[1.] Babatope Abimbola OLUFEMİ, [2.] Oluwasola ORİBAYO

NUMERICAL SURFACE RESPONSE PREDICTIVE OPTIMIZATION OF A SPRAY DRYER FOR EVAPORATING CAUSTIC SODA SOLUTION

[1,2.] Department of Chemical and Petroleum Engineering, University of Lagos, Akoka, Lagos, NIGERIA

Abstract: The numerical optimization of the spray dryer operations for the evaporation of caustic soda solution was investigated in this work. The optimization for the drying operation was explored with the Response Surface Methodology (RSM) by utilization of the Design Expert software Version 11.1.2.0 64–bit. The numerical optimization of the spray dryer operations at three inlet air temperatures of 391, 382 and 373 K revealed various operating characteristics, performances and result outputs. The operation at 391 K provided the highest w/w% of the NaOH drying operation and factors or variables needed to achieve the operation. Justifiable optimum parameters obtained are 71.82%, 0.2631 kg/s, 9077.53 kJ/kg, 0.2978 kg/s and 99.5364% for the inlet w/w% of the NaOH solution, outlet mass flow rate of the dried product, specific drying energy per kg of NaOH required, inlet mass flow rate of NaOH solution and final w/w% of the NaOH dried product at inlet air temperature of 391 K respectively.
Keywords: Spray dryer, caustic soda, Response Surface Methodology, optimization, energy

1. INTRODUCTION

Process variables coordination and utilization is very important efficient and improved operation in chemical processing. As globally known, the thermal energy consumption of the chlor–alkali industry are huge. This justifies the need for exploring ways of reducing the energy cost. Conventionally, the initial catholyte liquid solution product coming out of the electrochemical cells in the production of caustic soda needs further evaporation to the grades ready for sale of about 73% w/w NaOH and more, extending to almost 100% anhydrous grade.

High thermal energy consuming multiple effect evaporators are utilized to achieve these saleable grades. Moreover, the materials of construction needed are very costly, thereby require replacement from time to time as a result of the high temperature operations. In addition, steam is usually the energy source for evaporation, which requires high energy consumption. Sodium chloride precipitates from the concentration operations requires that the evaporator must be provided with scraper blades or other mechanisms to remove them successfully. With about 12% w/w NaOH solution catholyte from the electrolytic cell, the steam required to achieve 50% w/w NaOH was estimated to be 2.68 x 10^6 J/kg NaOH according to Tilak *et al*, (2007). According to Worrel *et al.*, (2000), estimated values of about 2.35 x 10^6 J/kg and 3.18 x 10^6 J/kg of energy are required to generate 73% w/w and 100% w/w from 50% w/w caustic soda solution respectively. Patel (2009) noted that the energy requirement cost of the chlor–alkali caustic soda processing represents 60 to 70% of the production cost.

As a means of finding alternatives to the conventional multiple effect evaporators, some efforts geared towards evaporation of caustic soda solution catholytes are available (Olufemi *et al.*, 2012a, 2012b, Olufemi and Ayomoh, 2019). The spray dryer technique seeks to eliminate most of the limitations in the traditional multiple effect evaporators which are highly energy consuming, have material corrosion issues, with short term periodic replacement of materials of construction, which causes intermittent shut–downs and man–hour losses. Utilization of the spray drying technique enables contacting finely generated sprays of the solution to be evaporated in a countercurrent moving hot dry air without making physical contacts with the dryer walls, thereby reducing the quantity of moisture in the solution feed.

The contributions of response surface methodology in process development and operations cuts across various applications with resounding achievements. According to Said and Amin (2015), response surface methodology (RSM) can be viewed as a method that requires complex calculation for process optimization. The method formulates aa appropriate experimental design that incorporates all independent variables and engages the input data from the experiment to finally come up with a set of useful equations that provides output theoretical values. The output products rely on regression analysis based on controlled values of the independent variables. Finally, the dependent variable becomes predictable based on the new independent variables according to Meilgaard *et al.*, (1991) and Resurreccion (1998).

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

There are various scenarios where RSM had been applied for processes and equipment operations with the ultimate view of achieving better results. Taheri Dezfouli *et al.*, (2021) had examined two materials performances and electricity production optimal conditions with RSM. Statistical analysis confirmed that the carbon aerogel performed better than the activated carbon in power production and facilitated cathodic redox reactions. Optimization performed using RSM (Design Expert 11) enroute 13 different turns, have produced optimum conditions composed of blended oil concentration of 11%, drying temperature of 63.4°C as well as drying time, acidity, total sugars, ascorbic acid, and phenols quantity of 22.80 hrs., 1.26%, 64.2%, 5.60 mg, and 924 mg, respectively as reported by Singh *et al.*, (2021). In a reported study, the effects of main spray drying conditions like inlet air temperature, maltodextrin concentration and aspiration rate as it affects the physicochemical properties of sour cherry powder such as moisture content, hygroscopicity, water solubility index and bulk density have been investigated by employing RSM that yeilded process conditions optimization (Moghaddam *et al.*,2017).

Furthermore, RSM had been used to investigate the controlling effects of process variables on the time of drying, retention of vitamin C, ratio of rehydration and browning of dried samples for cauliflower drying (Gupta *et al.*, 2013). Their statistical analysis indicated that time of drying depends on the cauliflower initial size, temperature of drying air and velocity, but the ratio of rehydration was significantly affected by the combined effect of airflow velocity and temperature. A RSM assessment of convective dehydration and osmotic pre–drying processes on the anti–oxidant property of some Hausa variety of tomato had been investigated by Obajemihi *et. al.*, (2019). As reported, optimal process conditions required for producing the best tomato product containing a vitamin C content of 22 mg/100 g were 35.43 °Bx osmotic concentration, 23.86 °C osmotic temperature and 11.10 min osmotic time having a desirability function of 1. Exploring further, modelling and optimization of conditions needed for dog rose drying in the preparation of a functional tea using RSM had been investigated by Pashazadeh *et. al.*, (2021). The drying conditions were optimized in such a way as to have maximum antioxidant properties. Nasser *et al.*, (2015) studied the application of RSM to optimize and explore the capability of yeast extract, $CaCO_3$, $MgSO_4$ and K_2HPO_4 to maximize biobutanol production using a novel local isolate of Clostridium acetobutylicum YM1. The central composite design was employed and analysis of variance (ANOVA) was utilised to analyze the experimental data. Process optimization of spray–drying manufacture of cyclodextrin complex powder drug using RSM had been reported (Nekkanti *et. al.*, 2009). The optimization of process variables produced a significant improvement of process yields well above 90% as well as moisture content below 6% w/w value.

In this work, RSM is utilized in the investigation of a spray dryer for evaporating caustic soda solution to achieve an entirely dry particulate product through the characterized optimization of various parameters that are relevant in the equipment and operational development.

2. METHODOLOGY

Figure 1 show the schematic view of the less expensive stainless steel spray dryer. The dryer has a height of 1.8 m and a diameter of 1 m. The feed inlet solution with 50% w/w NaOH solution is introduced to the top of the spray dryer using the feed pipe. The dryer operating pressure is atmospheric. With the aid of a rotary atomizer, the caustic solution fine spray is generated and flows in a counter–current manner against heated dry air coming from the dryer bottom. Wet and cooler air flows out at the top of the dryer through the outlet pipe. The evaporated caustic soda solution product is collected at the bottom of the dryer through an opening. Well–controlled inlet air temperatures of 373, 382 and 391 K were used to contact various inlet NaOH mass flow rates with values of 0.0115, 0.0179, 0.0303, 0.0488, 0.0490, 0.0599 and 0.066 kg/s. The heater maximum capacity was 3 kW. The ambient temperature ranged from 298 to 307 K. The optimization for the drying operation was carried out by the Design Expert software Version 11.1.2.0 64–bit, Stat–Ease, Inc. 1300 Godward Street Northeast, Suite 6400 Minneapolis, MN 55413. The operational

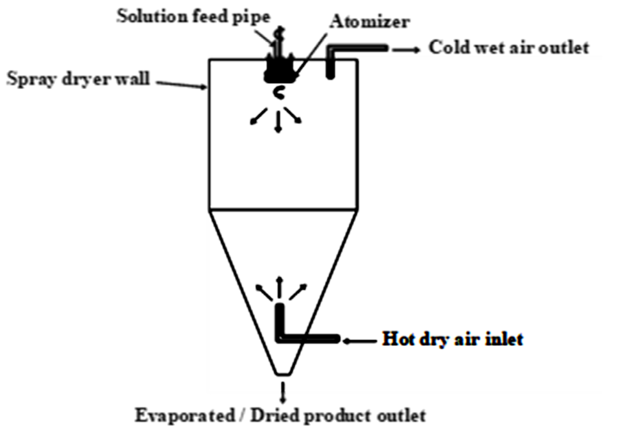

Figure 1: Schematic view of re–optimized spray dryer (Olufemi and Ayomoh, 2019)

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

variables (factors) recorded and utilised are inlet w/w% of the caustic soda solution (WBOI), inlet mass flow rate of the caustic soda solution (MCI) in kg/s, outlet mass flow rate of the caustic soda solution (MCO) in kg/s, percent moisture reduction (RED%) and specific energy of drying (ENERGY) in J/kg NaOH, while the output variable (response) is the outlet w/w% of the caustic soda solution (W%). The desired operation is to make the response W to be 99.99% or even higher.

3. RESULTS AND DISCUSSİONS

The results for the operation at air inlet temperature of 391 K is presented first, followed by the operation at 382 K, while the operation at 373 K is presented last. Table 1 gives the values of the various factors at air inlet temperature of 391 K as obtained from the experimental data of Olufemi and Ayomoh (2019).

Table 1: Experimental values for various factors at air inlet temperature of 391 K

Std	Run	Factor 1 A:WBOI	Factor 2 B:MCO (Kg/s)	Factor 3 C:ENERGY (J/KgNaOH)	Factor 4 D:RED (%)	Factor 5 E:MCI (kg/s)	Response 1 W (%)
33	1	64	0.00851888	12500	99.468	0.0115	0.7385
27	2	64	0.0131827	8070	99.656	0.0179	0.7385
18	3	62	0.0219796	4840	99.794	0.0303	0.7243
45	4	62.7	0.0355171	3000	99.872	0.049	0.7243
11	5	61.5	0.042522	2500	99.893	0.0599	0.7101
38	6	60.3	0.0459201	2320	99.901	0.066	0.6959

The build information for the experimental design at air inlet temperature of 391 K is given in Table 2 with 46 runs and quadratic type design model. Table 3 shows the factors limits, mean, standard deviations and units at air inlet temperature of 391 K. RED is ignored because the percent variation is of little or no effect. All the factors are numeric. Table 4 shows the linear model response limits, mean, standard deviations and units at air inlet temperature of 391 K.

Table 2: Build information for Design Expert calculation at air inlet temperature of 391 K.

FILE Version	11.1.2.0
Study Type	Response Surface
Design Type	Box–Behnken
Design Model	Quadratic
Subtype	Randomized
Runs	46
Blocks	No blocks

In Table 5, the ANOVA analysis is a linear model for the response W. The sum of squares is type III partial. The model F value of 30,390.87 shows that the model is significant. The p value of 0.0043 in the 95% confidence interval (CI) further confirmed the model significance. The degrees of freedom (df) values of each factors are given.

Table 3: Factors limits and units at air inlet temperature of 391 K.

Factor	Name	Unit	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	WBOI		60.30	64.00	−1↔60.30	+1↔100.00	6242	1.46
B	MCO	kg/s	0.0085	0.0459	−1↔0.01	+1↔0.50	0.2234	0.1608
C	ENERGY	J/kgNaOH	2320.00	12500.00	−1↔2320.00	+1↔20000.00	10192.50	5710.07
D	RED	%	99.47	99.90	−1↔99.47	+1↔99.90	99.69	0.1316
E	MCI	kg/s	0.0115	0.0660	−1↔0.01	+1↔0.50	0.2318	0.1551

Table 4: Response limits and units at air inlet temperature of 391 K.

Response	Name	Units	Observations	Analysis	Minimum	Maximum	Mean	Std. Dev.	Ratio	Transform
R1	W	%	6	Polynomial	0.6959	0.7385	0.7219	0.0166	1.06	None

Table 5: Analysis of Variance (ANOVA) model at air inlet temperature of 391 K.

Source	Sum of Squares	df	Mean Square	F–value	P–value	
Model	0.0014	4	0.0003	30390.87	0.0043	Significant
A–WBOI	0.0000	1	0.0000	2371.26	0.0131	
B–MCO	9.886E–06	1	9.886E–07	872.17	0.0215	
C–ENERGY	4.688E–07	1	4.688E–07	41.36	0.0982	
E–MCI	0.0000	1	0.0000	1066.29	0.0195	
Residual	1.133E–08	1	1.133E–08			
Cor Total	0.0014	5				

Table 6: The fit statistics of the model at air inlet temperature of 391 K.

Std. Dev	0.0001
Mean	0.7219
C.V. %	0.0147
R^2	1.0000
Adjusted R^2	1.0000
Predicted R^2	0.9831
Adeq Precision	438.0183

In Table 6, the predicted R^2 value of 0.9831 is in agreement with the adjusted value of 1.000, which showed a high and adequate precision of the model. The adequate precision represents a measure of the signal to noise ratio. Since the Adeq precision value of 438.0138 is much greater than the desirable value of 4, the model can be used to navigate the design space.

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

Table 7: The Coefficients in terms of coded factors at air inlet temperature of 391 K.

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	1.18	1	0.0142	0.9978	1.36	
A–WBOI	0.1108	1	0.0023	0.0819	0.1397	12.27
B–MCO	1.45	1	0.0491	0.8260	2.07	4308.34
C–ENERGY	–0.0026	1	0.0004	–0.0077	0.0025	14.94
E–MCI	–1.10	1	0.0337	–1.53	0.6722	4270.87

In Table 7, the coefficient estimate represents the expected change in response per unit change in factor value when every remaining factors are held constant. The intercept in an orthogonal design depicts the overall average response of all the runs. The coefficients are adjustments around that mean based on the factor settings. The variance inflation factor (VIF) estimates how much the model variance is inflated by the lack of orthogonality in the design. If the factor is orthogonal to all the other factors in the model, the VIF is 1.0. VIFs greater than 1.0 represents multi–collinearity, the higher the VIF, the more severe the correlation of factors. As a rough guide, VIF values less than 10 are tolerable.

The final equation given in terms of the coded factors is given in Equation (1).

$$W = 1.18 + 0.1108A + 1.45B - 0.0026C - 1.10E \quad (1)$$

The equation in terms of coded factors is useful in making predictions about the response for given levels of each factor. By default, high levels of the factors are usually coded as +1 and the low levels coded as –1. The coded equation enables identification of the relative impact of the factors by comparing their coefficients. The contributory relative importance in this case is that B>E>A>C for the drying operation at 391 K. The final equation expressed in terms of the actual factors is given in Equation (2).

$$W = 0.386510 + 0.005582(WBOI) + 5.89926(MCO) - 2.93345 \times 10^{-7}(ENERGY) - 4.50539(MCI) \quad (2)$$

The equation written as the actual factors in their original units is useful in making predictions about the response for given levels of each factor. As a precaution, the equation should not be used to determine the relative impact of each factor, as the coefficients are scaled to accommodate the units of each factor, with the intercept not at the centre of the design space.

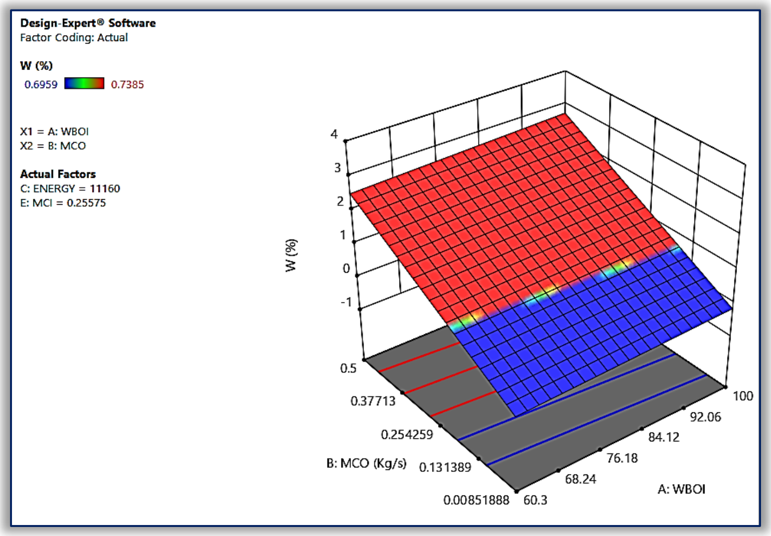

Figure 2: Three dimensional (3D) plots of actual factors in the design space at air inlet temperature of 391 K.

The 3D plots of the actual factors are shown in Figure 2. As shown W increases linearly with MCO and WBOI. The target for w the final w/w% is for a lower limit of 0.6959 and an upper limit of 0.9999 in the optimization of variables. The coded five factors given in Table 3 show the non–relevance of RED in the computation, being outside the design space. Eighteen solutions were found as depicted in Table 8 with two–side 95% confidence and 99% population. The predicted outlet w/w% of the caustic soda solution (W) has a value of 99.5364%, with associated input variables given in Table 3. Tables 9 and 10 provides tools to confirm results from the modeled response. The 95% in the predicted interval (PI) confirmed the suitability of the model.

Table 8: Point Prediction of Predicted output of spray drying optimization at air inlet temperature of 391 K.

Solution 1 of 18 Response	Predicted Means	Predicted Median	Std. Dev.	SE Mean	95% CI low for Mean	95% CI high for Mean	95% TI low for 99% pop	95% TI high for 99% pop
W	0.995364	0995364	0.000106464	0.0108562	0.857423	1.1333	0.709957	1.28077

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

Table 9: Confirmation location of predicted factors at air inlet temperature of 391 K

WBOI	MCO	ENERGY	RED	MCI
71.8235*	0.263112	9077.53	*	0.297763

*Factor value is outside of the design space.

Table 10: Confirmation location of predicted response at air inlet temperature of 391 K

Solution 1 of 18 Response	Predicted Mean	Preicted Median	Std. Dev.	n	SE Pred	95% PI low	95% PI high
W	0.995364	0.995364	0.000106464	1	0.0108567	0.857416	1.13331

Table 11: Coefficients of constants and significance at air inlet temperature of 391 K

	Intercept	A	B	C	E	AB	AC	AE	BC	BE	CE	A^2	B^2	C^2	E^2
W	1.17829	0.110794	1.44969	−0.0026	−1.1004										
p–values		0.0131	0.0215	0.0982	0.0195										

Table 11 show the coefficients and statistical significance of each based on their p–values in the 95% CI. Table 12 gives the values of the various factors at air inlet temperature of 382 K as obtained from the experimental data of Olufemi and Ayomoh (2019).

Table 12: Experimental values for various factors at air inlet temperature of 382 K

Std	Run	Factor 1 A:WBOI	Factor 2 B:MCO (kg/s)	Factor 3 C:ENERGY (J/kgNaOH)	Factor 4 D:RED (%)	Factor 5 E:MCI (kg/s)	Response 1 W (%)
31	1	60.3	0.00819123	15000	99.36	0.0115	0.7101
7	2	59	0.0121686	10100	99.57	0.0179	0.6817
20	3	57.8	0.0202557	6080	99.74	0.0303	0.6675
12	4	57.8	0.0327315	3760	99.84	0.049	0.6675
54	5	56.6	0.0391202	3150	99.87	0.0599	0.6533
48	6	56.6	0.0431087	2860	99.88	0.066	0.6533

Table 13: Build information for calculation at air inlet temperature of 382K

FILE Version	11.1.2.0
Study Type	Response Surface
Design Type	Box–Behnken
Design Model	Quadratic
Subtype	Randomized
Runs	54
Blocks	No blocks

The build information for the experimental design at air inlet temperature of 382 K is given in Table 13 with 54 runs and quadratic type design model. Table 14 shows the factors limits, mean, standard deviations and units at air inlet temperature of 382 K. RED is ignored because the percent variation is of no effect. All the factors are numeric. Table 15 shows the response limits, mean, standard deviations and units at air inlet temperature of 382 K.

Table 14: Factors limits and units at air inlet temperature of 382 K.

Factor	Name	Unit	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	WBOI		56.60	64.30	−1↔60.30	+1↔100.00	58.02	1.43
B	MCO	Kg/s	0.0082	0.0431	−1↔0.01	+1↔0.50	0.2138	0.1716
C	ENERGY	J/KgNaOH	2860.00	15000.00	−1↔2860.00	+1↔20000.00	2663E+307	INF
D	RED	%	99.36	99.88	−1↔99.47	+1↔99.90	99.68	0.1531
E	MCI	Kg/s	0.0115	0.0660	−1↔0.01	+1↔0.50	0.0391	0.10226

Table 15: Response limits and units at air inlet temperature of 382 K.

Response	Name	Units	Observations	Analysis	Minimum	Maximum	Mean	Std. Dev.	Ratio	Transform	Model
R1	W	%	6	polynomial	0.6533	0.7101	0.6722	0.0214	1.09	None	Linear

Table 16: Analysis of Variance (ANOVA) model at air inlet temperature of 382 K.

Source	Sum of Squares	df	Mean Square	F–value	P–value	
Model	0.0023	4	0.0006	3333.75	0.0130	Significant
A–WBOI	2.179E–06	1	2.179E–06	12.72	0.01741	
B–MCO	0.0000	1	0.0000	97.28	0.0643	
C–ENRGY	0.0000	1	0.0000	149.55	0.0519	
E–MCI	0.0000	1	0.0000	94.64	0.0652	
Residual	1.714E–07	1	1.714E–07			
Cor Total	0.0023	5				

In Table 16, the ANOVA analysis is a linear model for the response W. The sum of squares depicts type III partial. The model F value of 3,333.75 indicated that the model is significant. The p value of 0.0130 in the 95% CI further confirmed the model significance. The degrees of freedom (df) values of each factors are also given.

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

Table 17: The fit statistics of the model at air inlet temperature of 382 K.

Std. Dev	0.0004
Mean	0.6722
C.V. %	0.0616
R^2	0.9999
Adjusted R^2	0.9996
Predicted R^2	0.4198
Adeq Precision	151.0597

In Table 17, the predicted R^2 value of 0.4198 is not so close to the adjusted R^2 of 0.9996 as expected. The difference is more than 0.2. This indicated large block effect or a possible problem with the data or model. Possible considerations are model reduction, response transformation, outliers, and so on. Therefore, it becomes necessary to test all empirical models by confirmation runs. The adequate precision depicts the signal to noise ratio. Since the Adeq precision value of 151.0597 is much greater than the desirable value of 4, the model is useful in navigating the design space.

Table 18: The Coefficients in terms of coded factors at air inlet temperature of 382 K.

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	3.52	1	0.2502	0.3356	6.69	
A–WBOI	–0.1647	1	0.0462	–0.7514	0.4221	324.91
B–MCO	8.24	1	0.8359	–2.38	18.86	70903.73
C–ENERGY	0.0612	1	0.0050	–0.0024	0.1248	232.27
E–MCI	–5.25	1	0.5395	–12.10	1.61	72406.82

In Table 18, the calculated coefficients depicts expected changes in response per unit change in factor value in a situation whereby all remaining factors are held constant. The intercept represents an orthogonal design, which is an overall mean response of all the runs. The coefficients are adjustments around that mean based on the factor settings. When the factors are orthogonal, the VIFs are 1. VIF values higher than 1 points to multi–collinearity. The higher the VIF, the more severe the factors' correlation. As a rough guide, VIFs less than 10 are tolerable.

The final equation presented with coded factors is given in Equation (3).

$$W = 3.52 - 0.1647A + 8.24B + 0.0612C - 5.25E \quad (3)$$

As usual, the high levels of the factors are coded as +1, while the low levels are coded as –1. The coded equation is useful for the identification of the relative impact of the factors by comparing their coefficients. The contributory relative importance in this case is that B>C>A>E for the drying operation at 382 K.

The final equation presented as the actual factors is given in Equation (4).

$$W = 1.07570 - 0.008295(WBOI) + 33.52511(MCO) + 7.14092 \times 10^{-6}(ENERGY) - 21.48875(MCI) \quad (4)$$

The equation in terms of the actual factors in their original units is effective for making predictions about the response for given levels of each factor.

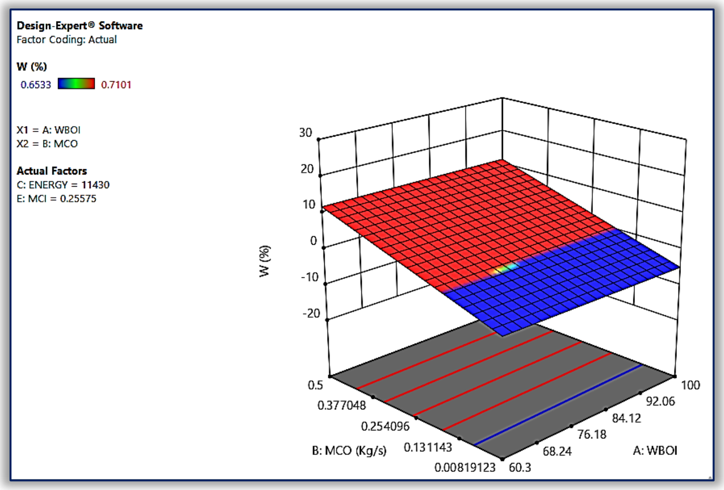

Figure 3: Three dimensional (3D) plots of actual factors in the design space at air inlet temperature of 382 K.

The 3D plots of the actual factors are shown in Figure 3. As shown W increases linearly with MCO and WBOI. The target for w the final w/w% is for a lower limit of 0.6533 and an upper limit of 0.9999 in the optimization of variables. Three solutions were found.

The coded five factors are given in Table 14, showing the non–relevance of RED in the computation, being outside the design space.

Table 19: Predicted output of spray drying optimization at air inlet temperature of 382 K.

Solution 1 of 3 Response	Predicted Means	Predicted Median	Std. Dev.	SE Mean	95% CI low for Mean	95% CI high for Mean	95% TI low for 99% pop	95% TI high for 99% pop
W	0.931675	0.931675	0.000413956	0.0401667	0.421308	1.44204	–0.125962	1.98931

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

Three solutions were found as depicted in Table 19. The predicted outlet w/w% of the caustic soda solution (W) has a value of 93.1675%, with associated input variables given in Table 14. Tables 20 and 21 confirmed results from the modelled response. The 95% in the predicted interval (PI) for 99% population confirmed the suitability of the model. The predicted mean was smaller than that obtained for the operation at 391 K.

Table 20: Confirmation location of predicted factors at air inlet temperature of 382 K

WBOI	MCO	ENERGY	RED	MCI
95.6125*	0.15832	6006.53	*	0.218787

* Factor value is outside of the design space.

Table 21: Confirmation location of predicted response at air inlet temperature of 382 K

Solution 1 of 3 Response	Predicted Mean	Preicted Median	Std. Dev.	n	SE Pred	95% PI low	95% PI high
W	0.931675	0.931675	0.000413956	1	0.0401689	0.421281	1.44207

Table 22: Coefficients of constants and significance at air inlet temperature of 382 K

	Intercept	A	B	C	E	AB	AC	AE	BC	BE	CE	A^2	B^2	C^2	E^2
W	3.51527	–0.1647	8.24397	0.0612	–5.25										
p–values		0.1741	0.0643	0.0519	0.0652										

Table 22 show the coefficients and statistical significance of each based on their p–values in the 95% CI at air inlet temperature of 382 K. Table 23 gives the values of the various factors at air inlet temperature of 373 K as obtained from the experimental data of Olufemi and Ayomoh (2019).

Table 24: Build information for calculation at air inlet temperature of 373 K.

File Version	11.1.2.0
Study Type	Response Surface
Design Type	Box–Behnken
Design Model	Quadratic
Subtype	Randomized
Runs	54
Blocks	No blocks

Table 23: Experimental values for various factors at air inlet temperature of 373 K

Std	Run	Factor 1 A:WBOI	Factor 2 B:MCO (kg/s)	Factor 3 C:ENERGY (J/kgNaOH)	Factor 4 D:RED (%)	Factor 5 E:MCI (kg/s)	Response 1 W (%)
31	1	60.3	0.00802741	17900	99.23	0.0115	0.6959
7	2	59	0.0121686	1180	99.5	0.0179	0.6817
20	3	57.8	0.0202557	7090	99.7	0.0303	0.6675
12	4	57.8	0.0327315	4390	99.81	0.049	0.6675
54	5	56.6	0.0391202	3670	99.84	0.0599	0.6533
48	6	55.4	0.0421715	3400	99.86	0.066	0.6391

The build information for the experimental design at air inlet temperature of 373 K is given in Table 24 with 54 runs and quadratic type design model. Table 25 shows the factors limits, mean, standard deviations and units at air inlet temperature of 373 K. RED is ignored because the percent variation is of little or no effect. All factors considered are numeric in nature.

Table 25: Factors limits and units at air inlet temperature of 373 K.

Factor	Name	Unit	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	WBOI		55.40	64.30	–1↔55.40	+1↔100.00	57.82	1.73
B	MCO	kg/s	0.0080	0.0422	–1↔0.01	+1↔0.50	0.2138	0.1717
C	ENERGY	J/kgNaOH	1180.00	17900.00	–1↔3400.00	+1↔20000.00	2663E+307	INF
D	RED	%	99.23	99.86	–1↔99.47	+1↔99.90	99.67	0.1593
E	MCI	kg/s	0.0115	0.0660	–1↔0.01	+1↔0.50	0.0391	0.10226

Table 26: Response limits and units at air inlet temperature of 373 K.

Response	Name	Units	Observations	Analysis	Minimum	Maximum	Mean	Std. Dev.	Ratio	Transform	Model
R1	W	%	6	Polynomial	0.6391	0.6959	0.6675	0.0201	1.09	None	Linear

Table 27: Analysis of Variance (ANOVA) model at air inlet temperature of 373 K.

Source	Sum of Squares	df	Mean Square	F–value	P–value	
Model	0.0020	4	0.0005	1.5×10^6	0.0006	Significant
A–WBOI	0.0001	1	0.0001	2.45×10^5	0.0013	
B–MCO	9.162E–08	1	9.162E–08	272.65	0.0385	
C–ENERGY	4.676E–07	1	1.676E–07	498.62	0.0285	
E–MCI	9.401E–08	1	9.401E–08	1279.76	0.0380	
Residual	3.360E–08	1	3.360E–10			
Cor Total	0.0020	5				

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

In Table 27, the ANOVA analysis is a linear model for the response W. The sum of squares is type III partial. The model F value of 1.5 x 10^6 shows that the model is highly significant. The p value of 0.0006 in the 95% CI further confirmed the model significance. The degrees of freedom (df) values of each factors are also given.

Table 28: The fit statistics of the model at air inlet temperature of 373 K.

Std. Dev	0.0000
Mean	0.6675
C.V. %	0.0027
R^2	1.0000
Adjusted R^2	1.0000
Predicted R^2	0.9991
Adeq Precision	3394.7170

In Table 28, the predicted R^2 with value of 0.9991 is in agreement with the adjusted value of 1.000, which showed a high and adequate precision of the model. The adequate precision measures the signal to noise ratio. Since the Adeq precision value of 3394.7170 is much greater than the acceptable value of 4, the model is suitable to navigate the design space at air inlet temperature of 373 K.

In Table 29 as stated earlier, the coefficient estimation represents the change expected in response to a unit change in factor value when every remaining factors are held constant. In an orthogonal design, the intercept represents the overall average response of all the runs. The coefficients represents adjustments around the mean based on the factor settings. The variance inflation factor (VIF) is a measure of how much the variance of the model is inflated by orthogonality lack in the design. In a case where the factor is orthogonal to all the other factors in the model, the VIF has a value of 1.0. VIFs that are greater than 1.0 points towards multi–collinearity. The higher the VIF, the more severe the factors correlation. As a rough guide, VIFs that are less than 10 are usually tolerable.

Table 29: The Coefficients in terms of coded factors at air inlet temperature of 373 K.

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	0.9299	1	0.0019	0.9054	0.9544	
A–WBOI	0.2555	1	0.0005	0.2489	0.2620	23.73
B–MCO	0.1067	1	0.0065	0.0246	0.1888	2111.51
C–ENERGY	–0.0004	1	0.0000	–0.0006	–0.0002	2.44
E–MCI	–0.0726	1	0.0043	–0.1277	–0.0174	2388.00

The final equation in terms of coded factors is given in Equation (5).

$$W = 0.9299 + 0.2555A + 0.1067B - 0.0004C - 0.0726E \qquad (5)$$

The equation as it apear in terms of coded factors enables predictions about the response for given levels of each factor. The high levels of the factors are usually coded as +1, while the low levels are coded as –1. The coded equation identifies the relative impact of the factors by comparing their coefficients. The contributory relative importance in this case is that A>B>C>E for the drying operation at 373 K.

The final equation written as the actual factors is given in Equation (6).

$$W = 0.005936 + 0.011455(WBOI) + 0.434241(MCO) - 4.75693 \times 10^{-8}(ENERGY) - 0.297120(MCI) \qquad (6)$$

The equation written as the actual factors in their original units is useful in making predictions about the response for given levels of each factor. However, the use of the equation to determine the relative impact of each factor is not encouraged because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the design space center.

The 3D plots of the actual factors are shown in Figure 4. As shown W increases linearly with MCO and WBOI. The target for w the final w/w% is for a lower limit of 0.6391 and an upper limit of 0.9999 in the optimization of variables. Sixty nine solutions were found.

The coded five factors are given in Table 23, showing the non–relevance of RED in the computation, being outside the design space.

Table 30: Predicted output of spray drying optimization at air inlet temperature of 373 K.

Solution 1 of 69 Response	Predicted Means	Predicted Median	Std. Dev.	SE Mean	95% CI low for Mean	95% CI high for Mean	95% TI low for 99% pop	95% TI high for 99% pop
W	0.994206	0.994206	1.83311E–05	0.000766252	0.98447	1.00394	0.973199	1.01521

Sixty nine solutions were found as presented in Table 30. The predicted outlet w/w% of the caustic soda solution (W) has a value of 99.4206%, with associated input variables given in Table 25. Tables 31 and 32 confirmed results from the modelled response. The 95% in the predicted interval (PI) for a population of 99% confirmed the suitability of the model.

Table 31: Confirmation location of predicted factors at air inlet temperature of 373 K

WBOI	MCO	ENERGY	RED	MCI
86.3061*	0.144139	5229.91	99.8385	0.211105*

.* Factor value is outside of the design space

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN-L 1584 – 2665

Table 32: Confirmation location of predicted response at air inlet temperature of 373 K

Response	Predicted Mean	Predicted Median	Std. Dev.	n	SE Pred	95% PI low	95% PI high
W	0.994206	0.994206	0.000106464	1	0.0108567	0.857416	1.13331

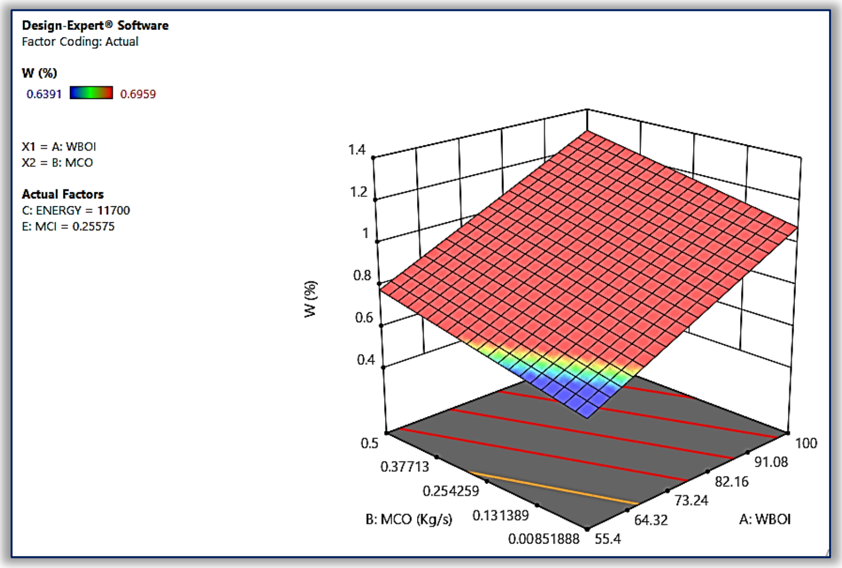

Figure 33: Three dimensional (3D) plots of actual factors in the design space at air inlet temperature of 373 K.

Table 34: Coefficients of constants and significance at air inlet temperature of 373 K

	Intercept	A	B	C	E	AB	AC	AE	BC	BE	CE	A^2	B^2	C^2	E^2
W	0.9299	0.2555	0.1067	−0.0004	−0.0726										
p–values		0.0013	0.0385	0.0285	0.0380										

Table 34 show the coefficients and statistical significance of each coefficient based on their p–values in the 95% CI at air inlet temperature of 373 K.

Comparing Tables 8, 19 and 30, the operation at 391 K yielded the highest w/w% of the spray drying operation with a value of 0.995364 as well as associated factors or variables needed to achieve the best optimum operation.

4. CONCLUSIONS

The numerical optimization of the spray dryer operations at three inlet air temperatures of 391, 382 and 373 K revealed various operating characteristics, performances and result outputs. All the generated operational models were suitable for the design and operation from the results obtained. The operational variables were also specified accordingly within reasonable limits. The percent RED is the only variable ignored in the analysis because for all the operations, the difference from the highest and lowest limits is even smaller than 0.0067%. The operation at 391 K yielded the highest w/w% of the spray drying operation as well as associated factors or variables needed to achieve the best operation. The realistic preferred optimum parameters obtainable are 71.82%, 0.2631 kg/s, 9077.53 kJ/kg, 0.2978 kg/s and 99.5364% for the inlet w/w% of the caustic soda solution, outlet mass flow rate of the dried product, specific drying energy per kg of NaOH required, inlet mass flow rate of NaOH solution and final w/w% of the NaOH dried product at inlet air temperature of 391 K respectively. With the usage of this current technique compared to the traditional usage of multiple effect evaporators to achieve 100% w/w NaOH, the improvement on the energy savings achieved was approximately 3.18×10^6 J/kg of energy, representing over 99.66% specific energy savings.

Acknowledgement

The Central Research Committee (CRC) of the University of Lagos, Akoka, Nigeria gave the financial support to accomplish this work. The provision is appreciated. Thanks to Issac Alipoe for assisting with some computing tasks.

References

[1] Gupta, M. K., Sehgal, V. K., and Arora, S. (2013). Optimization of drying process parameters for cauliflower drying. Journal of food science and technology, 50(1), 62–69.
[2] Meilgaard, B. T, Civille, M. and Carr, G. V., (1991). Sensory evaluation techniques, 2nd ed., CRC Press, Boca Raton, FL.
[3] Moghaddam, A. D., Pero, M., and Askari, G. R. (2017). Optimizing spray drying conditions of sour cherry juice based on physicochemical properties, using response surface methodology (RSM). Journal of food science and technology, 54(1), 174–184.
[4] Nasser Al–Shorgani, N. K., Mohd Isa, M. H., Yusoff, W. M. W., Kalil, M. S., and Hamid, A. A. (2015). Response surface methodology for biobutanol optimization using locally isolated Clostridium acetobutylicum YM1. International journal of green energy, 12(12), 1236–1243.

[5] Nekkanti, V., Muniyappan, T., Karatgi, P., Hari, M. S., Marella, S., and Pillai, R. (2009). Spray–drying process optimization for manufacture of drug–cyclodextrin complex powder using design of experiments. Drug development and industrial pharmacy, 35(10), 1219–1229.

[6] Obajemihi, O. I., Olaoye, J. O. Sanisi, M. S., Akpenpuun, T. D., Salawu, K. M., Asipa, A. A., and Dikko, D. O. (2019). Response surface methodology assessment of osmotic pre–drying and convective dehydration processes on the anti–oxidant property of Hausa variety of tomato. Croatian Journal of Food Science and Technology, 11(2), 187–194.

[7] Olufemi, B. A, Popoola, G., Towobola, O. and Awosanya, O., (2012a). Mathematical modelling of a reduced thermal energy consuming spray dryer for evaporating caustic soda solution, Research Journal of Applied Sciences, Engineering and Technology (RJASET), 4(11), 1550 – 1556.

[8] Olufemi, B. A., Popoola, G. O, Towobola O. R and O. G. Awosanya, (2012b). Operational characterization of a spray dryer for drying water, caustic soda and sodium chloride solutions, ARPN Journal of Engineering and Applied Sciences, 7(2), 222 – 228.

[9] Olufemi, B. A., and Ayomoh, M. K. (2019). Parametric optimization and statistical evaluation of a spray dryer for the evaporation of caustic soda solution. Heliyon, 5(7), e02026.

[10] Pashazadeh, H., Zannou, O., and Koca, I. (2021). Modeling and optimization of drying conditions of dog rose for preparation of a functional tea. Journal of Food Process Engineering, 44(3), e13632.

[11] Resurreccion, A.V.A., (1998). Quantitative of quality attributes as perceived by the consumer. In Consumer sensory testing for product development, Aspen Publishers, Inc., Gaithersburg, MD.

[12] Said, K. A. M., and Amin, M. A. M. (2015). Overview on the response surface methodology (RSM) in extraction processes. Journal of Applied Science & Process Engineering, 2(1), 8–17.

[13] Singh, G., Kaushal, N., Tokusoglu, O., and Singh, A. (2021). Optimization of process parameters for drying of red Grapes (Vitis vinifera) to raisin: A design expert laden approach. Journal of Food Processing and Preservation, e15248.

[14] Taheri Dezfouli, T., Tabrizi, N. S., Emtyazjoo, M., Javaheri, M., Marandi, R., and Kashefiolasl, M. (2021). Response surface methodology to investigate the comparison of two carbon–based air cathodes for bio–electrochemical systems. Environmental Technology, 1–15.

[15] Tilak, V. B, Orosz, P. J., and Sokol, E. A., (2007). Brine Electrolysis, http://electrochem.cwru.edu/ed/encycl. (accessed 5 September 2022).

[16] Worrel, E., Phylipsen, D., Einstein D. and N. Martin. (2000). Energy Use and Energy Intensity of the U.S. Chemical Industry, http://ies.lbl.gov/iespubs/44314.pdf, pp 1–40. (accessed 7 March 2022).

ISSN 1584 – 2665 (printed version); ISSN 2601 – 2332 (online); ISSN–L 1584 – 2665