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Formulation optimisation for pilot-scale honey powder production: A response surface methodology and central composite design approach

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Abstract: This research aimed to optimise a pilot-scale formulation for seamless scale-up, considering critical variables such as the honey-to-maltodextrin ratio, temperature, and drying time. Employing response surface methodology with a central composite design approach, the investigation systematically assessed the impact of four key factors within predetermined upper and lower limits: honey volume (90–900 g % dry basis), maltodextrin (60–600 g), drying temperature (60–70 °C), and drying time (180–300 min). Subsequently, these factors were randomised and optimised using the Design Expert software system. The analysis of variance revealed the significant impact of each drying factor, their interactions, and squared squares on the honey-to-maltodextrin ratio, as well as the effects of drying temperature and time. Validation results underscored the model reliability, exhibiting narrow standard deviations ranging from 0.001% to 1.3%. These outcomes emphasise the efficacy of Response Surface Methodology (RSM) and Central Composite Design (CCD) in refining formulations, offering valuable insights into appropriate product development and a seamless scale-up process.

Keywords: Design Expert 13; drying time; maltodextrin; scaling up; temperature

Honey, a natural substance produced by bees through the collection and transformation of nectar or honeydew, is prised for its distinct flavour, enticing aroma, and numerous health-enhancing attributes (Alvarez-Suarez et al. 2010). Its composition and quality are diverse, influenced by factors such as the source of honey bee feed, climate, honey maturity, and processing/storage conditions (Jaya et al. 2022). The honey

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content significantly affects the physicochemical properties of honey.

Powdered honey, a processed form with a dehydrated, powder-like consistency, offers advantages like extended shelf life and improved transportability, which are particularly beneficial in the food, beverages, pharmaceuticals, and cosmetics industries (Samborska et al. 2017). Its quality hinges on factors like temperature, humidity, and duration during production (Samborska and Czelejewska 2014). Optimisation of honey powder production, especially at the pilot scale, is essential to ensure the highest quality products.

The use of Design Expert software (version 13.0.5.0), known for effective process optimisation through statistical methods and design of experiments, is pivotal in identifying influential factors and optimising their values for superior product quality (Said et al. 2016). Despite the honey powder potential, challenges persist in the production process, affecting the final product quality (Kılınç and Demir 2017). Addressing these challenges involves optimising the pilot-scale production process, identifying influential factors affecting quality, and utilising Design Expert software effectively.

Scale-up criteria require independent parameters from the process size, considering quantitative parameters and results, as well as physical and mechanical changes associated with scale changes, such as heat transfer or energy effects (Valentas et al. 1990). Geometric, mechanical, thermal, and chemical similarities are critical in scaling food processes and equipment. However, scaled-up products may differ from the original in taste, texture, aroma, and appearance (Ab-Latif et al. 2022).

To address these problems, this research focuses on optimising pilot-scale honey powder production using Design Expert software. Objectives include employing the design of experiments to optimise production factor values (honey-to-maltodextrin ratio, temperature, and drying time), analysing results for optimal conditions, and investigating the effects of these factors on moisture content, water activity, hygroscopicity, solubility, and dissolution time in scaled-up honey powder production. The study aims to optimise and verify the ideal combination of factors for minimal moisture content, water activity, hygroscopicity, solubility time, and maximum solubility in honey powder.

MATERIAL AND METHODS

Material. The present study obtained raw Acacia honey from *Apis mellifera* from Kembang Joyo Sriwijaya in Malang Regency, maltodextrin DE 10 (MD, Sorini Agro Asia Corporindo TBK, Indonesia), mineral water, and Polysorbate 60. Those materials were dried using a vacuum drying oven (VOV-50; B-one, China), and powdered using a multifunctional grinding machine (GM-800S1, Bosch, Germany; MKM6000, SkofjaLoka, Slovenia) with 34 000 rpm.

Experiment design based on (response surface methodology) RSM. The pilot-scale honey powder manufacturing process was optimised using the Design Expert software based on the response surface methodology (RSM). The experimental matrix, consisting of four scaling-up factors, which are honey (X_1), maltodextrin (X_2), drying temperature (X_3), and drying time (X_4), was created with 30 independent treatments. Each treatment underwent processing with specific configurations of X_1, X_2, X_3, and X_4 according to the experimental matrix. The responses of the five factors, namely moisture content, water activity (a_w), hygroscopicity, solubility, and dissolution time, were evaluated. The research employed the RSM Central Composite Design (CCD) generated by the Design Expert software, comprising 30 experiments and fitting to a second-order polynomial regression model with linear, quadratic, and interaction terms. An analysis of variance (ANOVA) at a 95% confidence level was performed for each response variable to assess model significance and suitability, with statistical analysis conducted for terms in the polynomial using *F*-values at probabilities (*P*-value) of 0.001, 0.01, or 0.05. Table 1 presents the complete CCD, conducted with a total block value of 2, and experiments carried out in a random order.

The optimal formula obtained was then verified to obtain the % error (Atanassova et al. 2011). The significance of the main components and their interactions was determined using an ANOVA with a significance threshold of 95% and a *P*-value of 0.050. The mathematical models were derived from the ANOVA table. These models were then used for optimisation purposes, the outcome of which was determined by the value of the coefficient of determination, R^2. If the % error value is less than 5%, it can be ignored and the suggested optimal formula can be considered the best formula (da Silveira et al. 2015).

Processing of powdered honey. The procedure described by Maharani et al. (2023) was modified for pilot-scale processing of honey powder. The materials were homogenised using a mixer, and the resulting sample was placed in a container. For the drying process, all treated samples were introduced into a vacuum machine equipped with a drying chamber. The temperature and time parameters for the vacuum machine were specified, and upon reaching the set temperature, the

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Table 1. Design matrix of the pilot-scale honey powder optimisation process experiment

Sample	X_1 honey (g of % d.b)	X_2 maltodextrin (g)	X_3 drying temperature (°C)	X_4 drying time (min)
A1	90	600	180	60
A2	90	600	300	60
A3	90	60	180	70
A4	90	60	300	70
A5	900	60	180	60
A6	495	330	240	65
A7	90	600	180	70
A8	90	600	300	70
A9	900	600	300	70
A10	900	60	180	70
A11	90	60	180	60
A12	495	330	240	65
A13	495	330	240	65
A14	900	600	180	70
A15	90	60	300	60
A16	900	600	300	60
A17	900	60	300	70
A18	900	60	300	60
A19	900	600	180	60
A20	495	330	240	65
B21	495	330	240	75
B22	495	330	120	65
B23	1305	330	240	65
B24	495	210	240	65
B25	495	330	240	55
B26	495	330	240	65
B27	315	330	240	65
B28	495	870	240	65
B29	495	330	360	65
B30	495	330	240	65

X_1, X_2, X_3, X_4 – factors; d.b. – dry basis

timer was automatically initiated. Next, the jet pump switch was activated, and the pressure lever was adjusted until it reached a pressure of 0.1 MPa on the vacuum gauge. Once the timer reached the predetermined time, the jet pump switch was turned off, and the pressure lever was reverted to restore the machine pressure to normal levels. The honey samples were ground into powder using a milling machine following the drying phase. The resulting honey powder was then transferred into standing pouches made of aluminium foil for storage.

Analysis of powder (physical) properties. In the analysis of the physical properties of honey powder, moisture content was determined using the gravimetric method (Official Methods of Analysis 2005), involving coding aluminium cups, heating at 105 °C, desiccating, and weighing. Water activity (a_w) was measured using hygrometric methods (Demarchi et al. 2013), involving calibration, sample insertion, and recording the a_w value. The hygroscopicity assessment followed the method by Cardoso and Pena (2014), where cups were conditioned, weighed, and calculations were performed for dry weight and water addition. Solubility was measured based on Fang et al. (2008), involving the formation of round filter paper, oven drying, and weighing before

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and after dissolution. Dissolution time was determined by measuring the time for the powder to completely dissolve in water, following the method of Nyhammar and Eksborg (1991), using one gram of the sample dissolved in 50 mL of water with a magnetic stirrer.

RESULTS AND DISCUSSION

Table 2 displays the design configuration derived from the Design Expert software and the experimental response data.

Modelling and response analysis of moisture content. The results of the moisture content response analysis suggest the quadratic model is the recommended model. In the summary statistical model, the quadratic model exhibits the highest adjusted R^2 and predicted R^2 values, standing at 0.9803 and 0.9397, respectively, surpassing the linear and 2FI models. The quadratic model further demonstrates a notably low prediction error sum of squares (PRESS) value of 4.54. Additionally, all primary factors are highly significant, with P-value of 0.000. Figure 1 visually represents the quadratic

Table 2. Design matrix and response value for response tests

Sample	Responses				
	moisture content (%)	water activity a_w	hygroscopicity (g H_2O per week)	solubility (%)	dissolving time (min)
A1	2.09	0.210	0.0664	98.88	48
A2	1.78	0.201	0.0672	98.99	47
A3	2.50	0.230	0.0635	98.45	49
A4	2.23	0.227	0.0658	98.60	49
A5	7.15	0.597	0.0551	93.00	70
A6	2.78	0.279	0.0622	98.00	50
A7	2.54	0.232	0.0636	98.24	50
A8	1.65	0.196	0.0699	99.20	47
A9	2.58	0.267	0.0631	98.20	50
A10	7.01	0.536	0.0553	93.20	72
A11	3.46	0.412	0.0620	97.65	52
A12	2.88	0.288	0.0644	97.98	51
A13	2.85	0.286	0.0642	98.00	51
A14	2.97	0.393	0.0640	97.98	51
A15	3.20	0.387	0.0639	97.56	52
A16	2.79	0.279	0.0651	98.03	50
A17	6.37	0.510	0.0563	93.98	67
A18	6.83	0.522	0.0558	93.90	68
A19	3.09	0.401	0.0635	97.42	52
A20	2.87	0.286	0.0641	98.00	50
B21	2.80	0.278	0.0648	98.43	49
B22	3.20	0.388	0.0639	97.56	52
B23	6.13	0.499	0.0569	94.00	66
B24	2.96	0.392	0.0644	97.90	50
B25	2.98	0.393	0.0644	97.89	51
B26	2.54	0.233	0.0630	98.48	50
B27	2.42	0.220	0.0633	98.55	49
B28	2.10	0.214	0.0652	98.76	49
B29	2.81	0.280	0.0646	98.47	49
B30	2.83	0.282	0.0645	98.00	49

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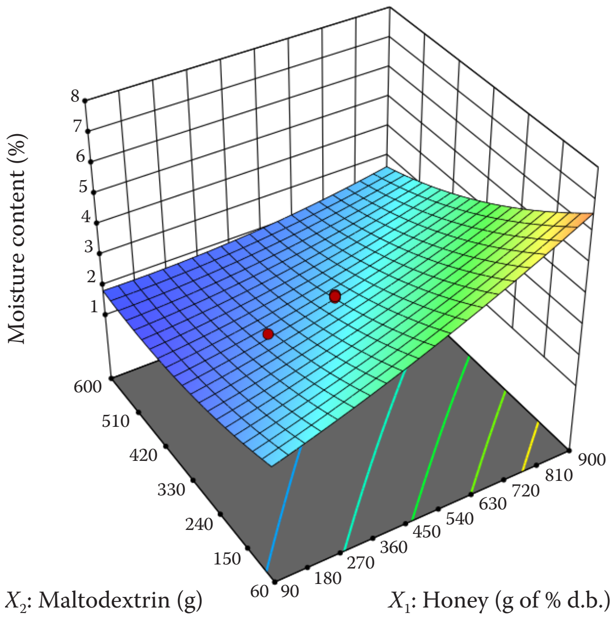

Figure 1. Response surface for moisture content with 30 treatments

Blue, green, yellow, and red hues – surface symbolising the gradient range from the lowest to the highest response values; red dot – response value above the surface; pink dot – response value below the surface; d.b. – dry basis

model generated by the moisture content response, featuring an impressive R^2 value of 0.9901, approaching the ideal value of 1. This proximity to 1 signifies an increasingly appropriate correlation between observational and predictive values (Saniah and Hasimah 2008).

Figure 1 illustrates the effect of four factors (X_1, X_2, X_3, and X_4) on the moisture content of honey powder pilot-scale formulation with a 3D surface graph. Drying conditions and formulation with low moisture content were detected in sample A8 with 90 g of % dry basis (d.b.) of honey and 600 g of maltodextrin, with drying temperatures of 70 °C for 300 min of time. The lowest observed concentration of moisture content in honey powder pilot-scale formulation was 1.65%. The longer drying time causes water evaporation from the material to be greater (Jedlińska 2012). The decrease in water content can occur when some of the water content in food ingredients decreases due to the process of water evaporation from food ingredients to the outside environment. In the roasting process, the water contained in the material will evaporate due to the difference in heat between the material and the temperature in the drying machine (Nurhadi et al. 2012).

Modelling and response analysis of water activity. The analysis of a_w response yields compelling evidence in favour of the quadratic model as the optimal choice. In the summary statistical model, the quadratic model excels with the highest adjusted R^2 and predicted R^2 values, amounting to 0.9160 and 0.8018, respectively, outperforming both the linear and 2FI models. Additionally, the quadratic model shows an exceptionally low PRESS value of 4.98. Figure 2 visually represents the quadratic model derived from the water activity response, revealing an impressive R^2 value of 0.9580. This R^2 value approaches the ideal maximum of 1, underscoring the model's efficacy in capturing the variance in the data and reinforcing its suitability for accurate predictions in the context of water activity analysis. The a_w is an important parameter in the production of honey powder as it affects the quality and shelf life of the final product. The a_w is defined as the ratio of the vapour pressure of water in a sample to the vapour pressure of pure water at the same temperature and pressure (Chen 2019). The water activity of honey powder should be low enough to prevent microbial growth and spoilage, but high enough to maintain the desired physical and chemical properties of the powder (Ganaie et al. 2021). Drying and formulation conditions with low water activity were detected in sample A8, the same as the sample with the lowest moisture content, with 90 g of % d.b. of honey and 600 g of maltodextrin, with drying temperatures of 70 °C for 300 min. The lowest observed concentration of water activity in honey powder pilot-scale formulation was 0.0196%. This is related to the study by Chirifem et al. (2004), who found that the a_w and moisture content were positively correlated, indicating that an increase in moisture content led to increased water activity.

Modelling and response analysis of hygroscopicity. The results of the program analysis aimed at the response of hygroscopicity indicate that the quadratic model was the recommended model. In the summary statistical model, the quadratic model excels with the highest adjusted R^2 and predicted R^2 values, amounting to 0.8750 and 0.7032, respectively, outperforming both

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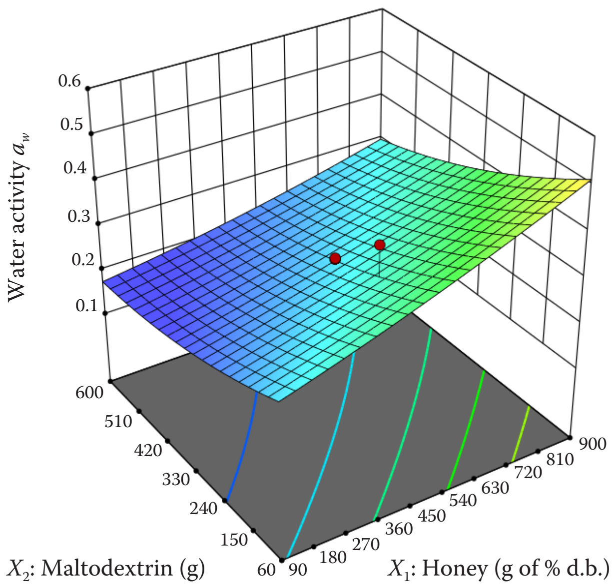

Figure 2. Response surface for water activity (a_w) with 30 treatments

Blue, green, yellow, and red hues – surface symbolising the gradient range from the lowest to the highest response values; red dot – response value above the surface; pink dot – response value below the surface; d.b. – dry basis

the linear and 2FI models. Additionally, the quadratic model shows an exceptionally low PRESS value of 3.15. Figure 3 provides a visual representation of the quadratic model derived from the hygroscopicity response, revealing an impressive R^2 value of 0.9375. This R^2 value approaches the ideal maximum of 1.

The hygroscopicity of honey powder pilot-scale formulation ranged from 0.0551 to 0.0699. The lowest hygroscopicity among 30 treatments was observed in sample A5 with 900 g of % d.b. of honey and 60 g maltodextrin, with drying temperatures of 60 °C for 180 min. Furthermore, the highest hygroscopicity was found in sample A8 with 90 g of % d.b. of honey and 600 g of maltodextrin, with drying temperatures of 70 °C for 300 min.

Modelling and response analysis of solubility. The results of the solubility response analysis suggest the quadratic model as the recommended model. In the summary statistical model, the quadratic model excels with the highest adjusted R^2 and predicted R^2 values, amounting to 0.9745 and 0.9195, respectively, outperforming both the linear and 2FI models. Additionally, the quadratic model shows an exceptionally low PRESS value of 6.58. Figure 4 provides a visual representation of the quadratic model derived from the solubility response, revealing an impressive R^2 value of 0.9872, approaching the ideal value of 1.

In the study by Goula et al. (2004) it was found that the lower the moisture content of the powder, the better is the solubility. This is in accordance with the re-

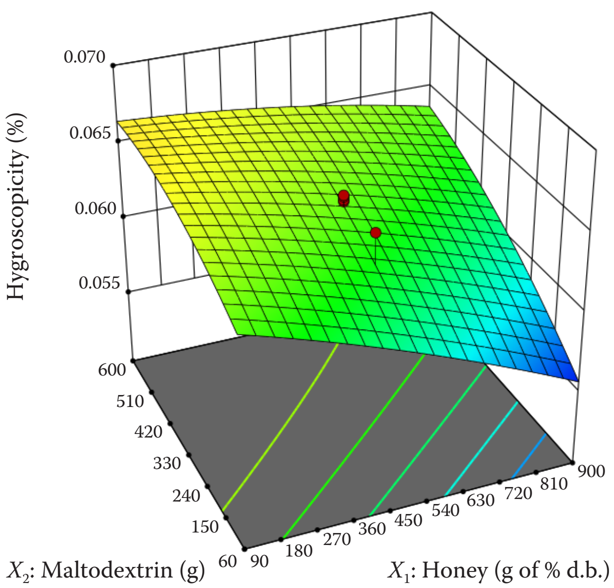

Figure 3. Response surface for hygroscopicity with 30 treatments

Blue, green, yellow, and red hues – surface symbolising the gradient range from the lowest to the highest response values; red dot – response value above the surface; pink dot – response value below the surface; d.b. – dry basis

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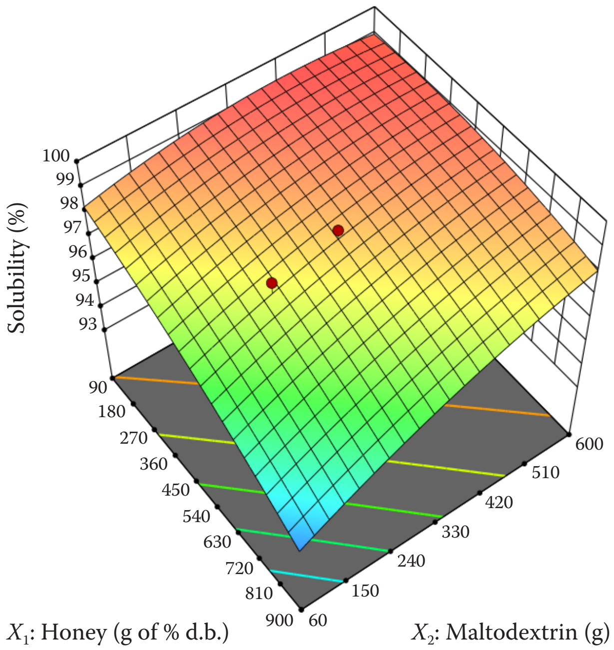

Figure 4. Response surface for solubility with 30 treatments

Blue, green, yellow, and red hues – surface symbolising the gradient range from the lowest to the highest response values; red dot – response value above the surface; pink dot – response value below the surface; d.b. – dry basis

sults of this study where the highest solubility was found in sample A5, which had the lowest moisture content among the 30 experimental samples.

Modelling and response analysis of dissolution time. The analysis of dissolution time response yields compelling evidence in favour of the quadratic model as the optimal choice. In the summary statistical model, the quadratic model excels with the highest adjusted R^2 and predicted R^2 values, amounting to 0.9698 and 0.9189, respectively, outperforming both the linear and 2FI models. Additionally, the quadratic model shows an exceptionally low PRESS value of 20.90. Figure 5 provides a visual representation of the quadratic model derived from the dissolution time response, revealing an impressive R^2 value of 0.9849. This R^2 value approaches the ideal maximum of 1, underscoring the model's efficacy in capturing the variance in the data and reinforcing its suitability for accurate predictions in the context of water activity analysis. The instant property of a powder is defined as its ability to dissolve in water. Hence, the ideal powder would dissolve quickly. The solubility time decreased with the rise in inlet air temperature as well as outlet air temperature, being the first factor that most affected the powder solubility time (Samborska and Bieńkowska 2013).

Optimisation process of honey powder pilot scale. The optimisation process conducted in this study aimed to determine the optimal ratio of maltodextrin to honey, as well as the temperature and drying time for scaling up honey powder. The desired composite

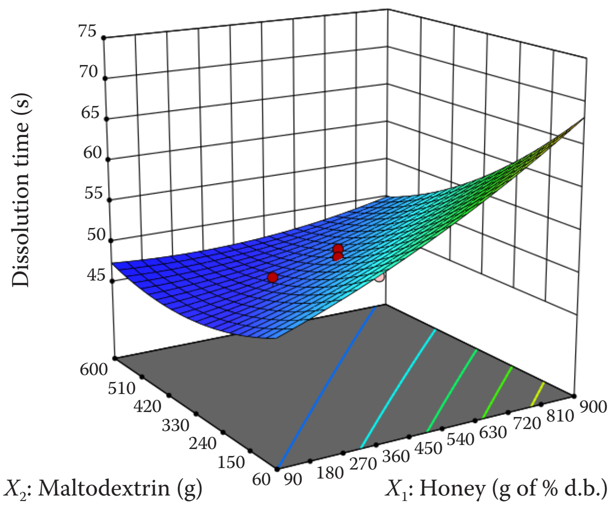

Figure 5. Response surface for dissolution time with 30 treatments

Blue, green, yellow, and red hues – surface symbolising the gradient range from the lowest to the highest response values; red dot – response value above the surface; pink dot – response value below the surface; d.b. – dry basis

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value (desirability), D, was calculated to be close to 1, indicating that all factors were within the workable range. The D value of the honey powder pilot-scale formulation was 0.687, which suggests that the optimisation process successfully achieved the desired quality parameters. Figure 6 displays the honey powder pilot-scale formulation optimisation plot. The optimal values for honey volume were 591.945 g of % d.b., maltodextrin was 360.976 g, and temperature and time for drying were 290 min and 60 °C, respectively.

Experimental verification. Experimental verification is the final phase in the modelling procedure and it is used to check that the predicted model (the regression coefficient model) is accurate (Thacker et al. 2022). In Table 3, by comparing the experimental (actual) value with the predicted figures, this verifies the predictability of the model and indicates that the RSM-based empirical model can accurately explain the correlation between the variables and the goal response, thereby successfully confirming the optimal process conditions. Verification of the suggested formula by RSM produces a standard deviation in the range of 0.001–1.3%; this indicates the variability of the data points around the fitted values. The standard deviation (SD) of the residuals

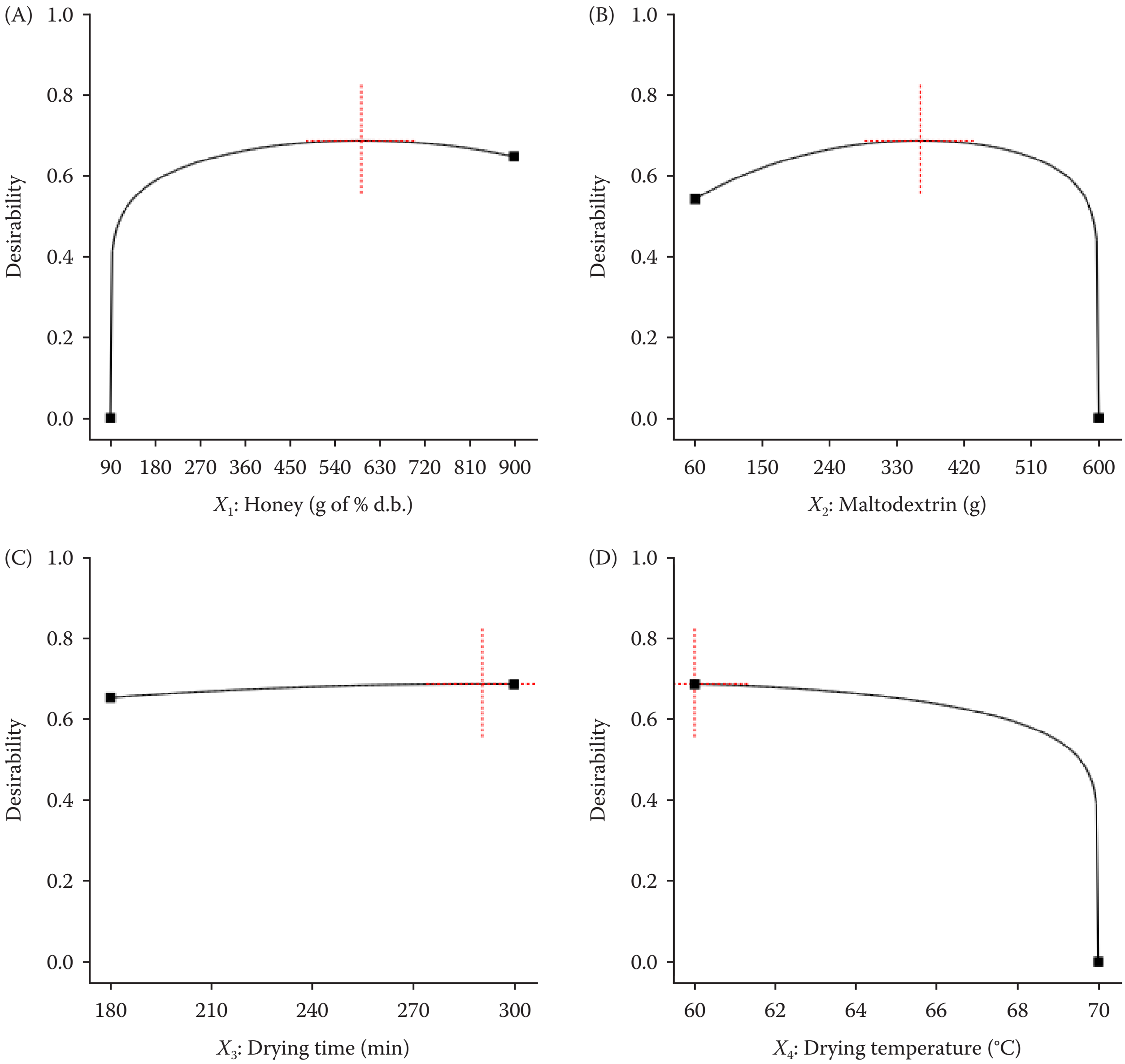

Figure 6. Optimisation plot for all the factors: (A) X_1 – honey, (B) X_2 – maltodextrin, (C) X_3 – drying time, and (D) X_4 – drying temperature

d.b. – dry basis

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Table 3. Experiment verification

Independent variable				Response value			
X_1 (g of % d.b.)	X_2 (g)	X_3 (min)	X_4 (°C)	response	prediction mean	actual	standard deviation (%)
				moisture content (%)	3.0054	2.6825	0.2
				water activity a_w	0.3107	0.3402	0.03
591.945	360.976	290	60	hygroscopicity (g H2O per week)	0.0639	0.0625	0.001
				solubility (%)	97.94	98.20	0.3
				dissolving time (min)	50	48	1.3

Scaling-up factors for X_1 –honey; X_2 – maltodextrin; X_3 – drying temperature; X_4 – drying time; d.b. – dry basis

is a measure of the goodness-of-fit of the model and can be used to assess how well the model describes the response (da Silveira et al. 2015). A smaller SD of the residuals suggests a better fit of the model to the data.

CONCLUSION

Formulating products at both the lab scale and pilot scale involves intricate considerations that demand careful attention. The interplay of factors such as the ratio of honey to maltodextrin, drying temperature, and time significantly influences key parameters like moisture content, water activity, hygroscopicity, solubility, and dissolution time. The application of statistical analysis through RSM and CCD demonstrated the robustness of these methods across all five factors. The constructed prediction model, represented by the regression coefficient model, exhibited remarkable accuracy in predicting outcomes. Validation results further affirm the reliability of the model, with the standard deviation ranging from 0.001% to 1.3%. These findings underscore the efficacy of RSM and CCD in optimising formulations, providing valuable insights into precision in product development and scale-up processes.

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