

## STATISTICAL STUDY OF EFFECTIVE VARIABLES ON WATER CONTENT OF THE DRIED GASOIL USING A VACUUM DRYER

Elhameh Narimani, Javad Alaei Kadijani

*Gas Department, Research Institute of Petroleum Industry, West Boulevard of Azadi Stadium, P.O. Box 14665-1998, Tehran, Iran, E-mail address: JAVAD.ALAEI@ripi.ir*

Received November 23, 2013, Accepted December 16, 2013

---

### Abstract

It is considered that the water content of the dried gasoil using vacuum dryer is largely dependent on temperature, residence time and pressure of the drying process in which the gasoil is dried. In this study, a laboratorial vacuum dryer was applied to investigate the dehydration process of gasoil. The experiment design was carried out by Face Center Central Composite (FCC) design of Response Surface Methodology (RSM). Consequently, a second order quadratic model of gasoil water content was obtained estimating the relationship between water content and three independent variables of temperature, pressure and residence time. It was shown that pressure has the greatest effect on water content of the dried gasoil. The optimal values of these variables were determined in order to achieve the minimum amount of water content of the dehydrated gasoil. Based on results, the minimum amount of gasoil water content was approximately 0.0013% in which the process parameters were calculated to be 80°C temperature, 6.04 minutes residence time and 7.22 cmHg pressure.

**Keywords:** Gasoil dehydration; Vacuum dryer; Experiment Design; Response Surface Methodology; Face Center Central Composite design; Optimization.

---

### 1. Introduction

Vacuum dehydration is among the most efficient industrial processes used in different chemical plants to dry products whose structures are sensitive to high temperature like food, drugs or even petroleum products. In this process, heat and vacuum are simultaneously applied to promote the evaporation of water from the food or other products. Vacuum drying can produce high-quality product however, it is a costly and time-consuming process, which requires accurate designing and optimization.

In addition to food and drug industry, vacuum dryers are widely used in petroleum industries and refineries especially sweetening units including Hydrodesulphurization and Demercaptanization plants. As it was shown in OSHA Technical Manual, some mercaptans are removed by water-soluble chemicals that react with the mercaptans [1]. In this process, drying is required to remove water from the products. This drying step can significantly influence all the parts of sweetening plant and improve the product quality.

The water content is considered as one of the hydrocarbon contaminants that can exerts major effects on hydrocarbon system. The corrosion is definitely the most apparent influence that the hydrocarbon water content has on surfaces [2-3].

Glancing at some troubles, which can be caused by extra water of petroleum products in refineries, one can realize that petroleum industries need to reduce water content of hydrocarbons. Regarding the research paper by K. Pater [4], there are different devices to dehydrate hydrocarbon products - dewatering using Coalescer filter, dewatering using vacuum dryer, and inert gas stripping to name but a few. However, vacuum dryers reduce water content of hydrocarbon more effectively than other types of dryers. Therefore, the aim of the current study was to investigate a type of laboratorial vacuum dryer used to dry gasoil.

Drying is one of the most important and most energy-consuming industrial operations. Indeed, it is a combination of material science and transport phenomena. However, our knowledge about drying at microscopic level is still rudimentary as it was mentioned by A. S. Mujumdar.

The computer-based modeling will play an important part in modeling of drying. In addition, mathematical models can facilitate the scale-up and optimization of dryers' operating conditions.

Generally, modeling of dryers requires two sub-models: a drying process model and a dryer model, the former deals with the drying characteristics by which the materials are dried and the latter with the dryer's condition in which the material is dehydrated affecting the heat and mass transfer rates and residence times of process in dryer [5]. Focusing on simulation and optimization of dryers, this group of models contains some statistical techniques and algorithms, namely, RSM, Neural Network, and Genetics Algorithm. In one study conducted by M. Zhang *et al.* [6], RSM was used to optimize preservation of Selenium in sweet pepper under low-vacuum dehydration. The gelatin-microcrystalline cellulose model of food system was used in a study by V. A.E. King *et al.* [7] in which the effects of solid concentration, drying temperature, and sample thickness on various responses were studied applying RSM. Another study investigated the effect of process variables on osmotic dehydration of Okra in sucrose solution based on RSM with Central Composite Rotatable Design (CCRD) [8]. Defining a relationship between input and output parameters, RSM uses regression analysis to find effective factors on chemical processes and optimizes them. Fittingly, RSM was used in several studies [9-22].

Regarding the importance of mathematical models in optimization of dryers, present study focused on optimization of gasoil dehydration process in a laboratorial vacuum dryer. In order to evaluate the effect of three variables of temperature, pressure and residence time on gasoil water content, RSM was used which led to a second-order quadratic model of water content. Subsequently this work aimed to optimize the water content of the dehydrated gasoil.

## 2. The experimental methods

### 2.1 Experimental set-up

In this work, in order to study the effect of three independent variables on the water content of dried gasoil, a small laboratory-scale setup was applied which was shown in Fig.1. Gasoil with 0.1 percent (wt) of water content was used as a feed for the vacuum dryer in experiments.

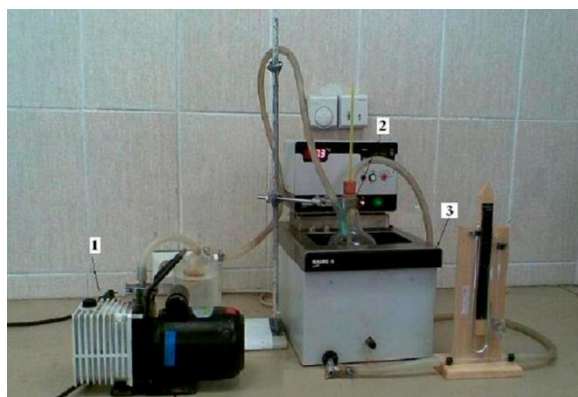


Figure 1 The vacuum dryer set-up

According to Fig. 1, the set-up was made up of a vacuum pump (1), a glass balloon with 3 outlets (2) and an oil bath (3). Each experiment was conducted for a determined temperature, residence time and pressure. In this condition, gasoil lost the large amount of its water content. At the end of each test, a sample was taken from balloon in order to analyze the dried gasoil for its water content.

The main aim of this study was to derive a mathematical model to estimate relationship between gasoil water content as a response, and three independent variables of residence time, temperature, and pressure that had to be used to minimize the water content of gasoil after dehydration in vacuum dryer.

### 2.2 Experimental design and statistical analysis

It was assumed that gasoil water content was affected by three independent variables of temperature, pressure, and residence time. The experiments were conducted under different conditions of these three factors at three levels defined based on FCC design of RSM.

The main advantage of FCC design in comparison with full factorial design, which involves  $3^3 = 27$  different experimental tests, is a significant decrease in the number of experiments. According to the FCC design for three factors, only 15 different experiments were required. These 15 points include 8 factorial points (a cube's vertices), 6 axial points and one centre point that were coded with the value of 0 as it was shown by K. Hinkelmann *et al.* [9]. Furthermore, for each point two different tests were conducted. Each independent coded variable had 3 levels of -1, 0 and +1. In Tab. 1 the high and low level of these three factors are presented.

Table1. Low and high levels of the factors

Independent variables	Coded levels		
	-1	0	1
T(°C)= $X_1$	80	90	100
Residence Time(min.)= $X_2$	3	5	7
P(cmHg)= $X_3$	7	10	13

The critical range of each independent variable was defined based on several preliminary experiments. Regarding the results of various experiments, the useful ranges of temperature, pressure and residence time were defined to be between 80-100°C and 3-7cmHg, 7-13 minutes, respectively.

The RSM fitted experimental data from FCC design into a second-order polynomial as given in Eq. (1). The design was generated by Minitab15 software and the unknown parameters of a mathematical model were estimated by least square regression analysis.

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{i < j}^3 \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

where Y is a response defined as gasoil water content.  $\beta_i$ ,  $\beta_{ii}$  and  $\beta_{ij}$  represent regression coefficients for the linear, quadratic and interaction terms and  $\varepsilon$  is the error.

There are two sources of error including modeling and experimental errors. Due to the use of experimental data in this study, the error  $\varepsilon$  is only due to the weakness of experiments as it was shown in the research conducted by S. Ghosh *et al.* [11] and A. I. Khuri *et al.* [14]. Three independent variables were indicated as temperature ( $X_1$ ), residence time ( $X_2$ ), and pressure ( $X_3$ ).

The coded values of independent variables were found from the following equations:

$$x_1 = \frac{X_1 - \bar{X}_1}{1/2(X_{1H} - X_{1L})} = \frac{X_1 - 90}{10} \quad (2)$$

$$x_2 = \frac{X_2 - \bar{X}_2}{1/2(X_{2H} - X_{2L})} = \frac{X_2 - 5}{2} \quad (3)$$

$$x_3 = \frac{X_3 - \bar{X}_3}{1/2(X_{3H} - X_{3L})} = \frac{X_3 - 10}{3} \quad (4)$$

Tab. 2 shows the values used for the FCC design.

Table 2. Central composite face-centered design with three independent variables

RUN	$x_1$	$x_2$	$x_3$	RUN	$x_1$	$x_2$	$x_3$
1	-1	-1	-1	9	-1	0	0
2	+1	-1	-1	10	+1	0	0
3	-1	+1	-1	11	0	-1	0
4	+1	+1	-1	12	0	+1	0
5	-1	-1	+1	13	0	0	-1
6	+1	-1	+1	14	0	0	+1
7	-1	+1	+1	15	0	0	0
8	+1	+1	+1				

### 3. Results and Discussion

#### 3.1 Analysis of Variance (ANOVA)

The results of experiments with respect to the water content of the dried gasoil were summarized in Tab. 3 for 15 different combinations of independent factors and 2 replications of each point. The coefficients of Eq. (1) called the second-order response surface model were calculated via multiple regressions methods using Minitab15 software. All coefficients regardless of their probability values were included in the response surface model. Tab. 4 shows these coefficients and their probability values for the equation of the water content. According to this table, the coded second-order model for gasoil water content was obtained as follows:

Table 3. Experimental tests results for gasoil water content

RUN	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Y <sub>N</sub> (%)	
				N=1	N=2
1	80	3	7	0.0036	0.00365
2	100	3	7	0.00181	0.00187
3	80	7	7	0.0016	0.00155
4	100	7	7	0.00153	0.00152
5	80	3	13	0.00979	0.00983
6	100	3	13	0.00446	0.00446
7	80	7	13	0.00567	0.00568
8	100	7	13	0.00259	0.00262
9	80	5	10	0.00367	0.00367
10	100	5	10	0.0014	0.0014
11	90	3	10	0.0056	0.00567
12	90	7	10	0.00302	0.00306
13	90	5	7	0.00282	0.00286
14	90	5	13	0.0063	0.00655
15	90	5	10	0.00332	0.00338

$$Y = 0.00367 - 0.0013x_1 - 0.001x_2 + 0.0016x_3 - 0.0012x_1^2 + 0.0006x_2^2 + 0.0009x_3^2 + 0.0005x_1x_2 - 0.0008x_1x_3 - 0.0005x_2x_3 \quad (5)$$

Moreover, the uncoded second order quadratic model obtained as follows:

$$Y = -0.09 + 0.0022X_1 - 0.0035X_2 + 0.0015X_3 - 0.000012X_1^2 + 0.000145X_2^2 + 0.000097X_3^2 + 0.000025X_1X_2 - 0.000027X_1X_3 - 0.000076X_2X_3 \quad (6)$$

Table 4. Estimated regression coefficients for dehydration efficiency using data in coded variables

Term	Coefficient	Standard Error	F-Value	Prob(F)	Remarks
Constant	0.0036	0.000071	51.904	0.000	Significant
X <sub>1</sub>	-0.0013	0.000042	-30.052	0.000	Significant
X <sub>2</sub>	-0.0011	0.000042	-26.272	0.000	Significant
X <sub>3</sub>	0.0018	0.000042	42.162	0.000	Significant
X <sub>1</sub> X <sub>1</sub>	-0.0012	0.000082	-14.870	0.001	Significant
X <sub>2</sub> X <sub>2</sub>	0.0006	0.000082	7.070	0.002	Significant
X <sub>3</sub> X <sub>3</sub>	0.00085	0.000082	10.661	0.003	Significant
X <sub>1</sub> X <sub>2</sub>	0.0005	0.000047	10.788	0.003	Significant
X <sub>1</sub> X <sub>3</sub>	-0.0008	0.000047	-17.685	0.000	Significant
X <sub>2</sub> X <sub>3</sub>	-0.0005	0.000047	-9.742	0.003	Significant

$$R^2 = 99.57\%, Ra^2 = 98.78\%$$

The first assumption to find unknown coefficients of a polynomial in regression is that the coefficients equal zero. Consequently, the less probability value for each parameter, the more significant they are in an estimated model. It means when the probability value of a factor is

greater than 0.05, the influential degree of this factor is less than 95% confidence level. In this regression, all the factors were significant in the gasoil water content.

It should be pointed out that Eq. (5) was valid in the defined range of involved variables and for a specified set-up, which was applied in this study.

In order to examine the ordinary least squares assumption, the normal probability of the residual had to be plotted using Minitab 15 software. The points in this plot had to form a straight line if the residuals were normally distributed. According to Fig.2, the normal probability plot of residual was approximately straight line for the water content of gasoil.

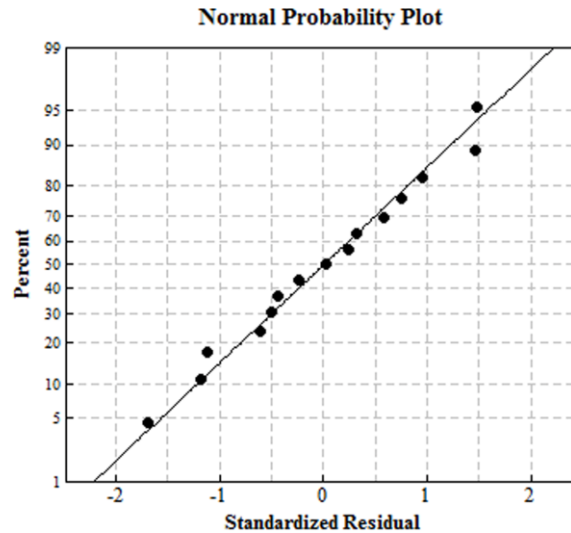


Figure 2. Normal probability plot of gasoil water content

To analyze the second-order model statistically, the corresponding analysis of variance for equation (5) is tabulated in Tab. 5. According to Tab. 5, the extremely small probability value (far smaller than 0.050) indicates that the experimental data are fitted well by the quadratic models, which is higher than the 95% confidence level. Moreover, determination coefficient ( $R^2$ ) and adjusted determination coefficient ( $Ra^2$ ) for gasoil water content were indicated at the bottom of Tab. 4.

Table 5. The analysis of variances of second-order model of gasoil water content

Source	D.F.	Seq. SS	Adj. SS	Adj. MS	F-Value	Prob(F)
Y						
Regression	9	0.000146	0.000146	0.000016	466.50	0.000
Linear	3	0.000117	0.000117	0.000039	1123.65	0.000
Square	3	0.000011	0.000011	0.000004	101.16	0.002
Interaction	3	0.000018	0.000018	0.000006	174.69	0.000
Residual error	20	0.000001	0.000001	0.000000		
Lack-of-Fit	5	0.000001	0.000001	0.000000	45.78	0.000
Pure Error	15	0.000000	0.000000	0.000000		
Total	29	0.000146				

The amount of  $R^2$  was 0.9957. This value for  $R^2$  suggests that more than 99.5 % of variations in the dependent or response variable of Y can be explained by the regression models.  $Ra^2$  was used to balance the cost of using a model with more parameters against the increase in  $R^2$  that was 0.9878.

### 3.2. The Effect of Individual Factors and Interaction of Factors on Response

In order to compare the influence of independent variables on gasoil water content at the points of the design space, the factor plot was required which was shown in Fig. 3. The effect of each factor was evaluated and plotted against gasoil water content while other factors were kept constant. According to Fig. 3, the pressure (graph C) showed the greater positive

effect on gasoil water content than other factors. The temperature and residence time (graph A and B) had the same and negative effect on gasoil water content. This fact can be understood from statistical data summarized in Tab. 4. The factors with larger F-value exert the greater influence on response equation as described in a research done by G. Kavoshi *et al.* [16].

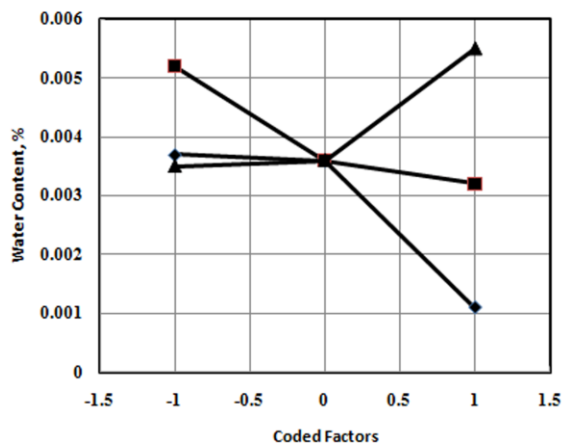


Fig. 3. Factor plot of gasoil water content

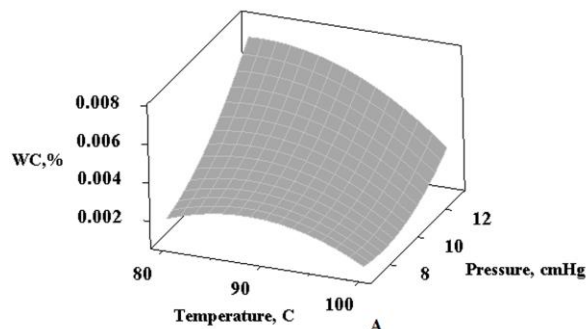


Fig. 4. 3D plot of gasoil water content at Residence time=5 minute

Tab. 4 shows that the interaction of temperature and pressure has positive effect on gasoil water content while, the interaction of pressure and temperature, residence time and pressure affected water content of dried gasoil negatively. Surface plots of water content via three independent variables are shown in Fig. 4-6.

Fig. 4 suggests that a rise in temperature resulted in a fall in gasoil water content and a decrease in pressure reduced the boiling point and consequently diminished the gasoil water content. Thus, high temperature and low pressure could keep gasoil water content down. Furthermore, the influence of the interaction of residence time and temperature on gasoil water content is shown in Fig. 5. According to this figure, an increase in temperature and residence time causes a significant fall in water content of gasoil. Besides, the effect of interaction term of pressure and residence time on gasoil water content is illustrated in Fig. 6 in which, the minimum gasoil water content was obtained at the highest residence time and the lowest amount of pressure. Although these results were roughly predictable, the mathematical models as well as the experimental procedure were missed from the large number of relevant literature. In addition, as it was shown at introduction, a mathematical model was urgently required to optimize the gasoil drying process using vacuum dryer.

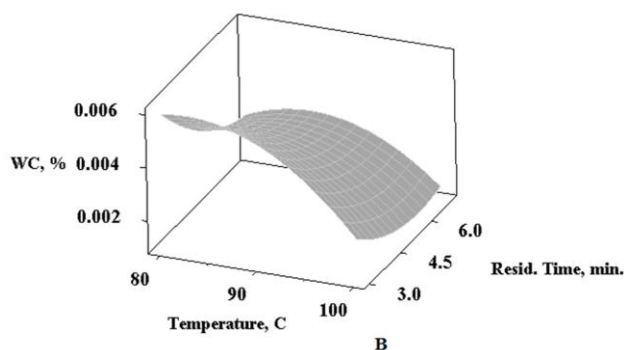


Fig. 5. 3D plot of gasoil water content at P=10cmHg

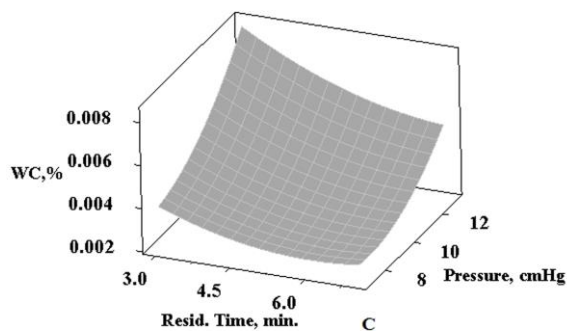


Fig. 6. 3D plot of gasoil water content at T=90°C

### 3.3 Process Optimization

The optimization of the water content of the dried gasoil was conducted using the numerical feature of the Matlab R2011b software. T. F. Eldgar *et al.* [23] and S. R. Otto [24] investigated different optimization methods using Matlab software in recent studies. Minimizing the gasoil

water content, Matlab codes calculated the optimum amount of the dehydration parameters of temperature, residence time and pressure, which were 80 °C, 6.04 minutes, and 7.22 cmHg, respectively. Based on optimization results calculated by Matlab software, at this point, the minimum amount of gasoil water content was 0.0013 % that was shown in Tab.6. In this table, the predicted amount of water content of the dried gasoil at minimum point was compared with the experimental amount of water content at this point. The results suggest that the experimental water content is slightly higher than the predicted water content at the optimum point. It means that the proposed statistical model is adequate.

Table 6. Optimal conditions

Conditions	Value
Temperature, °C	80
Residence time, minutes	6.04
Pressure, cm Hg	7.22
Predicted water content of gasoil, %	0.0013
Experimental water content of gasoil, %	0.0015

#### 4. Conclusion

In this study, the dehydration process of gasoil using vacuum dryer was studied based on Response Surface Methodology (RSM). In order to find the mathematical relationship between gasoil water content after dehydration and process parameters of temperature, residence time and pressure, 15 tests with 2 replications were conducted applying FCC design of RSM. The results suggested that temperature and residence time affected gasoil water content negatively which was the main aim of dehydration process; however, water content was positively affected by pressure. Subsequently, it was shown that it is possible to reach the lowest amount of gasoil water content in vacuum dryer, which was 0.0013% with dehydration conditions such as 80°C temperature, 6.04 minutes residence time and 7.22 cmHg pressure. It may be believed that the vacuum condition can compensate the lowness of temperature in vacuum dryers, which is considered as the great merit of vacuum dryer in drying processes of different materials.

#### 5. Acknowledgements

The financial support provided for this project by the Research Institute of Petroleum Industry (RIPI) of Iran is gratefully acknowledged.

#### List of Symbols

T	Temperature (°C),
P	Pressure (Pa),
Time	Residence Time (min.)
Y	Gasoil Water Content Response (%)
$X_i$	Coded variables, (Dimensionless)
$\bar{X}_i$	Mean value of uncoded variables, (Dimensionless)
$X_{iH}$	The high level of the $i_{th}$ factor, (Dimensionless)
$X_{iL}$	The low level of the $i_{th}$ factor, (Dimensionless)

#### Greek letters

$\beta_0$	Intercept
$\beta_i$	Linear coefficient
$\beta_{ii}$	Squared coefficient
$\beta_{ij}$	Interaction coefficient

## References

- [1] OSHA Technical Manual, Government Institutes: Bellingham, Washington, 1999.
- [2] Booser, E. R.: CRC Handbook of Lubrication: Theory and Practice of Tribology, CRC, Boca Raton, Florida, 1997.
- [3] Arizmendi, L.; Rincon, A.; Bernardo, J. M.: Tribol. Int. J., 1985, 18, 282.
- [4] Pater, K.: In Proceeding of the 15th World Pet. Congr., 1997, Beijing, China
- [5] Mujumdar, A. S.: R&D Needs Challenges and Opportunities for Innovation in Drying Technologies, Jangam, S. V.; Thorat, B. N., Eds., 2010.
- [6] Zhang, M.; Li, C.; Ding, X.; Cao, C. W.: Drying Technol. J., 2003, 21, 569.
- [7] King, V. A. E.; Zall, R. R.: Food Res. Int., 1992, 25, 1.
- [8] Agarry, S. E.; Owabor, C. N.: Niger. J. Technol., 2012, 31, 370.
- [9] Hinkelmann, K.; Kempthorne, O.: Design and Analysis of Experiments, Advanced Experimental Design, John Wiley and Sons, New York, 2009.
- [10] Narimani, E.; Shahhoseini, S.: Appl. Therm. Eng., 2011, 31, 188.
- [11] Ghosh, S.; Rao, C. R.: Handbook of statistics, Elsevier Science, Amsterdam, 1996.
- [12] Hasan, D.; Melo, M. N. C.; Fiho, R. M.: Chem. Eng. Process, 2005, 44, 335.
- [13] Bradley, N.: the Response Surface Methodology, Dissertation, Indiana University of South Bend, South Bend, Indiana, 2007.
- [14] Khuri, A. I.; Mukhopadhyay, S.: WIREs Comput. Stat., 2010, 2, 128.
- [15] Myers, R. H.; Montgomery, D. C.; Anderson cook, C. M.: Response Surface Methodology: Process and Product Optimization Using Designed Experiments, John Wiley & Sons, New York, 2009.
- [16] Kavoshi, L.; Hatamipour, M. S.; Rahimi, A.: Chem. Eng. Technol., 2012, 36, 500.
- [17] Zhao, J.; Jin, B.; Zhong, Z. J.: J. Hazard. Mater, 2007, 147, 363.
- [18] De Lima, C.; Coelho, J. B. L. F.; Contiero, J.: Food Technol. Biotechnol., 2010, 48, 175.
- [19] Kafuku, G.; Tat Tan, K.; Lee, K. T.; Mbarawa, M.: Chem. Eng. Technol., 2011, 34, 1827.
- [20] Cheyner, V.; Feinberg, M.; Chararas, C.: Appl. Environ. Microbiol., 1983, 45, 634.
- [21] Yang, Y.: Multiple Criteria Third-Order Response Surface Design and Comparison, Dissertation, Famu- FSU collage of engineering, Florida State University, Florida, the US, 2008.
- [22] Huang, L.; Lu, Z.; Yuan, Y.; Lu, F.; Bie, X.: J Ind. Microbiol. Biotechnol., 2006, 33, 55.
- [23] Eldgar, T. F.; Himmelblau, D. M.: Optimization of Chemical Engineering Processes, McGraw-Hill, Texas, 1989.
- [24] Otto, S. R.; Denier, J. P.: an Introduction to Programming and Numerical Methods in MATLAB, Springer, London, 2005.