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Modification on Drying Chamber Wall of Spray Dryer towards Better Yield

(Pengubahsuaian pada Dinding Kebuk Pengeringan Pengering Semburan ke arah Hasil yang Lebih Baik)

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ABSTRACT

Spray drying is a widely used industrial process that converts liquid or slurry feed materials into dry powder or granules and it commonly shrouded with the stickiness problem. This study was carried out by response surface methodology (RSM) to minimize fouling during the spray drying process by optimizing the condition of the drying chamber wall of the spray dryer. The concentration (%) and exposure time (min) of polytetrafluoroethylene (PTFE) were examined as independent variables in order to modify the dryer wall. Responses including flux adhesion weight, product recovery, hygroscopicity, and moisture content of the powder were evaluated. Statistical analysis showed that experimental data were best fitted into a quadratic polynomial model with regression coefficient values greater than 0.75 for all responses. The optimum conditions in reducing fouling were discovered at PTFE concentration of 17.27% and PTFE exposure time of 6 min. These conditions would result in low flux adhesion weight (35.28 mg), high product recovery (39.38%), low hygroscopicity (6.08%) and low moisture content (7.97%). The observed outcomes aligned with the predicted values, affirming the suitability of the model in improving the flowability of the spray drying process.

Keywords: Fouling; polytetrafluoroethylene; response surface methodology; spray drying

ABSTRAK

Pengeringan semburan ialah proses perindustrian yang digunakan secara meluas yang menukar bahan suapan cecair atau buburan kepada serbuk kering atau butiran dan ia biasanya diselubungi dengan masalah kelekitan. Penyelidikan ini dijalankan dengan kaedah permukaan respons (RSM) untuk meminimumkan kotoran semasa proses pengeringan semburan dengan mengoptimumkan keadaan dinding kebuk pengeringan pengering semburan. Kepekatan (%) dan masa pendedahan (min) politetrafluoroetilena (PTFE) telah dikaji sebagai pemboleh ubah tak bersandar untuk ubah suai dinding kebuk pengeringan tersebut. Tindak balas termasuk berat lekatan fluks, hasil produk, higroskopisiti dan kandungan lembapan serbuk telah dinilai. Analisis statistik menunjukkan bahawa data uji kaji adalah paling sesuai dengan model polinomial kuadratik dengan nilai pekali regresi melebihi 0.75 untuk semua tindak balas. Keadaan optimum bagi mengurangkan kotoran telah ditemui pada kepekatan PTFE ialah sebanyak 17.27% dan masa pendedahan PTFE selama 6 minit. Keadaan ini akan menghasilkan berat lekatan fluks yang rendah (35.28 mg), hasil produk yang tinggi (39.38%), higroskopisiti yang rendah (6.08%) serta kandungan kelembapan yang rendah (7.97%). Hasil yang diperhatikan sejajar dengan nilai yang diramalkan, mengesahkan kesesuaian model dalam meningkatkan aliran proses pengeringan semburan.

Kata kunci: Kaedah permukaan respons; kotoran; pengeringan semburan; politetrafluoroetilena

INTRODUCTION

The food and pharmaceutical sectors use spray drying as a method to create powders (Woun et al. 2011). It has been frequently used to produce fruit and vegetable juice powders for commercial purposes. The process of spray drying involves converting feed material in liquid or slurry form into a dry powder (Muzaffar, Nayik & Kumar 2015). The feed material is atomized in a drying chamber, where it interacts with hot air. It causes the liquid part of the spray to evaporate, leaving the dried particles behind (Goula & Adamopoulos 2010). Spray drying is mostly applied to create free-flowing powders for the purpose of convenience, longer shelf life and easy incorporation with other products (Ahmad & Nguyen 2017). Many studies have been conducted using spray drying process, such as spray drying of jackfruit pulp and hydrolyzed juice (Navarrete-Solis et al. 2020) and spray drying of pineapple-mint juice (Braga et al. 2020). There was also a study that investigated the spray drying of beetroot juice powders under different temperatures and ratios of inulin to whey protein isolate (do Carmo et al. 2019). In addition, another study has explored the process of producing papaya powder through the combination of enzyme liquefaction and spray drying technique (Chang, Tan & Pui 2020).

However, challenges occur in spray drying particularly with sugar-rich food because it consists of large amounts of sugar and organic acids, which cause the product to become sticky. Powder stickiness is a part of fouling and it occurs when the powder adheres to the wall of dryer, especially in the cone or in the cyclone (Gianfrancesco, Kockel & Palzer 2014). Sugar-rich foods are defined as those food components that are mostly composed of low molecular weight sugars like sucrose, glucose, and fructose. Low molecular weight sugars lead to low glass transition temperature (Sobulska & Zbicinski 2020). If the surface temperature of drying droplets is higher than the glass transition temperature of an amorphous component, and if the droplet contacts the chamber wall, the droplet may adhere to the wall of the dryer, coating the wall surface over time. Particle-particle cohesion and particle-wall adhesion are the characteristics of this stickiness (Ahmad & Nguyen 2017).

Polytetrafluoroethylene (PTFE) is a common type of fluoropolymer composed of C-F bond in the composition, which is present in its molecular structure with the formula $[(CF_2\text{-}\ CF_2)_n]$ (Dhanumalayan & Joshi 2018; Ohkubo et al. 2018). PTFE is widely recognized as a non-stick coating employed in the foodservice sector and for kitchenware (Schlummer et al. 2015). It is renowned for its great thermal stability, low surface tension, strong resistance to water and oil, chemical inertness, effective resistance to fouling and low coefficient of friction (Liu et al. 2004). Due to the tightly packed fluorine atoms, the linear helical $(\text{-}CF_2\text{-}\ CF_2\text{-})$ chains that form PTFE have a smooth cylindrical rod-like shape, which provides a protective barrier. PTFE is a highly crystalline polymer with a melting point of approximately 330 °C (Rondinella et al. 2021).

Response surface methodology (RSM) is a widely used empirical modelling technique that is regarded as comprehensive, simple, and very efficient (Elias et al. 2023; Movahhed & Mohebbi 2016). Mathematical and statistical approaches were implemented in RSM to evaluate the relationship between the variables in order to establish the optimum conditions for all significant responses (Andrade & Flores 2004). Additionally, RSM decreased the quantity of experiments required, thus it can save time (Mohd Yusof et al. 2018; Wan Azizee & Abu Tahrim 2022). There are several studies conducted the analysis using RSM previously, such as optimization of the spray drying conditions for custard apple pulp, with an emphasis on product quality (Shrivastava et al. 2021) and optimization of spray drying process of sour cherry juice, aiming to evaluate physicochemical properties the of the spray-dried product (Moghaddam, Pero & Askari 2017).

The objective of the current study was to analyze the impact of PTFE application in modifying dryer wall of spray dryer in order to reduce stickiness and to determine the optimal parameters of PTFE to create lowest flux adhesion weight, highest product recovery, lowest hygroscopicity and lowest moisture content by applying RSM.

Materials and Methods

SURFACE COATING USING PTFE

The optimization of the surface coating conditions was performed using response surface methodology (RSM) with a central composite rotatable design (CCRD) using Design Expert software version 13.0 (Stat Ease, 2021, USA) which two independent variables included, PTFE concentration (%v/v) and PTFE exposure time (minutes), based on Table 1.

A polytetrafluoroethylene (PTFE) dispersion (60 wt% dispersion in water, Sigma-Aldrich, USA) was diluted to achieve the desired concentration (ranged from 0.69 to 23.31%). A modified version of the coating technique described by Liu et al. (2022) was employed to coat the drying chamber wall at room temperature and based on desired PTFE exposure time (ranged from 1.03 to 34.97 min). The suspension was sprayed on the wall surface with a nozzle diameter 2.5 mm at a spray distance of 50 cm, and then the coating was solidified in a 180 °C oven dryer (Protech, Malaysia) for 30 min to cure the coating materials and evaporate the carrier solution.

MODEL SOLUTION PREPARATION

A model solution containing a mixture of sucrose (Merck, Germany) and maltodextrin (Sigma-Aldrich, USA) with dextrose equivalent (DE) of 4–7 was prepared (Woo et al. 2008) and used in spray drying process. The ratio of sucrose to maltodextrin was 44:55, and the total amount of dissolved solids in the solution was 20%wt. All the materials were carefully measured using an analytical balance (Mettler Toledo AB54, Switzerland). Afterward, the mixture was stirred using magnetic stirrer on a hot plate (Thermo Scientific, China) at 350 rpm for 5 min. Finally, the model solution was stored in a chiller for future use.

SPRAY DRYING PROCESS

Dehydration of model solution was performed according to method by Leyva-Porras et al. (2019) with a slight modification. The 50 mL solution was introduced into the spray dryer (Mini Spray Dryer B-290, Buchi, Switzerland) at room temperature, and it was dried using hot air as drying medium, at a volumetric flow rate of 35 m^3/h and a constant pressure of 0.23 bar. The inlet temperature was set at 150 °C. A cyclone air separator/ powder recovery system was employed for efficient separation and recovery of the product.

TABLE 1. Actual and coded (in parentheses) levels of PTFE concentration (X_1/x_1) and PTFE exposure time (X_2/x_2) used for optimization of drying chamber wall modification in model solution spray drying

Run#	PTFE concentration, $X_1(x_1)$	PTFE exposure time, $X_2(x_2)$
1*	12.00 (0.000)	18.00 (0.000)
2	4.00 (-1.000)	6.00 (-1.000)
3*	12.00 (0.000)	18.00 (0.000)
4*	12.00 (0.000)	18.00 (0.000)
5	20.00 (1.000)	6.00 (-1.000)
6	20.00 (1.000)	30.00 (1.000)
7*	12.00 (0.000)	18.00 (0.000)
8*	12.00 (0.000)	18.00 (0.000)
9	0.69 (-1.414)	18.00 (0.000)
10	4.00 (-1.000)	30.00 (1.000)
11	12.00 (0.000)	1.03 (-1.414)
12	12.00 (0.000)	34.97 (1.414)
13	23.31 (1.414)	18.00 (0.000)

*center point

FLUX ADHESION WEIGHT

For this evaluation, borosilicate glass microscope slides (Labchem Sdn Bhd, Malaysia) were used as substrates to replicate the chamber wall of a spray dryer and were introduced to the PTFE coating treatment. The weight of flux adhesion was evaluated by weighing the substrates before and after the feed spray using an analytical balance (GR-200, A&D Weighing, Japan) (Ramlan et al. 2018). The determination of flux adhesion was performed 30 min after the spray drying process. The substrates were randomly placed on the stage inside the drying chamber, and the weight of the flux deposited on the substrates was calculated using Equation (1):

$$\underset{\text{(mg)}}{\text{Flux adhesion weight}} = \underset{\text{after drying (mg)}}{\text{Substrate weight}} - \underset{\text{before drying (mg)}}{\text{Substrate weight}} \quad (1)$$

PRODUCT RECOVERY

Product recovery can be defined as the ratio of the powder mass to the total mass of solids present in the feed mixture (Zareifard et al. 2012). The assessment of product recovery for the spray-dried liquid samples was conducted following the method by Pui et al. (2020), with minor adjustments. The calculation of the product yield was based solely on the powder collected from the product vessel, while any powder that adhered to the drying chamber or cyclone wall was not included in the calculation. The product recovery was determined based on Equation (2):

$$\text{Product recovery (\%)} = \frac{\text{Mass of powder (g)}}{\text{Solid mass of model solution (g)}} \times 100 \quad (2)$$

HYGROSCOPICITY

The analysis was conducted following the methodology described by Oliveira, Clemente and da Costa (2014), with slight modifications. It involved exposing the powder to an air relative humidity (RH) of 75.3% and monitoring the weight increase until it reached its maximum value. The powder was positioned in the apparatus, and the analysis was initiated. The calculation of hygroscopicity is determined by Equation (3):

$$\text{Hygroscopicity (\%)} = \frac{\%\text{WI} + \%\text{FW}}{100 + \%\text{WI}} \times 100 \quad (3)$$

where %FW is the % free water; %WI is the $\frac{c-b}{b-a} \times 100$; a is the weight of plate (g); b is the weight of plate + powder (g); and c is the weight of plate + powder in equilibrium (g).

MOISTURE CONTENT

The moisture content of the powder samples was analyzed following the method by Ehiem et al. (2019), with slight modification. The samples were dried in a drying oven (Venticell, Germany) at a temperature of 102 °C ± 2 °C for a duration of 2 h until a constant weight was achieved. The drying process was repeated as necessary until two consecutive weighing differed by not greater than 0.5 mg, indicating a constant weight of the samples.

STATISTICAL ANALYSIS

The experimental design and statistical analysis were achieved using response surface methodology (RSM) with Design Expert Version 13.0 (Stat Ease, 2021, USA) software. All experiments were done in triplicates. Statistical models were created using the experimental data to establish a response surface. The suitability of the models was assessed by checking the significance of the two-way analysis of variance (ANOVA), ensuring a coefficient determination (R^2) greater than 0.75, and performing an insignificant lack-of-fit test (Othman et al. 2017). The selected models were then optimized based on the criteria of minimizing PTFE concentration and exposure time, while minimizing flux adhesion weight, hygroscopicity, moisture content and maximizing product recovery. The optimal point will suggest the conditions for PTFE concentration and PTFE exposure time, along with its predictive value. This is followed by the additional confirmation experiments to verify the accuracy of statistical experiment design. The validation of the optimum point was confirmed through the root-mean-squared error (RMSE) (Mamat & Razali 2023), as depicted in Equation (4):

$$\text{RMSE} = \sqrt{\frac{\sum_{i=1}^{n} (y_i - \hat{y}_i)^2}{n}} \quad (4)$$

where y_i is the experimental value; $\hat{y}_i$ is the predicted value; and n is the number of samples.

RESULTS AND DISCUSSION

Response Surface Methodology (RSM) enables the simultaneous assessment of the effects of independent variables, either main and interaction effects (Isa et al. 2022). These effects are evaluated through linear, quadratic, and interaction terms of PTFE concentration (x_1) and PTFE exposure time (x_2) on each response. The experimental results depicting the effect of different

PTFE coatings on spray dried model solution powder are presented in Table 2.

According to the statistical results, it is suggested that the quadratic model was the most suitable model for all responses. Table 3 presents the response surface equations derived from fitting the experimental data to the models. The analysis of variance indicates that all the models were statistically significant. R^2 values for all responses exceed 0.75, suggesting a good fit. The lack-of-fit tests were found to be insignificant for all responses, further confirming the strong agreement between the experimental data and the model (Ramlan, Zubairi & Maskat 2022).

The coefficients analysis for each model used to fit the data of flux adhesion weight, product recovery, hygroscopicity, and moisture content are presented in Table 4. The results indicate that the independent variable which was PTFE concentration, had significant effects ($p < 0.05$) on both product recovery and hygroscopicity. Regarding the interaction variables, the model coefficient for flux adhesion weight showed significance ($p < 0.05$) for x_{22}. The model coefficients for product recovery and hygroscopicity were significant ($p < 0.05$) for x_{11}. In addition, the model coefficient for moisture content was significant ($p < 0.05$) for both x_{11} and x_{22}. The coefficients for PTFE exposure time were in positive values for flux adhesion weight and product recovery, while the coefficient for PTFE concentration were in positive values for only product recovery. These showed that when the variables were increased, the responses values also increased. In contrast, the coefficients for PTFE concentration were in negative values for flux adhesion weight, hygroscopicity and moisture content while coefficients for PTFE exposure time were in negative values for hygroscopicity and moisture content. It was good results when the values for flux adhesion weight, hygroscopicity and moisture content decreased.

TABLE 2. Experimental responses of the effect of PTFE coating on spray dried model solution powder

Run	X_1: PTFE concentration (%)	X_2: PTFE exposure time (min)	Flux adhesion weight (mg)	Product recovery (%)	Hygroscopicity (%)	Moisture content (%)
1	12.00	18.00	23.40	29.32	7.23	13.12
2	4.00	6.00	25.50	26.16	18.83	8.21
3	12.00	18.00	28.80	28.36	8.95	10.07
4	12.00	18.00	27.00	35.36	11.93	12.24
5	20.00	6.00	28.80	47.60	6.45	4.77
6	20.00	30.00	33.00	56.88	3.99	4.23
7	12.00	18.00	27.90	32.20	7.68	13.56
8	12.00	18.00	32.40	33.44	8.24	9.00
9	0.69	18.00	25.80	27.80	19.22	12.00
10	4.00	30.00	33.30	34.24	12.33	2.77
11	12.00	1.03	52.20	30.76	6.49	11.48
12	12.00	34.97	52.50	31.24	7.46	3.86
13	23.31	18.00	22.20	53.24	6.83	3.56

TABLE 3. Model equations fitted for flux adhesion weight, product recovery, hygroscopicity and moisture content experimental data for the effect of PTFE coating on spray dried model solution powder

Response	Model equation	Model significance	Lack of Fit	R^2
Flux adhesion weight	*Actual Equation* $38.0282 + 1.61967X_1-$ $2.31277X_2- 0.009375X_1X_2-$ $0.0618164X_1^2 + 0.0709635X_2^2$ *Coded Equation* $27.9 - 0.261396x_1 +$ $1.55303x2 - 0.9_{x1x2}-$ $3.95625x_1^2 + 10.2188x_2^2$	0.0130 (Significant)	0.0802 (Not significant)	0.8287
Product recovery	*Actual Equation* $29.2144 - 0.964225X_1\ 0.0988456X_2$ $+ 0.003125X_1X_2 + 0.0899531\ X_1^2 +$ $0.00692361X_2^2$ *Coded Equation* $31.736 + 10.0072x_1+$ $2.25485x_2 + 0.3x_1x_2 +$ $5.757x_1^2 + 0.997x_2^2$	0.0032 (Significant)	0.1037 (Not significant)	0.8875
Hygroscopicity	*Actual Equation* $22.8057 - 1.61538X_1 -$ $0.00124099X_2 + 0.0104893X_1X_2+$ $0.0345352X_1^2 - 0.00565604X_2^2$ *Coded Equation* $8.80494 - 4.78183x_1 -$ $0.947839x_2 + 1.00698x_1x_2 +$ $2.21026x_1^2 - 0.81447x_2^2$	0.0029 (Significant)	0.3569 (Not significant)	0.8914
Moisture content	*Actual Equation* $8.31929 + 0.524477X_1 +$ $0.334177X_2+ 0.0127667X_1X_2$ $0.0404893X_1^2 - 0.0183845X_2^2$ *Coded Equation* $11.5988 - 1.73974x_1$ $2.09357x_2 + 1.2256x_1x_2 -$ $2.59132x_1^2 - 2.64737x_2^2$	0.0357 (Significant)	0.1920 (Not significant)	0.7659

TABLE 4. Analysis of coefficients for coded models used to fit flux adhesion weight, product recovery, hygroscopicity and moisture content experimental data for the effect of PTFE coating on spray dried model solution powder

	Flux adhesion weight		
	Coefficient	F	Prob<F
Independent variables			
PTFE concentration, x_1	-0.2614	0.0195	0.8928
PTFE exposure time, x_2	1.55	0.6896	0.4337
Interactions			
x_{12}	-0.9000	0.1158	0.7436
x_{11}	-3.96	3.89	0.0891
x_{22}	10.22	25.96	0.0014
	Product recovery		
	Coefficient	F	Prob<F
Independent variables			
PTFE concentration, x_1	10.01	41.21	0.0004
PTFE exposure time, x_2	2.25	2.09	0.1913
Interactions			
x_{12}	0.3000	0.0185	0.8956
x_{11}	5.76	11.86	0.0108
x_{22}	0.9970	0.3557	0.5697
	Hygroscopicity		
	Coefficient	F	Prob<F
Independent variables			
PTFE concentration, x_1	-4.78	44.41	0.0003
PTFE exposure time, x_2	-0.9478	1.74	0.2281
Interactions			
x_{12}	1.01	0.9846	0.3541
x_{11}	2.21	8.25	0.0239
x_{22}	-0.8145	1.12	0.3250
	Moisture content		
	Coefficient	F	Prob<F
Independent variables			
PTFE concentration, x_1	-1.74	3.70	0.0957
PTFE exposure time, x_2	-2.09	5.36	0.0537
Interactions			
x_{12}	1.23	0.9189	0.3697
x_{11}	-2.59	7.14	0.0319
x_{22}	-2.65	7.46	0.0293

INFLUENCE OF INDEPENDENT VARIABLES ON FLUX ADHESION WEIGHT

The findings stated in Table 4 indicate an inverse correlation between flux adhesion weight and the concentration of PTFE, while a positive correlation exists with PTFE exposure time. The study highlighted that the interaction of x_{22} had a significant impact ($p < 0.05$) on flux adhesion weight. Figure 1 illustrates the effects of PTFE concentration and exposure time on flux adhesion weight. A slight decrease in flux adhesion weight was observed between 12% and 20% PTFE concentration, while the flux adhesion weight values decreased with increasing PTFE exposure time up to 18 min.

Increasing the concentration of PTFE in the coating can enhance the non-stick properties of the drying chamber walls. Treatment with PTFE alter the roughness of the surface thus reduce the powder stickiness on the drying chamber. The effective contact area is significantly reduced on a rough surface due to surface asperities (Ashokkumar & Adler-nissen 2011). Previous research has demonstrated that an increase in the contact angle on a surface lead to a reduction in adhesion (Avram et al. 2008; van der Wal & Steiner 2007). This phenomenon can be attributed to changes in the surface roughness of the glass. When the surface roughness increases, the contact angle also increases, resulting in decreased adhesion to the surface. Increased surface roughness in another study was shown to reduce adhesion (Bowden & Tabor 2001). Extended PTFE exposure time may allow for a well-bonded PTFE coating on the drying chamber wall. The enhanced surface roughness results in reduction of wettability properties (Veeramasuneni et al. 1997). This can improve the hydrophobic characteristic on the surface, making it more difficult for particles or substances to adhere to the wall during the spray drying process.

INFLUENCE OF INDEPENDENT VARIABLES ON PRODUCT RECOVERY

For 13 experimental runs, the yield of the model solution powder ranged from 26.16% to 56.88% (Table 2). The highest yield was 56.88%, obtained from surface treatment of 20% PTFE concentration and 30 min PTFE exposure time. The outcomes displayed in Table 4 demonstrate that the concentration of PTFE had a significant impact ($p < 0.05$) on product recovery. In terms of interaction variables, the model coefficient exhibited significance ($p < 0.05$) for x_{11}. The coefficients for the independent variables, PTFE concentration and PTFE exposure time, were positively valued. This indicates that as these variables increased, the values of product recovery also increased.

Based on Figure 2, powder yield increased with increasing PTFE concentration and PTFE exposure time possibly due to uniformity of hydrophobic surface that can reduce the adhesion of the powder to the surface of the drying chamber. Thus, the stickiness problem that contribute to operational problems and low product yield (Muzaffar, Nayik & Kumar 2015) can be reduced and improve the flowability of the spray drying process.

INFLUENCE OF INDEPENDENT VARIABLES ON HYGROSCOPICITY

The results presented in Table 4 demonstrate that the hygroscopicity of the spray-dried model solution powder were inversely correlated to the concentration of PTFE. Higher concentrations of PTFE were found to correspond with lower levels of hygroscopicity. The study showed that the PTFE concentration (x_1) and the interactions of x_{11} had a significant impact ($p < 0.05$) on the hygroscopicity of the model solution powder. The hygroscopicity values for the spray-dried model solution powders ranged from 3.99% to 19.22%, as indicated in Table 2. Figure 3 illustrates the effects of PTFE concentration and exposure time on hygroscopicity, showing that the hygroscopicity of the model solution powder has decreased substantially with the increase of PTFE concentration. Meanwhile, the same trend appears for the effect of PTFE exposure time on hygroscopicity but at a slower rate.

Hygroscopicity pertains to a powder ability to absorb moisture from surroundings (Vidović et al. 2014). The high hygroscopicity leads to the issue of stickiness (Du et al. 2014), thus resulting in low efficiency in spray drying process. Therefore, the PTFE application in modifying the surface chamber may assist in reducing this stickiness issue. By increasing concentration of PTFE, a thicker coating forms on the drying chamber surfaces, enhancing water repellency (Paz-Gómez et al. 2019). A thicker PTFE coating acts as a more effective barrier against moisture adhesion, thus reducing the powder's hygroscopic nature. An increase of PTFE exposure time for the coating on the drying chamber wall can potentially enhance its effectiveness in preventing particle agglomeration and reducing the moisture absorption of the powder. This is because a longer exposure time allows for a more thorough and uniform coating of the chamber wall, creating a better barrier between the powder particles and the wall surface. A clean hydrophobic surface can make the particles experience less adhesion (Figgis et al. 2018), thus reduce the stickiness of the powder.

INFLUENCE OF INDEPENDENT VARIABLES ON MOISTURE CONTENT

The moisture content of the powder varied from 2.77% to 13.56%. The interactions of x_{11} and x_{22} had a significant impact ($p < 0.05$) on reducing the moisture content, as indicated in Table 4. Figure 4 illustrates that an increase in both the concentration of PTFE and PTFE exposure time led to a decrease in the moisture content of the model solution powder.

The moisture content of the spray dried product is a critical characteristic that significantly influences its flowability, stickiness, and storage stability (Sarabandi et al. 2017). Low moisture was needed because it can hinders particle agglomeration, thereby preventing the formation of powder clumps or caking (Daza et al. 2016). PTFE is renowned for its hydrophobic characteristics and low wettability (Lojen et al. 2022), and when utilized as a surface coating, it possesses the ability to diminish the propensity of surfaces to adhere moisture. Thus, this surface characteristic may help in lowering the moisture content of the powder that had contact with the surface of drying chamber, besides it can enhance the flowability of spray drying process.

OPTIMIZATION OF COATING AND VERIFICATION OF THE MODEL

The Design Expert software was used to perform a numerical optimization in order to find the precise optimum level of independent variables that would result in the overall optimum condition. The optimum point was determined based on the highest desirability of the responses (Haslaniza et al. 2013). The results of the numerical optimization showed that the optimal region for the effect of PTFE coating on spray dried model solution powder was achieved at the minimum PTFE concentration (17.27%) and minimum PTFE exposure time (6 min). The optimum condition for the responses were considered as minimum flux adhesion weight, maximum product recovery, minimum hygroscopicity and minimum moisture content. All the corresponding response values can be found in Table 5. The results indicated that there was no significant difference ($p>0.05$) between the predicted and experimental values for the variables being studied. These findings confirmed that the corresponding response surface models, which relate the experimental data to the independent variables of PTFE concentration and PTFE exposure time, were suitable and accurate. In addition, the RMSE values were also obtained using Equation (4) and indicate the small RMSE values, which verifies the selected model.

TABLE 5. Predicted and experimental values for the response variables studied

Response	Target	Predicted value	Experimental value	RMSE
Flux adhesion weight (mg)	Minimum	35.28	38.67 ± 2.25	3.85
Product recovery (%)	Maximum	39.38	41.88 ± 1.89	2.94
Hygroscopicity (%)	Minimum	6.08	5.27 ± 0.19	0.82
Moisture content (%)	Minimum	7.97	5.67 ± 0.66	2.36

Results are mean ± standard deviations, based on three replicates

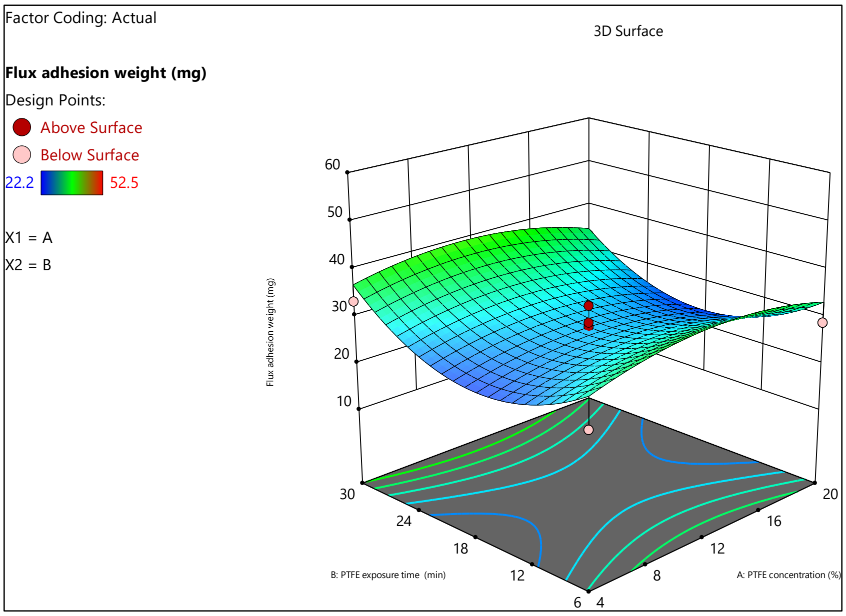

FIGURE 1. Response surface plot for effects of PTFE concentration and exposure time on the flux adhesion weight of spray-dried model solution powder

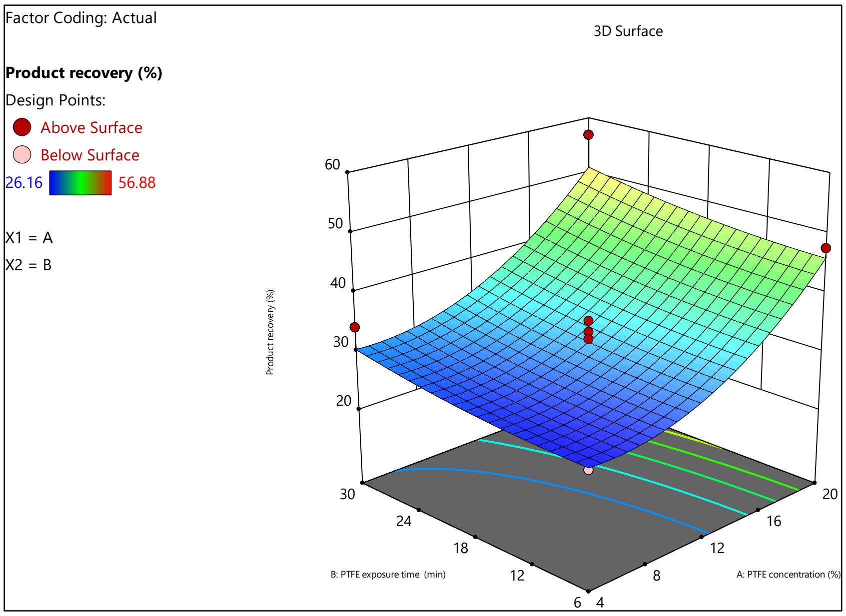

FIGURE 2. Response surface plot for effects of PTFE concentration and exposure time on the product recovery of spray-dried model solution powder

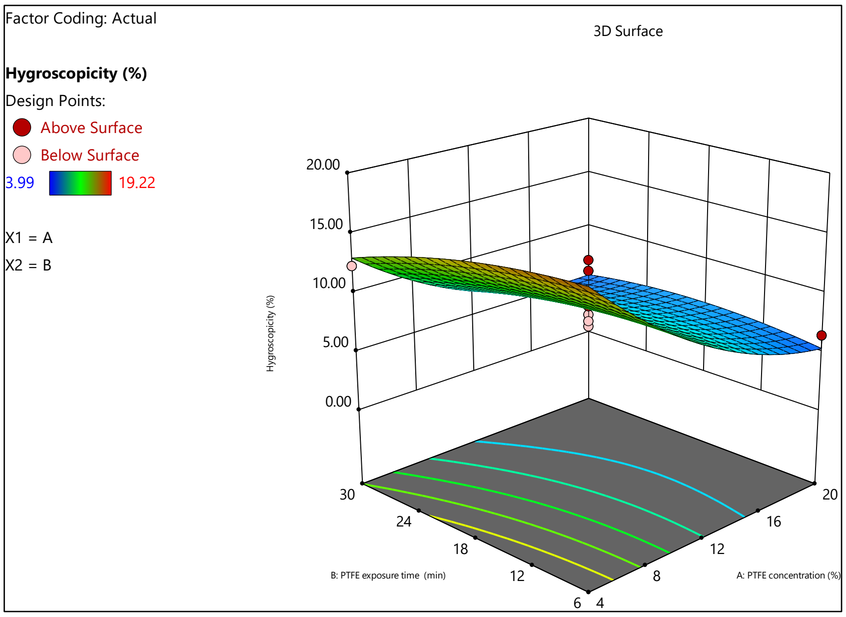

FIGURE 3. Response surface plot for effects of PTFE concentration and exposure time on the hygroscopicity of spray-dried model solution powder

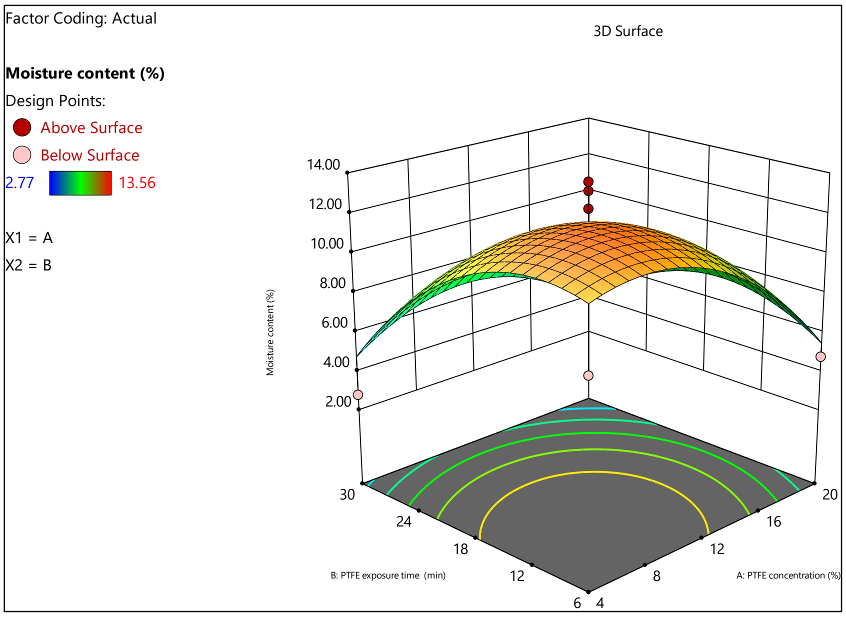

FIGURE 4. Response surface plot for effects of PTFE concentration and exposure time on the moisture content of spray-dried model solution powder

CONCLUSIONS

Thirteen different experimental trials were conducted using the Central Composite Rotatable Design (CCRD) to investigate the impact of modifying the wall of the drying chamber on the spray dried model solution powder. The modifications involved varying levels of PTFE concentration and PTFE exposure time. The concentration of PTFE had a significant effect only on the product recovery and hygroscopicity. Based on the findings, the optimum conditions for modifying the drying chamber wall were determined to be a PTFE concentration of 17.27% and a PTFE exposure time of 6 min. Under these optimum conditions, the interaction between the spray-dried model solution powder and the modified drying chamber wall resulted in a flux adhesion weight of 35.28 mg, a product recovery of 39.38%, a hygroscopicity of 6.08%, and a moisture content of 7.97%. These conditions proved to be the most effective in mitigating fouling issues and improving the flowability of the spray drying process.

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