high solubility, and low cost. However, excessive maltodextrin addition can

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negatively affect the nutritional composition and properties of the milk powder. Thus, its addition must be optimized.

Spray-drying conditions for cow's milk have already been established and characterized using various parameters, such as inlet air temperature, feed rate, atomizer speed, outlet air temperature, product temperature, and thermal and evaporative efficiencies (Perez-Munor and Flores 1997). Schuck et al. (2012) emphasized the interaction between outlet air temperature and the resulting powder's functional properties, while Bhandari et al. (2023) highlighted the importance of monitoring glass transition temperatures and evaporative efficiencies to prevent stickiness during the production of high-solids dairy powders. However, because of differences in composition and physicochemical properties, these factors might not apply to buffalo milk. Thus, to produce powdered buffalo milk with desirable quality and processing characteristics, the processing conditions for spray-dried buffalo milk must be optimized, taking cow's milk quality standards as a baseline. The main goal of this research was to establish and verify the optimized spray drying conditions for buffalo's milk to produce an acceptable milk powder. Specifically, the study aimed to determine the physical characteristics of the resulting milk powder, namely, water activity, hygroscopicity, moisture content, and solubility, which are important indicators of product quality and stability. It also aimed to evaluate the effects of varying spray drying conditions on the physical characteristics of the resulting milk powder.

METHODS

Material Preparation

Raw buffalo milk (Italian and crossbreed of native and Italian buffaloes) was sourced from the Rosario Dairy Raisers' Association in Rosario Batangas, a cooperative supported and assisted by the Philippine Carabao Center-University of the Philippines Los Baños. Other materials, such as food-grade maltodextrin (DE value of 10-12) and soy lecithin as an emulsifier, were secured from a food ingredient supplier. The buffalo milk was spray-dried using an industrial-scale gas-powered, contra-flow centrifugal spray dryer (Flexi Mach®), which operated at an atomizer speed of 24,000 rpm, a water evaporation rate of 15 kg/h, and a total feed capacity of 20 L/h. The machine was operated at varying feed pressures and inlet and outlet air temperatures.

Spray Drying of Buffalo Milk

About 10 L of fresh buffalo milk was filtered using cheesecloth to remove impurities. The milk was then pasteurized at 72°C for 5 min in a double boiler and immediately cooled to about 40°C to prepare the milk slurry formulation. This protocol followed the thermal processing guidelines of the International Dairy Foods Association (IDFA 2026) and was consistent with the safety standards established by the USFDA (2019). The

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milk slurry was prepared by adding maltodextrin (DE 10-12) as a carrier agent at a concentration of 10%, 15%, and 20% (w/w). Soy lecithin was incorporated at a constant concentration of 0.1% (w/w) as an emulsifier. The slurry was thoroughly mixed using a BY-767A Drink machine high-performance blender at low speed for 30 s before it was fed into the spray dryer (3.0 × 1.5 × 3.5 m, contra-flow, Flexi Mach® Industrial type Gas powered LPG-15 High speed centrifugal spray dryer) under different processing conditions. The resulting powdered milk produced was packed in a laminated foil pouch, sealed, and stored in a cool dry place until further analysis. Approximately 2 kg of powder was produced per 10 L of milk, and less than 500 g was reconstituted for the various analyses.

Research Design

The processing conditions for spray-dried buffalo milk were optimized using response surface methodology (RSM). This method was selected to determine the individual and interaction effects of the processing variables on the powder's physical properties while minimizing the experimental workload. The optimization process utilized a central composite rotatable design employing three independent variables (factors) namely, maltodextrin concentration, feed pressure, and inlet air temperature at five levels ($-\alpha$, -1, 0, +1, $+\alpha$). The design included has 8 cube (factorial) points, 6 axial (star) points, and 6 center points with a total of 20 experimental runs. The specific levels for maltodextrin concentration, feed pressure, and inlet air temperature are detailed in Table 1.

The independent variables (maltodextrin concentration, inlet air temperature, and feed pressure) were selected based on their documented impact on dairy powder characteristics (Kim et al. 2009: Tan et al. 2011) and the specific operational parameters of the spray-drying system. Due to the high fat content of buffalo milk, which facilitates wall sticking and reduces product recovery, maltodextrin was utilized as a carrier agent to modify the feed composition and improve drying yields. Inlet air temperature was selected as a factor due to its significant influence on the moisture content and water activity of the final product. Furthermore, while previous studies highlighted the importance of outlet temperature, the spray dryer used in this study utilized feed pressure as a primary control variable. This allowed for the indirect regulation of the outlet temperature and droplet size distribution, which are critical to the solubility and hygroscopicity of the reconstituted powder.

The responses considered in the optimization of the processing conditions were water activity (a_w), moisture content, solubility/insolubility index, and hygroscopicity. Moisture content, water activity, and solubility of the powder were required to meet regulatory standards for milk powder. Moisture content and water activity can be used in determining the shelf life of the powder. A powder must have good solubility when reconstituted

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to maximize its functional properties. Solubility also reveals how the drying process affects the functional behavior of soluble proteins in milk. According to Anandharamakrishnan et al. (2008), the solubility of spray-dried dairy products depends on the structural state of their proteins, which can either be in native, denatured or aggregated form. Hygroscopicity, on the other hand, can be used to determine the properties of the powder during storage. Powder that absorbs moisture tends to cake during storage.

Table 1. Independent variables and their levels used in the central composite design for spray drying of buffalo milk.

Independent variables	-α (-1.68)	Low (-1)	Center (0)	High (+1)	+α (+1.68)
Maltodextrin level (%w/w)	6.59	10	15	20	23.41
Inlet Temperature (°C)	183.00	190	200	210	216.00
Feed Pressure (Hz)	6.64	8	10	12	13.36

Analysis of Physical Properties

The moisture content, water activity (a_w), solubility/insolubility index, and hygroscopicity of the product were the parameters considered as responses for the optimization experiment.

Moisture content. The oven-drying method was used for the determination of the moisture content of the sample as described in AOAC Official Method 990.20 (AOAC 2005). Approximately 1 g of sample was weighed in a previously tared crucible and placed in an oven at 100 ± 5°C for at least 5 h. The sample was removed from the oven, placed in a desiccator, and allowed to cool to room temperature before weighing. Crucibles with samples were placed back in the oven for another 30 min, cooled in a desiccator, and reweighed until constant weight was achieved (the difference between the two preceding readings did not exceed 0.001 g). The moisture content (MC, %) was computed using the formula:

$$\%MC = \frac{loss\ in\ weight}{weight\ of\ sample} \times 100 \quad (1)$$

Water activity. Water activity (a_w) of the sample was measured at 24°C using a Novasina LabSwift- a_w meter at the Food Analysis laboratory of the Institute of Food Science and Technology, UPLB, College, Laguna. About 2 g of the spray-dried milk powder was sampled, and the analyses were performed in triplicate.

Solubility index. To determine the reconstitution ability of the powdered milk produced, its solubility/insolubility index was determined using the method described by Sulieman et al. (2014). About 10 g of the spray-dried milk was mixed with 100 mL of distilled water at approximately 24°C at high speed for 90 s. The milk was then left for 15 min to allow the foam to dissipate and was stirred with a spatula. About 50 mL of the milk was transferred to a graduated 50 mL centrifuge tube and it was centrifuged for 5 min at 500 to 1000 rpm.

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The sediment-free liquid was removed, and the precipitate was washed with distilled water and centrifuged again for another 5 min. The solubility index, expressed in mL, was determined by collecting and measuring the sediments after centrifugation.

Hygroscopicity. Hygroscopicity was measured based on the method described by Cai and Corke (2000) with slight modifications. A total of 1 g of the sample was placed in a container at 25°C and equilibrated with a saturated NaCl solution (75.29% relative humidity). Samples were weighed after one week, and hygroscopicity was expressed as the weight (g) of adsorbed moisture per 100 g of dry matter. It was computed using the formula:

$$Hygroscopicity\left(\frac{g}{100g}\right) = \frac{final\ weight - initial\ weight}{initial\ weight \times (100 - \frac{moisture}{100})} \quad (2)$$

Statistical Analysis and Determination of Optimum Conditions.

Data from the experiment were subjected to response surface regression analysis following the method of Anderson and Whitcomb (2016) using Design Expert® 11 (Stat-Ease Inc., Minneapolis) statistical software to determine the effects of independent variables on the product's moisture content, water activity, hygroscopicity, and solubility. Sequential model fitting was performed to evaluate linear, two-factor interaction, and quadratic polynomial models. Model selection was based on the sequential model sum of squares (*P*-value), lack-of-fit test, and coefficient of determination. Analysis of variance (ANOVA) was conducted at a 5% level of significance to assess the adequacy of the selected models and the statistical significance of each term. To optimize predictive accuracy, non-significant model terms were systematically removed through model reduction to yield refined prediction models. Response surface and contour plots were created for all analyses to illustrate the effects of the variables on the responses studied.

The desirability function of Design Expert® 11 was used to locate the optimum processing condition. The criteria used to select the optimum conditions were a minimum insolubility index, low hygroscopicity, maximum water activity, and a moisture content within the range of 1.35-4.78%. The desirability function is a geometric mean of all transformed responses calculated using the formula:

$$D(x) = (d_1 \cdot d_2 \cdots d_n)^{1/n} = \left(\prod_{i=1}^{n} di\right)^{1/n} \quad (3)$$

Model Verification

To verify the predictive ability and accuracy of the model, a verification experiment was carried out. Buffalo milk was spray-dried under the predicted optimal conditions. The water activity, moisture content, hygroscopic moisture, and insolubility index of the spray-dried buffalo milk were predicted using the following empirical model equations:

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$$a_w = 8.49128 - 0.125824A - 0.072562B + 0.006718C + 0.000533\,AB + 0.000656\,A^2 + 0.000157B^2 \quad (4)$$

$$\%MC = 10.1664 - 0.0448132A - 0.00857976B - 0.858483\text{C} - 0.000617185\text{AB} - 0.00331774\text{AC} + 0.0100338\text{BC} + 0.00594413A^2 - 0.000258338B^2 - 0.0499379C^2 \quad (5)$$

$$Solubility(Square\ root) = 21.8097 + 0.122636A + 0.121317B + 1.73874C - 0.00108986AB - 0.00658697AC - 0.00864071\text{I}BC + 0.00457899A^2 \quad (6)$$

$$(\%Hygroscopic\ Moisture)^1 = -562.992 + 0.930291A + 5.39212B + 6.45853C - 0.00139905AB + 0.0490521AC - 0.0314552BC - 0.0333982\,A^2 - 0.0126619B^2 - 0.0495295C^2 \quad (7)$$

Where:

$A = Maltodextrin\ level\ \left(\%\frac{w}{v}\right)$

$B = Inlet\ temperature\ (°C)$

$C = Feed\ Pressure(Hz)$

The observed experimental means were compared with the predicted values generated by the software (Equations 4-7). A two-sided confirmation test was then performed. The model was considered validated if the experimental mean fell within the 95% prediction interval (PI).

RESULTS

Physical Properties of Spray-dried Buffalo Milk

Water activity. The water activity of the buffalo milk powder produced by spray drying under different conditions ranged from 0.130 to 0.312 (Table 2). An inverse relationship was observed between water activity and the inlet drying temperature and maltodextrin level of the spray dried milk powder. Conversely, the water activity was directly proportional to the feed pressure used during spray drying. Higher feed pressure resulted in spray-dried milk with higher water activity. The curved contour lines at the base of Figure 1A indicate notable quadratic and interaction effect between maltodextrin levels and inlet temperature.

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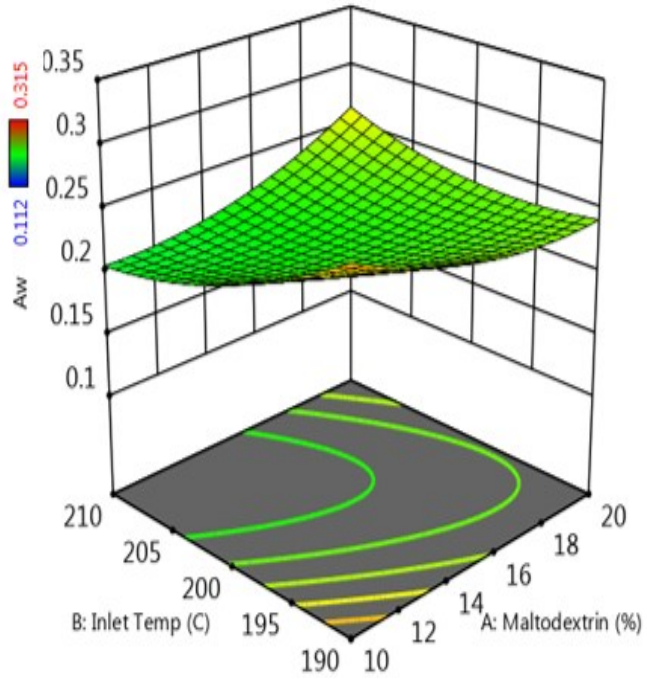
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Table 2. Mean (n=3) values of a_w, moisture content, hygroscopic moisture, and solubility index of powdered buffalo milk produced at different spray-drying conditions.

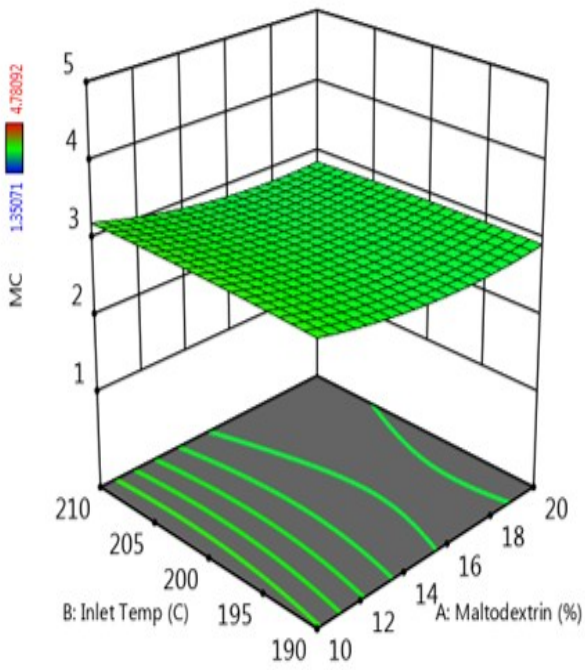
Maltodextrin level (% w/w)	Inlet Temp (°C)	Feed Pressure (Hz)	a_w	MC (%wb)	Hygroscopic Moisture (g/100g solids)	Solubility Index (mL)
10.00	190	8.00	0.235	2.8857	13.4951	1.500
10.00	190	12.00	0.268	3.4254	12.4682	1.833
10.00	210	8.00	0.130	1.9628	14.7227	4.000
10.00	210	12.00	0.203	3.7328	10.7356	2.333
20.00	190	8.00	0.217	2.4667	12.9542	1.100
20.00	190	12.00	0.253	3.3013	13.4456	1.000
20.00	210	8.00	0.256	1.8480	13.4582	2.867
20.00	210	12.00	0.256	3.0577	11.8769	0.750
6.59	200	10.00	0.298	3.6183	10.3045	2.133
23.41	200	10.00	0.232	3.2835	15.7641	1.917
15.00	183	10.00	0.289	3.2694	11.6803	0.733
15.00	216	10.00	0.237	2.7338	11.9489	2.233
15.00	200	6.64	0.193	2.8553	13.7300	1.933
15.00	200	13.36	0.217	1.8864	15.9411	0.467
15.00	200	10.00	0.312	3.0774	16.0726	1.067
15.00	200	10.00	0.149	2.7993	17.6314	1.000
15.00	200	10.00	0.137	1.7131	18.7668	2.500
15.00	200	10.00	0.185	2.7456	13.8526	0.917
15.00	200	10.00	0.219	3.1453	13.1611	0.783
15.00	200	10.00	0.250	4.0461	12.1402	2.500

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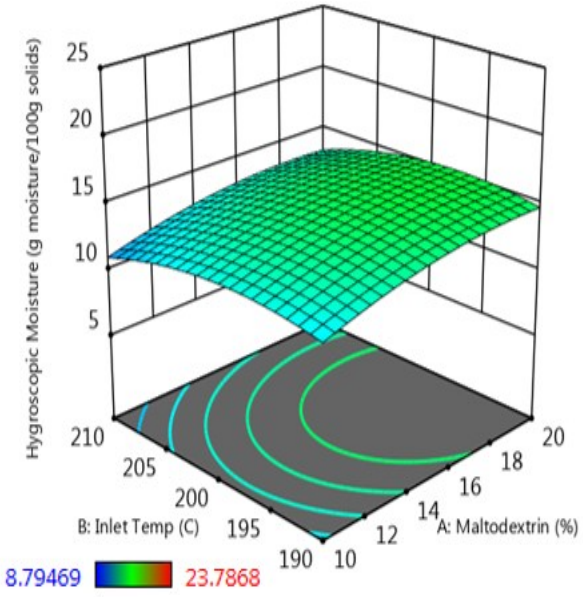
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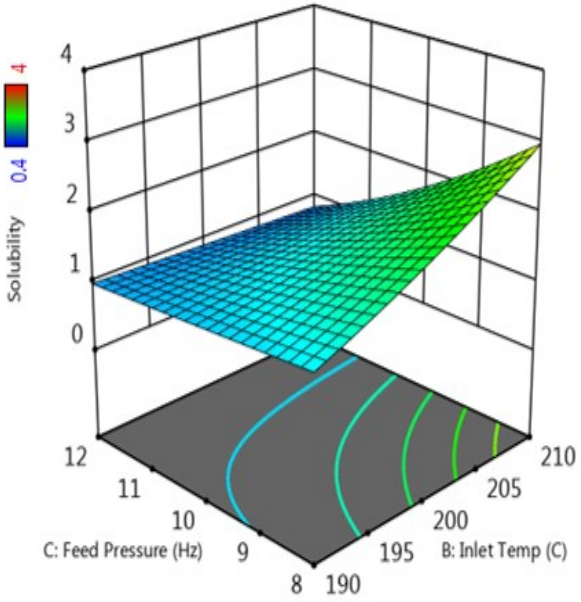
A. Water activity



B. Moisture Content



C. Hygroscopicity



D. Solubility

Figure 1. Response surface plots of the (A) water activity, (B) moisture content, and (C) hygroscopicity at 12 Hz feed pressure, and (D) solubility index at 20% maltodextrin.

Moisture content. The moisture content of the buffalo milk powder produced under varying spray-drying conditions ranged from 1.71% to 4.04% with an average of 2.89% (Table 2). Maltodextrin levels, inlet drying temperature, and feed pressure had no significant effect on the moisture content of the powdered milk produced (Tables 3 and 4; Figure 1B).

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Table 3. Summary of the ANOVA results for each response (A – Maltodextrin level; B- Inlet temperature; C- feed pressure; $P < 0.05$- significant; $P < 0.01$- highly significant).

Source*	df	a_w		Moisture Content		Hygroscopic Moisture		Solubility	
		Sum of squares	p-value	Sum of squares	p-value	Sum of squares	p-value	Sum of squares	p-value
Model	9	0.0587	0.0015	7.9600	0.1410	128.0000	0.0114	4.8000	< 0.0001
A	1	0.0003	0.6987	0.5467	0.3246	19.8100	0.0569	0.5820	0.0005
B	1	0.0102	0.0211	1.7800	0.0788	0.2746	0.8194	1.4100	< 0.0001
C	1	0.0074	0.0479	1.6300	0.0920	1.2500	0.6265	1.2700	< 0.0001
AB	1	0.0171	0.0033	0.0229	0.8396	0.1174	0.8813	0.0713	0.2016
AC	1	0.0019	0.3123	0.0264	0.8278	5.7700	0.2976	0.1041	0.1241
BC	1	6.000E-06	0.9542	0.9665	0.1919	9.5000	0.1831	0.7168	0.0001
A^2	1	0.0109	0.0174	0.9547	0.1946	30.1400	0.0199	0.5719	0.0006
B^2	1	0.0100	0.0222	0.0289	0.8202	69.3100	0.0006	0.0325	0.3861
C^2	1	0.0012	0.4106	1.7300	0.0833	1.7000	0.5708	0.0199	0.4971
Residual	50	0.0899		27.6200		260.6100		2.1300	
Lack of fit	5	0.0213	0.0275	7.3500	0.0134	46.4700	0.1042	0.4653	0.0430
Pure error	45	0.0685		20.2700		214.1300		1.6600	
Total									
R^2		0.3724		0.2236		0.3294		0.6927	
Adj-R^2		0.3143		0.0839		0.2087		0.6373	
Adeq.		8.5027		4.9932		4.6144		13.310	
Precision									
PRESS		0.1073		38.5900		326.5500		2.6500	
C.V		18.3300		25.9000		16.6600		16.5000	

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Table 4. Summary of the ANOVA results for each response variable and coefficient of prediction model after removing the insignificant factors from the model (A – Maltodextrin level; B- Inlet temperature; C- feed pressure; $P < 0.05$-significant; $P < 0.01$-highly significant).

Source	a_w		Moisture Content		Hygroscopic Moisture		Solubility	
	Coefficient estimate	p-value	Coefficient estimate	p-value	Coefficient estimate	p-value	Coefficient estimate	p-value
Model	0.0556	< 0.0002	2.9200	0.1410	128.0000	0.0114	4.7400	< 0.0001
A	0.0026	0.6951	-0.1155	0.3246	19.8100	0.0569	0.5820	0.0005
B	-0.0158	0.0195	-0.2083	0.0788	0.2746	0.8194	1.4100	< 0.0001
C	0.0134	0.0450	0.1995	0.0920	1.2500	0.6265	1.2700	< 0.0001
AB	0.0267	0.0029	-0.0309	0.8396	0.1174	0.8813	0.0713	0.1986
AC			-0.0332	0.8278	5.7700	0.2976	0.1041	0.1216
BC			0.2007	0.1919	9.5000	0.1831	0.7168	0.0001
A^2	0.0164	0.0125	0.1486	0.1946	30.1400	0.0199	0.5769	0.0005
B^2	0.0157	0.0162	-0.0258	0.8202	69.3100	0.0006		
C^2			-0.1998	0.0833	1.7000	0.5708		
Lack of fit		0.0671		0.0134	46.4700	0.1042	0.5233	0.0728
R^2	0.3743		0.2236		0.3294		0.6843	
Adj-R^2	0.3034		0.0839		0.2808		0.6418	
Adeq. Precision	7.8102		4.9932		4.6144		14.2820	
PRESS	0.1104		38.5900		326.5500		2.5700	
C.V	18.4700		25.9000		16.6600		16.4000	

Hygroscopic moisture. The hygroscopicity of buffalo milk powder produced using varying spray-drying conditions ranged from 10.30 to 18.77 g/100 g solids (Table 2). An increase in inlet drying temperature resulted in an increase in the hygroscopic moisture absorbed by the milk powder. The convex curvature along temperature axis in Figure 1C indicates a strong quadratic response to inlet temperature. In contrast, an increase in the amount of maltodextrin used resulted in a decrease in the hygroscopicity of spray-dried buffalo milk. The interaction among the three independent variables had no significant effect on the product's hygroscopicity.

Insolubility index (ISi). The ISi values, which measure how well the powder dissolves in water, ranged from 0.467mL to 4mL at 24°C (Table 2). The interaction between inlet temperature and feed pressure affected the insolubility index of the spray-dried buffalo milk (Tables 3 and 4). Insolubility increased when a higher inlet

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drying temperature and lower feed pressure were used during spray drying (Figure 1D). However, using a higher of maltodextrin concentration (20%) resulted in a lower ISi value, indicating better water solubility.

Optimum Processing Conditions and Model Verification

The optimum spray-drying conditions were identified by maximizing the desirability score (D) using Design Expert® 11. The settings of 20% (w/w) maltodextrin, an inlet drying temperature of 210°C, and a feed pressure of 12Hz yielded the highest desirability score, indicating the best balance between moisture control and powder functionality. At these optimized settings, the model predicted a water activity of 0.2613, a moisture content of 2.86%, a hygroscopicity of 13.75 g/100 g, and a solubility index of 0.2613, 2.86%w.b, 13.75g/100 g solids, and 0.82 mL, respectively. The results of the two-sided confirmation test confirm that the second-order polynomial equations could be used to predict the physical properties of buffalo milk powder (Table 5).

Table 5. Results of the verification tests for the proposed model equation for the different responses at the 95% confidence level.

Maltodextrin level	**Inlet temperature**	**Feed pressure**	**Two sided Prediction interval @ 95% confidence level**		
(%w/v)	(˚C)	(Hz)	Predicted mean	Observed mean	% Error
		a_w			
20	210	12	0.2639	0.2200^{ns}	16.62
		%MC			
20	210	12	2.8572	1.9820^{ns}	30.63
		Solubility			
20	210	12	0.7802	0.6605^{ns}	15.35
%Hygroscopic moisture					
20	210	12	15.2775	16.0393^{ns}	4.98

DISCUSSION

Physical Properties of Spray-dried Buffalo Milk

Water activity. Water activity (a_w) is a crucial indicator of the spray-dried milk's quality and stability. Milk powders are meant to be stored for a long time therefore it is important to minimize changes and damage during storage. Water activity represents the amount of free water present in food. This free/unbound water allows physical, chemical, microbiological and biochemical reactions to occur in the food system.

The water activity of the spray-dried milk ranged from 0.130 to 0.312 (Table 2). This range is critical for microbial safety and extended shelf life, as it remains significantly below the threshold of 0.6, where all microbial

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growth is inhibited (Labuza 1970; Chirife et al. 1996). At water activity levels below 0.6, the osmotic pressure is too high for cellular functions in most microorganisms, effectively halting spoilage and pathogenic growth. In terms of shelf-life, the low water activity maintains the powder in a stable glassy state. As reported by Jouppila and Roos (1994), water activity values in milk powder below 0.37 prevent the lactose crystallization that causes caking and loss of solubility. Furthermore, at these low levels, the rate of Maillard browning and lipid oxidation is significantly minimized, thereby preserving the sensory attributes and nutritional quality of the buffalo milk over time.

The a_w values of buffalo milk powders were comparable to those reported in other studies on whole milk powder. For instance, Pugliese et al. (2017) reported a_w values ranging from 0.249 to 0.329. Moreover, these values were also consistent with the a_w values of 0.360 and 0.230 for whole milk powder as reported by Szulc et al. (2016) and Thomsen et al. (2005), respectively.

Table 3 shows that the model used for predicting the a_w of the milk powder was significant. The quadratic terms of maltodextrin level and inlet temperature were also significant at $P < 0.05$. Moreover, the interaction between the maltodextrin level and inlet temperature, as well as the linear terms of inlet drying temperature and feed pressure, was found to be significant at $P < 0.05$. The initial lack-of-fit test for the water activity model was significant and there was only a 2.75% chance that a lack-of-fit *F*-value this large could occur due to noise. To improve the fit of the model used to predict the a_w of the powdered milk, factors found to be insignificant were removed (Table 4). The resulting reduced quadratic model exhibited a nonsignificant lack-of-fit, which means that it provided a better fit than the original proposed model. While the R^2 values indicate that some variability remains unexplained, the model's reliability was established through a lack-of-fit and a rigorous two-sided confirmation test. In biological systems like buffalo milk, inherent raw material variability often results in lower R^2 values than those observed in synthetic systems. However, because the experimental means for all responses fell within the 95% PI, the model was considered a robust tool for identifying the optimized processing conditions.

Figure 1 illustrates the response surface and contour plots of a_w at different feed pressures. The water activity (a_w) of the powdered milk was found to be inversely related to both the maltodextrin concentration and the inlet drying temperature. An increase in the amount of maltodextrin added as a carrier agent coupled with an increase in the inlet drying temperature resulted in a decrease in the water activity values of the powdered milk produced (Figure 1). A high inlet drying temperature resulted in the evaporation of more unbound water from liquid milk, while an increase in the addition of maltodextrin as a carrier to the milk feed resulted in a reduction in its initial moisture content. As a result, powdered milk with a low a_w value was produced. The result agrees

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with the observations of Quek et al. (2007), who reported a reduction in the moisture content of spray-dried watermelon powders as the amount of maltodextrin added was increased. This is because the addition of maltodextrin increases the total solids content of the feed and consequently reduces the amount of water that needs to be evaporated.

On the other hand, the a_w values obtained were directly proportional to feed pressure. Low feed pressure produced milk powder with a very low a_w value and increasing feed pressure increased its a_w value at a constant maltodextrin level and inlet drying temperature (Figure 1). Feed pressure determines the product flow rate into the spray dryer, with higher pressures increase the volume of milk atomized into the drying chamber per unit of time. This increased flow rate results in the formation of larger liquid droplets. Consequently, the surface-area-to-volume ratio of the atomized milk is reduced. These larger droplets possess a smaller surface area relative to their mass, which reduces the rate of heat exchange with the drying medium. This can lead to a decrease in outlet air temperature, subsequently increasing the outlet air humidity and relative humidity. Furthermore, larger droplets increase the distance required for heat to travel to the center of the droplet and for moisture to migrate from the core to the surface (Tan et al. 2011). As a result, moisture evaporation becomes less efficient, leading to powdered products with higher final moisture content and water activity. This mechanism explains why optimizing feed pressure is critical to ensuring the stability and quality of buffalo milk powder.

Moisture content. The moisture content of powdered milk products is related to their drying efficiency, storage, handling properties, microbial and chemical reactions, and has a significant effect on their quality and shelf-life. The moisture content of buffalo milk powder ranged from 1.71% to 4.04% with an average of 2.89% (Table 2). This mean value was utilized for comparison with existing literature to provide a standardized assessment of the efficiency of the spray-drying process. The average was lower than the moisture content of 3.37% to 4.49% obtained by Kajal et al. (2012) from different brands of whole milk powder sold in the local market in Bangladesh. Nevertheless, the observed moisture content range (1.71% to 4.04%) for all experimental runs was below the maximum moisture content of 5% established by the Codex Alimentarius Commission (1999) and is within the standards of 2-5% set by the American Dry Milk Institute Inc. (1959). In general, a moisture content below 5% is considered good for dried food products (Sinija and Mishra 2008).

The analysis of variance for the prediction model for moisture content of spray-dried buffalo milk as affected by different maltodextrin levels, inlet drying temperature, and feed pressure revealed no significant effect of these three variables on the moisture content of the powdered milk produced (Table 4). Response surface plots of moisture content (Figure 1) further illustrate the lack of significant differences in the moisture content of

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powdered milk produced under various spray-drying conditions. This implies that other parameters that were not considered in this experiment could be better predictors of the final moisture content, including the drying outlet temperature and humidity. Masters (1991) and Písecký (1997) showed that powder moisture content is related to the outlet air temperature during spray drying. Moisture content decreases with increasing air temperature. In addition, Schmitz-Schug et al. (2013) demonstrated that the relative humidity of the outlet air is correlated with a_w and the moisture content of spray-dried powders. Moreover, Schuck et al. (2008) explained that it is necessary to control the relative humidity (RH) of the outlet air to regulate the water activity and moisture content of dairy powders. In the production of powdered milk, managing the relative humidity of the outlet air is as crucial as controlling the outlet air temperature to achieve the desired moisture content, regardless of the humidity of the incoming air, mass flow rate, or inlet air temperature. However, in this experiment, it was not possible to control the outlet air temperature and humidity, as these were dependent on the inlet drying temperature. Keeping the outlet air temperature and humidity constant during spray drying would be particularly challenging, if not impossible. Hence, the feed flow rate and feed concentration were manipulated in addition to altering the inlet temperature of the spray dryer. Anandharamakrishnan and Ishwarya (2015) explained that increasing the feed flow rate decreases the outlet temperature, which in turn increases the temperature difference between the inlet and the outlet temperatures, resulting in powdered milk with higher residual moisture content. Moreover, the feed concentration was modified using various levels of maltodextrin. The higher the feed concentration, the greater the moisture content of the powder, as reported by Anandharamakrishnan and Ishwarya (2015). However, regardless of the conditions used, the moisture content of the spray-dried buffalo milk conformed to the standard set by the Codex Alimentarius Commission for powdered milk.

Hygroscopic moisture. Hygroscopicity measures the powder's tendency to absorb moisture from the environment. Powders that absorb large amounts of moisture may cake during storage (Tamime 2009). The hygroscopicity of spray-dried buffalo milk powder produced under different spray-drying conditions ranged from 10.30 to 18.77 g/100 g solids (Table 2). The highest value obtained was comparable to the hygroscopic moisture value of 18.8g/100 g solids reported for commercial dried milk sold in the United Arab Emirates (Sulieman et al. 2014). It was also significantly lower than the hygroscopicity of spray-dried camel milk and spray-dried cow's milk, which were 20.52 g/100 g solids and 20.64g/100 g solids, respectively, as reported by Sulieman et al. (2014). This lower hygroscopicity is likely due to the addition of maltodextrin (DE 12) to the buffalo milk slurry. Unlike the study by Sulieman et al. (2014), which used vacuum evaporation to increase the solids content, the inclusion of maltodextrin provides a protective effect. While higher DE maltodextrins generally have lower molecular

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weights and lower water permeability – which may explain the reduced hygroscopicity observed here – there is a trade-off in stability. Although some researchers suggest using lower DE maltodextrins to raise the glass transition temperature and prevent caking (Goula and Adamopoulos 2003), the concentration of maltodextrin remains the dominant factor. As reported in similar studies on mango (Jaya and Das 2004) and tomato powders (Goula and Adamopoulos 2008), increasing the maltodextrin ratio effectively reduces both hygroscopicity and caking, regardless of the specific DE value used.

Figure 1D illustrates the response surface and contour plots of hygroscopicity, showing a distinct interaction between inlet temperature and maltodextrin level. While the addition of maltodextrin is essential for creating a stable powder with lower hygroscopicity than carrier-free milk, increasing its level from 6.59% to 15% resulted in a rise in hygroscopic moisture from 10.30 to a peak of 18.76 g/100 g solids. This upward trend, visible in the curvature of the response surface, is attributed to the inherent hygroscopic nature of polysaccharides. Conversely, higher inlet temperatures (up to 210℃) generally lowered hygroscopicity. At a maltodextrin level of 10% and a feed pressure of 12Hz, increasing the temperature from 190℃ to 210℃ reduced hygroscopicity from 12.46 to 10.73 g/100 g. This is likely due to the increase in surface free fat content (~11%) at high inlet temperatures, which acts as a hygroscopic barrier against moisture. In this study, the final hygroscopicity of the powder was determined by the balance between the moisture-binding sites of the maltodextrin and the moisture-repelling properties of the surface fat.

Statistical analysis revealed that the effect of the quadratic terms of maltodextrin level and inlet temperature was significant at $P < 0.05$ (Tables 3 and 4). However, no significant effect was found for the interactions among the factors considered in the experiment. The lack-of-fit test was also not significant at $P < 0.05$ indicating that the model used adequately described or predicted the hygroscopicity of the milk powder.

Insolubility index (ISi). Powder solubility is an important feature of any powdered food product. A poorly soluble powder can cause reconstitution difficulties, affecting its functional characteristics and potentially resulting in economic losses. The insolubility index is an indirect method for measuring the solubility of milk powder. Milk powder with a high insolubility index is less soluble than milk powders with a low insolubility index value.

The ISi values obtained ranged from 0.467 mL to 4 mL at 24°C (Table 2). The wide range of ISi values obtained reflects the need for optimizing the spray drying conditions as well as the concentration of the milk feed as powders with high ISi values are unsuitable for applications requiring full reconstitution. This also means that

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not all powders produced under different spray drying conditions are suitable for use as raw materials for further processing, such as yogurt production and use as a coffee creamer, since milk powder has to be reconstituted before further processing.

The highest insolubility index was observed in milk powder produced at the maximum inlet drying temperature (210°C) and the lower feed pressure/product flow rate (8Hz) whereas the lowest value was observed in milk powders spray dried at 200°C and the maximum feed flow rate (Table 2). Anandharamakrishnan et al. (2008) noted that the solubility of spray-dried dairy products depends heavily on the structural state of their proteins. When subjected to elevated temperatures, milk proteins may transition from their native state to a denatured form. This is supported by Sharma et al. (2012) who explained that the insolubility of milk powder occurs when β-lactoglobulin unfolds at elevated temperatures and subsequently aggregates with casein. The particle temperature during the drying stage, when the moisture content of milk powder is between 10% and 30%, was identified as the main factor controlling the insolubility index of milk powder.

The insolubility index of spray-dried buffalo milk powder was higher compared to the ISi obtained by other researchers, including Pugliese et al. (2017) and Augustin et al. (2014), for whole cow's milk. However, some experimental treatments produced milk powder with an ISi value ≤1 mL which is the maximum ISi value considered as acceptable by the Codex Alimentarius Commission (1999). Analysis of variance for the solubility of milk powder revealed that the model used to predict its ISi value was highly significant which means that there was only a 0.01% chance that an *F*-value this large could occur due to noise. However, the lack-of-fit test showed a slightly significant result at $P < 0.05$. To improve the model fit, insignificant model terms were removed. The reduced quadratic model provided a better fit for predicting the solubility of milk powder (Table 4). The effect of the quadratic terms for maltodextrin as well as the cross-product effect of inlet temperature and feed pressure were highly significant at $P < 0.01$. Anandharamakrishnan and Ishwarya (2015) reported that maltodextrin is highly effective at protecting the core of the milk particles by rapidly forming a dense skin around the core during spray drying. On the other hand, the interaction between inlet temperature and feed pressure affects the insolubility index of the powder because it determines the droplet size and the temperature of contact between the droplets and the hot air. The use of a slower feed flow rate resulted in a smaller droplet size allowing the heating medium to penetrate more efficiently toward the center. The result is graphically illustrated in Figure 1. The ISi values of powdered milk increased when a higher inlet drying temperature and lower feed pressure were used at a constant maltodextrin level. However, when more maltodextrin was added (20%), the ISi value of the milk was reduced reflecting its significant effect ($P < 0.01$), as shown by the ANOVA (Tables 3 and 4). The results of the study

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422 confirmed the observation of Singh and Newstead (1992) that milk solubility primarily depends on the chemical
423 composition of the powder and its physical state; indicating that the type/condition of spray drying and heat
424 stabilizing agent (in this case, maltodextrin) added to milk before processing are among the factors that directly
425 influence the insolubility of milk powders.

426 **Optimum Processing Conditions**

427 The optimization of the spray-drying conditions in the production of powdered buffalo milk was
428 performed within the selected ranges of maltodextrin levels, inlet drying temperature, and feed pressure. The
429 optimization employed a multiple-response method called the desirability function, as described by Myers et al.
430 (2016). The method makes use of an objective function, D(x), called the desirability function which represents
431 the desirable ranges for each response (di). The desirability function of Design Expert version 11 was used with
432 the following criteria: maximum a_w, moisture content within the specified range, minimum hygroscopic moisture,
433 and minimum solubility index with hygroscopic moisture and solubility index given the highest importance score
434 of 5. Based on these criteria, the optimal drying conditions were identified as a maltodextrin level of 20% w/w,
435 an inlet drying temperature of 210°C, and a feed pressure of 12 Hz. These conditions yielded the highest
436 desirability score of 0.756. At these optimized conditions, the a_w, %MC, hygroscopic moisture content, and
437 solubility index of the powdered milk were calculated as 0.2613, 2.86%w.b, 13.75g/100 g solids and 0.82 mL,
438 respectively.

439 **Model Verification**

440 To check the predictive ability of the model, a confirmation test was conducted under the identified
441 optimum drying conditions (20% maltodextrin, 210℃ inlet drying temperature, and 12Hz feed pressure). A two-
442 sided prediction interval (PI) with a 95% confidence level was used in the model verification. No significant
443 differences were found between the predicted and observed means for all responses, as summarized in Table 5,
444 confirming the dependability of the models.

445 The percent error was also calculated to measure the difference of the observed values from the
446 predicted values of the responses. While the percent error for hygroscopic moisture was low at 4.98%, the errors
447 for water activity (16.62%), moisture content (30.63%), and solubility (15.35%) exceeded the traditional 10%
448 threshold. However, these higher relative errors are attributed to the low magnitude of the target values. Percentage
449 error measures are known to be highly sensitive and unstable when the denominators are near zero, which often
450 produces disproportionately large values despite minor absolute differences (Makridakis 1993). Because the
451 observed values remained within the 95% prediction intervals and the deviations did not compromise the physical

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functionality or shelf-stability of the powder, the models were considered statistically valid for optimization purposes.

The optimization of spray-drying conditions for buffalo milk was successfully achieved using response surface methodology and the desirability function. The study identified the optimal processing conditions for spray-dried buffalo milk as a maltodextrin level of 20% w/w, an inlet air temperature of 210°C, and a feed pressure of 12 Hz. These conditions yielded a powder with a high desirability score (0.756) and physical properties - moisture content (1.98%), a_w (0.220), hygroscopic moisture (16.04 g/100 g solids) and insolubility index (0.67 mL) – that conformed to the CODEX Alimentarius standards for powdered milk.

Although the percent error for a_w and solubility exceeded 10%, the absolute differences were functionally negligible, and the experimental values remained within the 95% prediction intervals, confirming the model's practical reliability.

In conclusion, this study successfully demonstrated that response surface methodology can optimize the spray-drying parameters to produce a shelf-stable, high-quality buffalo milk powder that complies with Codex Alimentarius quality standards. Beyond process optimization, the study offers a viable strategy for converting highly perishable fresh buffalo milk into a shelf-stable commodity, directly addressing SDG 2 (Zero Hunger) by reducing post-harvest dairy losses and enhancing food security. The study also addresses SDG 8 (Decent Work and Economic Growth) and SDG 12 (Responsible Consumption and Production) by enhancing the market value of the buffalo milk for smallholder farmers and promoting sustainable, resource-efficient food preservation, respectively. Based on these findings, it is recommended that further studies be conducted on the sensory profile and consumer acceptability of the powder. This would help ensure that the level of maltodextrin used does not negatively impact the traditional flavor profile of buffalo milk. Likewise, it is recommended that studies be conducted on the effect of high inlet temperature (210°C) on the retention of bioactive compounds and proteins inherent in milk. Lastly, it is recommended that other methods of enhancing the formulation, quality, stability, and consumer acceptability of the milk powder, such as the addition of butter flavor, anticaking agents, and antioxidants.

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GENERATIVE AI STATEMENT

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The authors declare that generative AI was not used to create or produce the content or data in this research article. Gemini was used strictly for grammar and spelling corrections, while Turnitin was utilized for plagiarism and similarity checking.

ETHICAL CONSIDERATIONS

The study did not use humans or animals as research subjects.

DECLARATION OF COMPETING INTEREST

The authors declare that there are no competing interests among the authors.

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