Optimization of inhibition efficiencies process of polyvinylpyrrolidone using response surface methodology

A. Benchadli[a], T. Mellal[b], T. Attar[a,c], B. Dali Youcef[b,*] and E. Choukchou-Braham[a]

[a]*Laboratory of Toxicomed, University Abou Beker Belkaid Tlemcen, BP119, 13000 Tlemcen, Algeria.*

[b]*Laboratory of Macromolecular Research, University Abou Beker Belkaid Tlemcen, BP119, 13000 Tlemcen, Algeria.*

[*]*e-mail: boumedienedali@yahoo.com*

Tel: +213555879111

[c]*Higher School of Applied Sciences of Tlemcen, BP 165, Bel Horizon, 13000 Tlemcen, Algeria.*

Received 12 October 2021; accepted 22 December 2021

Inhibition efficiencies (IE) process in polyvinylpyrrolidone (PVP) which is influenced by independent factors, concentration and size of PVP, temperature, time of immersion and perchloric acid concentration on XC38 carbon steel was investigated in this paper. The relationship between factors and their responses is established by the concept of response surface methodology (RSM) explicitly through regression statistical analysis and probabilistic analysis is used in this work. The concept is a combination between mathematical and statistical techniques allowing the modeling and problems analysis by experimental design. In this study, the results based on statistical analysis showed that the quadratic models for the inhibition efficiencies (IE) was significant at value of probability $P < 0.0001$ and the coefficient of multiple regressions $R^2 = 0.9997$, for further validation of the model, $R^2_{Adj} = 0.9993$ indicated a good model. The experimental observed values were in good agreement with predicted ones and the model was highly significant with $Q^2 = 0.9884$. The optimal conditions of inhibition efficiencies (IE) obtained are 104.301% for a concentration of $3.55\,10^{-3}$ mol/L, temperature of $20.15°$C, immersion time of $2h$, size of PVP 58000 g/mol and acid concentration of 0.5 mol/L.

Keywords: Inhibition efficiencies; polyvinylpyrrolidone; acid perchloric; central composite face-centered design (CCFCD); response surface methodology.

DOI: https://doi.org/10.31349/RevMexFis.68.041003

1. Introduction

Polyvinylpyrrolidone (PVP), also called as polyvidone or povidone, is a water-soluble, biodegradable, biocompatible, nontoxic and non-ionic polymer, derived from its monomer N-vinylpyrrolidone [1]. PVP is essentially chemically inert, colorless, temperature resistant, and pH stable because it has unique physical and chemical characteristics. PVP is widely used in different fields, like medicine and cosmetics [1, 2], for pharmaceutical and biomedical applications [2–5]. Based on different molecular weights and modified forms, PVP can lead to exceptional beneficial features with varying chemical properties.

Response surface methodology (RSM) is a statistical experimental design that enables simultaneous variation of process variables, unlike what obtains in the conventional experimentation, thereby eliciting the interaction between such variables. It is a faster and more economical method for gathering research results than the classic one-variable at a time or full-factor experimentation [6]. It also provides a model equation relating the response parameter to the process variables and optimization of the same. It is a veritable tool that has been deployed in a wide range of fields namely; transesterification [7, 8], solvent extraction [9, 10], adsorption [11, 12], Fenton process [13], drying operations [6], carrageenan production [14] etc. The (RSM) objective is to optimize the different responses by varying the influencing factors [15]. For example, the Inhibition Efficiencies (IE) of Polyvinylpyrrolidone is affected by coefficients, Concentration X_1, Temperature X_2, Immersion Time X_3, Different Size of PVP X_4 and Perchloric Acid Concentration X_5. The inhibition efficiency of corrosion can be changed with any combination of treatment X_1, X_2, X_3, X_4 and X_5, one of those factors can vary continuously. If treatments are from a continuous range of values, the RSM is useful for optimizing the different responses variables. In our study, the inhibition efficiencies Y (the response) are function of five factors. It can be developed as the dependent variable Y of X_1, X_2, X_3, X_4 and X_5, as follows:

$$Y = f(X_1, X_2, X_3, \ldots, X_n) + e, \tag{1}$$

where e is the experimental error.

The response surface methodology is an effective statistical tool for experimental design, developing model, relationship between factors and research optimum condition [16–19]. The RSM main goals are to understand the position and topography of response surface including the minimum, maximum and ridge lines and find region where the important response occurs [20]. Two experimental designs are used in response surface methodology [21], the Box-Behnken (BBD)

and Central Composite Design (CCD). The CCD has three different design points: edge points as in two level designs (± 1), star points at $\pm\alpha$; $\mid \alpha \mid \geq 1$ that take care of quadratic effects and center points. Three variants exist: circumscribed (CCC), inscribed (CCI) and face centered (CCF). The CCC design is the original central composite design that performs five-level testing. The edge points (factorial or fractional factorial points) are at the design limits. The star points are at some distance from the center depending on the number of factors in the design. The star points extend the range outside the low and high settings for all factors. The center points complete the design and verify the reproducibility. Figure 1 illustrates a CCC design. Completing an existing factorial or resolution V fractional factorial design with star and center points leads to this design. CCC designs provide high quality predictions over the entire design space, but care must be taken when deciding on the factor ranges. Especially, it must be sure that also the star points remain at feasible (reasonable) levels.

In CCI design, the star points are set at the design limits (hard limits) and the edge points are inside the range (Fig. 2). In a ways, a CCI design is a scaled down CCC design. It also results in five levels for each factor. CCI designs use only points within the factor ranges originally specified, so the prediction space is limited compared to the CCC design.

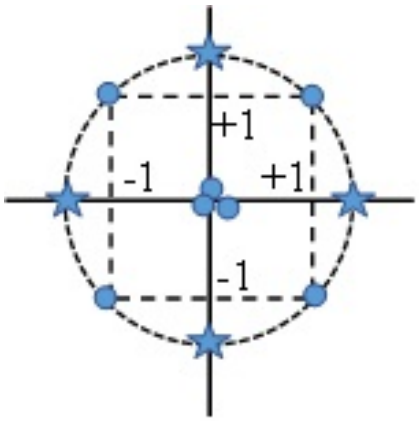

FIGURE 1. CCC design for two factors.

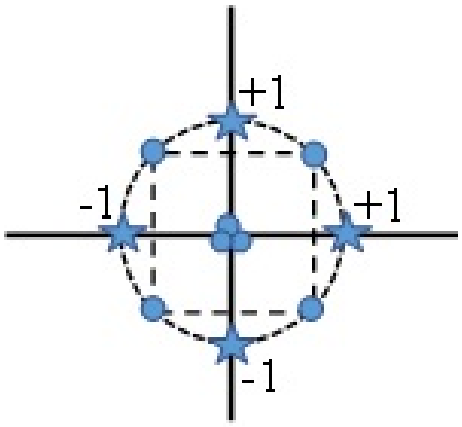

FIGURE 2. CCI design for two factors.

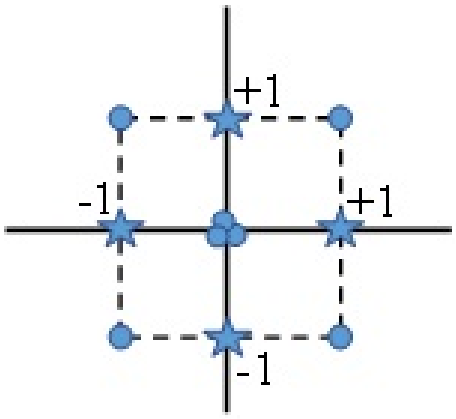

FIGURE 3. CCF design for two factors.

In CCF design the star points are at the center of each face of the factorial space, so $\alpha = \pm 1$ and only three levels are used (Fig. 3). Complementing an existing factorial or resolution V design with appropriate star points can also produce this design. CCF designs provide relatively high-quality predictions over the entire design range, but poor precision for estimating pure quadratic coefficients. They do not require using points outside the original factor range.

The experimental data are fitted by one of statistical model; Linear, Quadratic, Cubic or 2FI (two factor interaction). The linear coefficients for independent variables are A, B, and C, interactive term coefficient AB, AC and BC, quadratic term coefficient AA, BB and CC.

R^2, R^2_{Adj} coefficient and adequate precision to check the model adequacies are:

$$R^2 = 1 - \frac{SCE}{SCT}, \tag{2}$$

$$R^2_{Adj} = 1 - \frac{\frac{SCE}{N-P}}{\frac{SCT}{N-1}}, \tag{3}$$

$$SCT = \sum_{i=1}^{N} (Y_i - \bar{Y})^2, \tag{4}$$

$$SCE = \sum_{i=1}^{N} (Y_i - \hat{Y})^2, \tag{5}$$

where SCE: The sum of the squares of the residuals. SCT: The total sum of squares. (p-1) and (N-p) degrees of freedom. N: Number of experimental runs (excluding missing values). P: Number of terms in the model, including the constant. Conditions adequate model (probability value $P < 0.05$, lack of fit $P > 0.05$, R^2 and $R^2_{Adj} > 0.9$). Adeq precision> 4. The analysis of variance ANOVA [22] used to determine the differences between means.

2. Material and methods

2.1. Materials and sample preparations

Weight loss measurements were performed on the carbon steel XC38 samples with a rectangular form in perchloric acid medium with and without addition of different concentrations and size of polyvinylpyrrolidone. Carbon steel samples used as test materials contain: C: 0.37%, Mn: 0.68%, Cu: 0.16%, Cr: 0.077%, Ni: 0.059%, Si: 0.023%, S: 0.016%, Ti: 0.011%, Co: 0.009% and the balance being Fe. The samples were initially polished with emery paper starting with coarse until the mirror appearance was obtained, and were then washed with distilled water. They were cleaned again with acetone and, finally, dried using a hot air blower. Loss in weight of samples was resolved by finding the weight difference between the carbon steel substrates before and after immersion in corrosive attack. All measurements were done in triplicate, and the average value of the weight loss was noted. Each sample was weighed by an electronic balance (± 0.0001 g) and then placed in the acid solution (50 mL). The following equations were used to calculate the corrosion rate (*CR*) and the inhibition efficiencies (*IE*) [23–25].

$$CR = \frac{\Delta W}{S.t}, \tag{6}$$

$$IE(\%) = \frac{CR - CR_{Inh}}{CR}\, 100, \tag{7}$$

where ΔW is the weight loss (g); S is the total area of the specimen (cm^2); t is the exposure time (h); CR and CR_{Inh} are the corrosion rates of carbon steel samples in the absence and presence of inhibitor, respectively (g cm^{-2}h^{-1}).

2.2. CCFCD Response surface design

In our study, the Central Composite Face-Centered Design (CCFCD) based on standard surface response methodology was used to obtain the individual and interactive effects of process parameters on inhibition efficiencies of PVP. Concentration (X_1), Temperature (X_2), Time of immersion (X_3), different size of PVP (X_4) and acid perchloric concentration (X_5) were chosen as independent factors, while inhibition efficiencies (IE) was selected as response. Three levels (-1, 0, +1) for the variable range (see Table I) and five factors (see Table II below) were requiring 29 experimental runs.

The fit mode of inhibition efficiencies (IE) was modeled by a general function indicating the interaction between dependent and independent variables written as a second-degree polynomial equation,

$$\begin{aligned} Y = {} & a_0 + a_1 X_1 + a_2 X_2 + a_3 X_3 + a_4 X_4 + a_5 X_5 \\ & + a_{11} X_{11} + a_{22} X_{22} + a_{33} X_{33} + a_{44} X_{44} + a_{55} X_{55} \\ & + a_{12} X_{12} + a_{13} X_{13} + a_{14} X_{14} + a_{15} X_{15} \\ & + a_{23} X_{23} + a_{24} X_{24} + a_{25} X_{25} \\ & + a_{34} X_{34} + a_{35} X_{35} + a_{45} X_{45}, \end{aligned} \tag{8}$$

where, X_1, X_2, X_3, X_4 and X_5 are the input factors. a_0 is the intercept; a_1, a_2, a_3, a_4 and a_5 are the linear coefficients. a_{11}, a_{22}, a_{33}, a_{44} and a_{55} are the quadratic coefficients. a_{12}, a_{13}, a_{14}, a_{15}, a_{23}, a_{24}, a_{25}, a_{34}, a_{35} and a_{45} are the interaction coefficients.

3. Results and discussion

3.1. Validation and development of regression model

In the MODDE software, the residuals are plotted on a normal cumulative probability scale. This representation (Fig. 4) makes it possible to detect.

The normality of the residuals: when the residuals are normally distributed, the points describe a straight line, the aberrant values (atypical experiments): these are points of deviation from the line of normal probability, and having large absolute values of the studentized residuals [26] standard deviation indicated by red lines in Fig. 1.

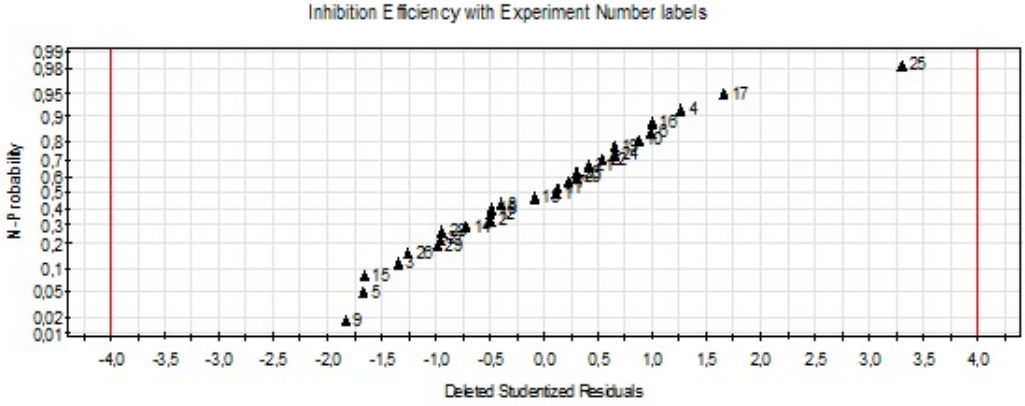

FIGURE 4. Normality of the residuals.

TABLE I. Levels of variables and factors considered for experimental design.

		Levels		
Variables	Factors	-1	0	+1
X_1	U_1 : Concentration (mol/L)	$5.0\,10^{-5}$	$2.52\,10^{-3}$	$5.0\,10^{-3}$
X_2	U_2 : Temperature (°C)	20	40	60
X_3	U_3 : Time of immersion (h)	1	2	3
X_4	U_4 : Different size (g/mol)	8000	33000	58000
X_5	U_5 : Acid concentration (mol/L)	0.5	1	1.5

TABLE II. Experimental Design Matrix of Inhibition Efficiency (Real values).

Exp. N°	X_1	X_2	X_3	X_4	X_5	IE_{Observed}	$IE_{\text{Predicted}}$
1	-1	-1	-1	-1	1	31.35	31.33
2	1	-1	-1	-1	-1	74.67	74.73
3	-1	1	-1	-1	-1	11.75	11.88
4	1	1	-1	-1	1	48.19	48.06
5	-1	-1	1	-1	-1	36.37	36.53
6	1	-1	1	-1	1	58.63	58.52
7	-1	1	1	-1	1	05.98	05.95
8	1	1	1	-1	-1	54.76	54.80
9	-1	-1	-1	1	-1	62.50	62.67
10	1	-1	-1	1	1	70.49	70.39
11	-1	1	-1	1	1	14.89	14.87
12	1	1	-1	1	-1	73.17	73.22
13	-1	-1	1	1	1	47.31	47.32
14	1	-1	1	1	-1	90.05	90.13
15	-1	1	1	1	-1	25.45	25.61
16	1	1	1	1	1	56.41	56.30
17	-1	0	0	0	0	29.24	28.65
18	1	0	0	0	0	64.68	64.89
19	0	-1	0	0	0	82.18	81.92
20	0	1	0	0	0	59.42	59.31
21	0	0	-1	0	0	59.57	59.40
22	0	0	1	0	0	58.12	57.91
23	0	0	0	-1	0	62.27	62.15
24	0	0	0	1	0	77.25	76.99
25	0	0	0	0	-1	69.51	68.63
26	0	0	0	0	1	56.03	56.53
27	0	0	0	0	0	64.65	65.14
28	0	0	0	0	0	64.63	31.33
29	0	0	0	0	0	64.64	74.73

The value 4 is specified as the limit where the associated experience can be considered to be atypical. All experiences are not atypical experiments we can conclude that the residuals are normally distributed.

According to Fig. 2 analysis we can see that the experiments values are placed perfectly on the straight line indicated the perfect quality of the mathematical model.

3.2. Statistical results

In this present work the observed Inhibition Efficiency for 29 experimental runs are presented in Table II. The data of Table V are used to determine the coefficient of the polynomial equation (see Sec. 2.2) and optimum parameter's that influenced the efficiency of polymer network. The estimated coefficients are shown in Table V.

The statistical analysis calculation was assured by analysis variance (ANOVA) given in Table III. The adequacy of the model and the determination of R^2 coefficient indicated good model. Ghasemzadeh *et al.*, [27] found that ANOVA for predicted model of antioxidant activity was significant ($F-$ value$17.21, P < 0.0001$) with a good coefficient of determination ($R^2 = 0.98$). In addition, R^2 calculation in-

TABLE III. Statistical results of experimental design model.

R^2	R^2_{Adj}	Q^2	SDY	RSD	N	Model Validity	Reproducibility
0.999797	0.99929	0.988461	21.2081	0.565237	29	-0.2	1
		DF=8	Conf. lev. =0.95	Cond. N^o=7.2			

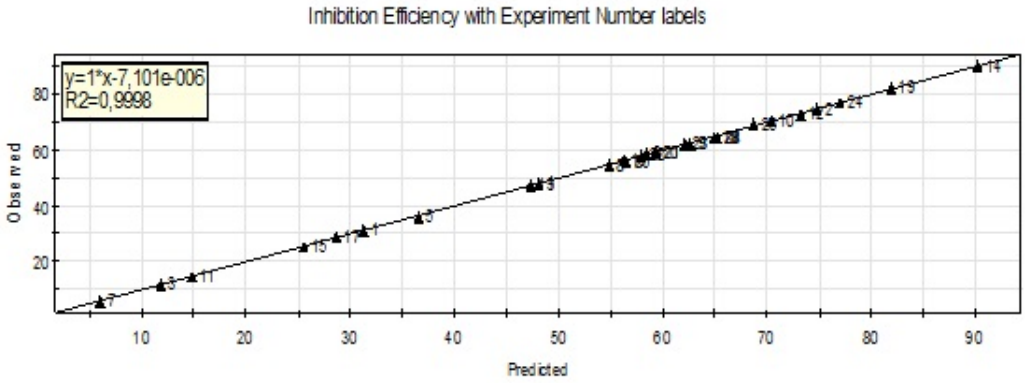

FIGURE 5. Model fit graph; relationship between the experimental and predicted values.

TABLE IV. The analysis of variance for the quadratic model of inhibition efficiency.

Inhibition Efficiency	DF	SS	MS (variance)	F	p	SD
Total	29	98041.5	3380.74			
Constant	1	85447.6	85447.6			
Total Corrected	28	12593.9	449.784			21.2081
Regression	20	1288.1	629.407	1857.65	0.000	25.088
Residual	8	2.55594	0.319493			0.565237
Lack of Fit	6	2.55574	0.425957	4256.94	0.000	0.652654
(Model Error)						
Pure Error	2	$2.00124\ 10^{-4}$	$1.00062\ 10^{-4}$			$1.00031\ 10^{-2}$
Conf. lev. =0.95						

dicates a good correlation between experiments and predicts data (using MODDE Software Version 9.1) [28].

The methods to estimate the model coefficients are Partial Least Squares (PLS) [29,30] and Multiple Linear Regression (MLR) [31]. In our study, the MLR method was used for the estimation of model coefficients.

The determination coefficients R^2, R^2_{Adj} and Q^2 are the statistical results of experimental design model presented; first R^2 (see Eq. (2) and Table III) for evaluated the correlation between two data predicted and measured experiments, the second coefficient R^2_{Adj} (see Eq. (3) and Table III) is also used to measure the quality of fit, the Q^2 coefficient shows an estimate of the future prediction precisions, their values should be greater than 0.1 for a significant model and greater than 0.5 for a good model. The Table III shows the values of R^2, R^2_{Adj} and Q^2.

Where SDY represents the Standard Deviation of the Y (response) and RSD corresponds to the Residual Standard Deviation.

Analyses of Table III revealed R^2 value of 0.99, indicated that the model can explain 99% of the data variation and 1% not explained. Chauhan and Gudta [32] emphazed in their work on the acceptance of any model with $R^2 > 0.75$, but according to Koocheki *et al.* [33] postulated that a large value of R^2 does not always imply that the regression model is good, it is necessary to check at the same time the value of R^2_{Adj}. Differently the model is adequate for prediction in the range of experimental variables. The value R^2 of 0.999797 and R^2_{Adj} of 0.99929 consequently confirmed that the model was highly significant, which designated a good agreement between predicted and experimental inhibition efficiency in PVP polymer network. In study of Rai *et al.* [34] R^2 and R^2_{Adj} should be within 20% to be in good agreement. Reproducibility is the variation of replicates compared to overall variability here a value $1 > 0.5$ is warranted. The validity of model is $-0.2 < 0.25$ indicates statistically significant model problems. The model validity might be low in very good models ($Q^2 > 0.9$) due to high sensitivity in the test or

TABLE V. Estimated regression coefficients of factors, interaction and P-values of the model.

Inhibition Efficiency	Coeff. SC	P
Constant	a_0 = 65.1385	4.92993 e^{-18}
Con	a_1 = 18.1228	9.53897 e^{-15}
Temp	a_2 = -11.3072	4.14372 e^{-13}
Tim	a_3 = -0.749996	0. 000493058
Siz	a_4 = 7.41944	1.19943 e^{-11}
Aci	a_5 = -6.05278	6,08538^{-11}
Con*Con	a_{11} = -18.3618	2.46261 e^{-11}
Temp*Temp	a_{22} = 5.47461	3.53143 e^{-7}
Tim*Tim	a_{33} = -6.4804	9.48598 e^{-8}
Siz*Siz	a_{44} = 4.4346	1.78911 e^{-6}
Aci*Aci	a_{55} = -2.55539	0. 000103947
Con*Temp	a_{12} = 3.63437	5,60208 e^{-9}
Con*Tim	a_{13} = -0.0806205	0. 583986 not significant
Con*Siz	a_{14} = -0.676875	0. 00137294 not significant
Con*Aci	a_{15} = -1.39937	9.12921 e^{-6}
Temp*Tim	a_{23} = 0.0781245	0. 595464 not significant
Temp*Siz	a_{24} = -1.25563	2,03556 e^{-5}
Temp*Aci	a_{25} = 1.00937	9. 77612 e^{-5}
Tim*Siz	a_{34} = 0.524378	0.00594903 not significant
Tim*Aci	a_{35} = 1.17938	3.21538 e^{-5}
Siz*Aci	a_{45} = -1.79187	1.40663 e^{-6}

extremely good replicates [35]. The analysis of variance ANOVA of each term of quadratic model is tabulated in Table IV. Analyzes were performed using Fisher's "F" test. According to Rene *et al.* [36], the "F" value with a low probability "P" value generally indicates high significance of the regression model. Based on the ANOVA summary (Table IV), the model was found to be statistically significant ($P < 0.05$) at the 95% confidence level. Due to the lower value for lack of fit and the lower "P" value ($P = 0$), the effects are highly significant.

3.3. Regression coefficients and probability

The linear, quadratic and interaction coefficients of different factors effects a_0 is constant; a_1 (Concentration), a_2 (Temperature), a_3 (Time), a_4 (different size) and a_5 (Acid concentration) a_{11}, a_{22}, a_{33}, a_{44}, a_{55}, a_{12}, a_{13}, a_{14}, a_{15}, a_{23}, a_{24}, a_{25}, a_{34}, a_{35} and a_{45} obtained from the experimental design model are represented in Table V.

In the first column, analysis of the coefficients indicated that only four were not significant a_{13} (Concentration*Time), a_{13} (Concentration*Size), a_{23} (Temperature*Time) and a_{34}

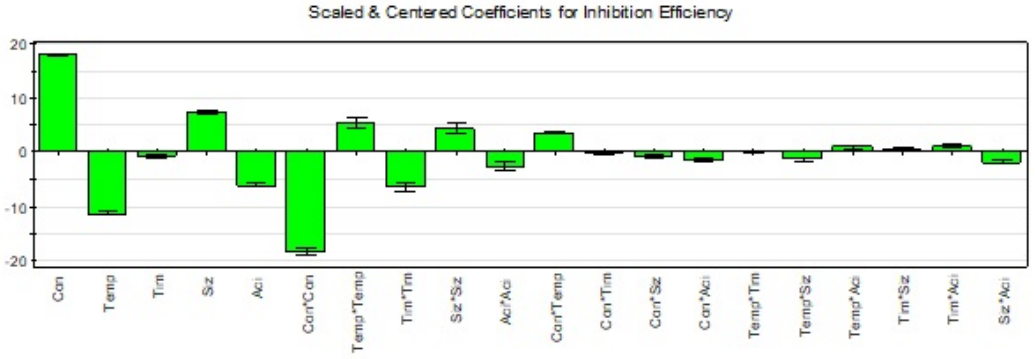

FIGURE 6. Main effect coefficients plot.

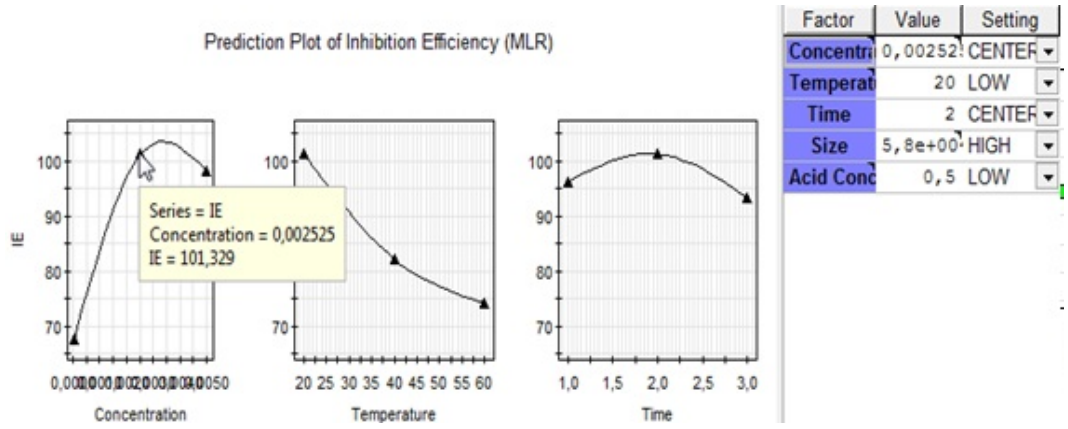

FIGURE 7. Prediction plot of inhibition efficiency (IE) in PVP polymer.

(Time*Size), while the other factors are all significant ($P < 0.0001$). However the positive and negative signs in the model signify a synergetic and antagonistic effect respectively. Furthermore, Inhibition Efficiency increases with concentration and size of PVP and decrease with temperature and acid concentration [37].

The histograms of the coefficient's effects on the inhibition efficiencies (IE) of PVP are shown in Fig. 6. We note that the most influential factor is $a_1 = 18.12$ (Concentration) flowed by $a_2 = -11.31$ (Temperature), $a_4 = 7.42$ (different size) and $a_5 = -6.05$ (Acid concentration). The coefficient $a_3 = -0.75$ (Time) is considered negligible compared to the other variables. We can say that the concentration, temperature and different size of PVP compared to immersion time, are the most influential factors on inhibition efficiencies (IE). Moreover, the positive coefficients for the independent variables indicated a favorable effect on the mechanical properties [38]. The pure quadratic effect a_{11} (Concentration*Concentration) is the most influential and greater than that linear effect a_1. It is important to note that the Eq. (8) is only valid in the range $5.0\,10^{-5} <$ Concentration (mol/L)$< 5.0\,10^{-3}$, $20 <$ Temperature ($^{\circ}C$) < 60, $1 <$ Time (hrs) $< 3h$, $8000 <$ Different size of PVP (g/mol) < 58000 and $0.5 <$ perchloric Acid Concentration (mol/L) < 1.5.

$$
\begin{aligned}
Y = {} & 65.1385 + 18.1228\,X_1 - 11.3072\,X_2 - 0.749996\,X_3 + 7.41944\,X_4 - 6.05278\,X_5 - 18.3618\,X_{11} \\
& + 5.47461\,X_{22} - 6.4804\,X_{33} + 4.4346\,X_{44} - 2.55539\,X_{55} + 3.63437\,X_{12} - 0.0806205\,X_{13} - 0.676875\,X_{14} \\
& - 1.39937\,X_{15} + 0.0781245\,X_{23} - 1.25563\,X_{24} + 1.00937\,X_{25} + 0.524378\,X_{34} + 1.17938\,X_{35} - 1.79187\,X_{45}, \quad (9)
\end{aligned}
$$

Prediction plot Wizard allowed us to vary each variable independently of the others. The prediction plots of inhibition efficiency in PVP polymer by MLR methodology shows that the (IE) maximum of 101.329 % is obtained for concentration of 2.5 (mol/L), temperature around 20°C, immersion time of 2 hours, PVP size of 58000 (g/mol) and perchloric acid concentration of 0.5 (mol/L) (Fig. 7).

4. Analysis of response surfaces and optimization conditions

The optimum condition of each factor to have maximum value of inhibition efficiencies (IE) must be assured by three-dimensional response surfaces plot and their projections. From the results obtained from the inhibition efficiency prediction plots (Fig. 7). Figure 8 represents the response surface illustrating the optimum of inhibition efficiency (IE) at two hours, different size 58000 (g/mol) and acid concentration of 0.5 (mol/L).

To confirm our results of prediction plot wizard, we exploit all the $4D$ contour plots to find the best optimum. We fixed the size of the PVP at 58000 (g/mol) by varying each time the other parameters from the low level to the high level. Figures 6 illustrate the $4D$ contour curves of inhibition efficiency (IE). Figure 9a) illustrates the $4D$ contour for the value of acid concentration at the maximum value (1.5 mol/L), Fig. 9b) that in the center of domain (1 mol/L) and Fig. 9c). corresponds to the minimum value (0.5 mol/L).

The results obtained show that the optimum is reached at a temperature of 20°C, the immersion time of 2 h, the concentration of 0.0025 (mol/L) and an acidity of 0.5 (mol/L) using the size of the PVP of 58000 (g/mol).

To better observe the optimum, we fixed the immersion time at 2 h, the size of the PVP at 58000 (g/mol), the acid concentration at 0.5 (mol/L). Figure 10 illustrates the $4D$ contour plot using the MLR method. The optimum of (IE) obtained is 104.301% for a concentration of 0.00354962 (mol/L) and

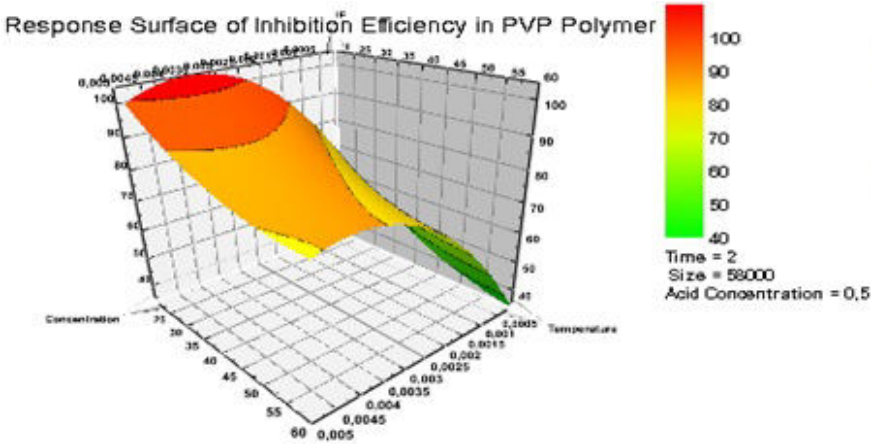

FIGURE 8. Response surface plot of inhibition efficiency (IE) in PVP polymer.

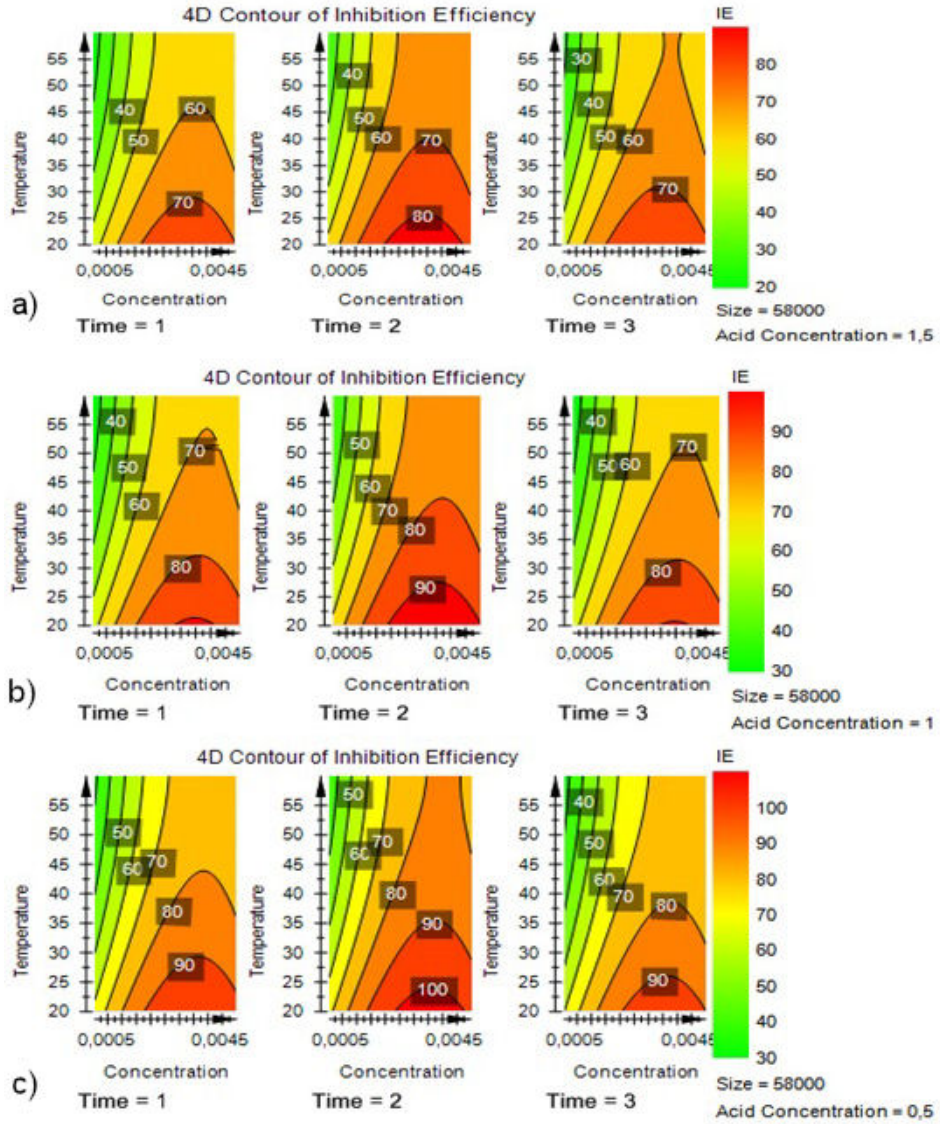

FIGURE 9. a) 4D Contour Plot of Inhibition Efficiencies (IE) for a PVP Size of $58000 g/mol$ vs reaction Concentration, Temperature, Time, and Acid Concentration (High). b) 4D Contour Plot of Inhibition Efficiencies (IE) for a PVP Size of 58000 g/mol vs reaction Concentration, Temperature, Time, and Acid Concentration (Center). c) 4D Contour Plot of Inhibition Efficiencies (IE) for a PVP Size of 58000 g/mol vs reaction Concentration, Temperature, Time, and Acid Concentration (Low).

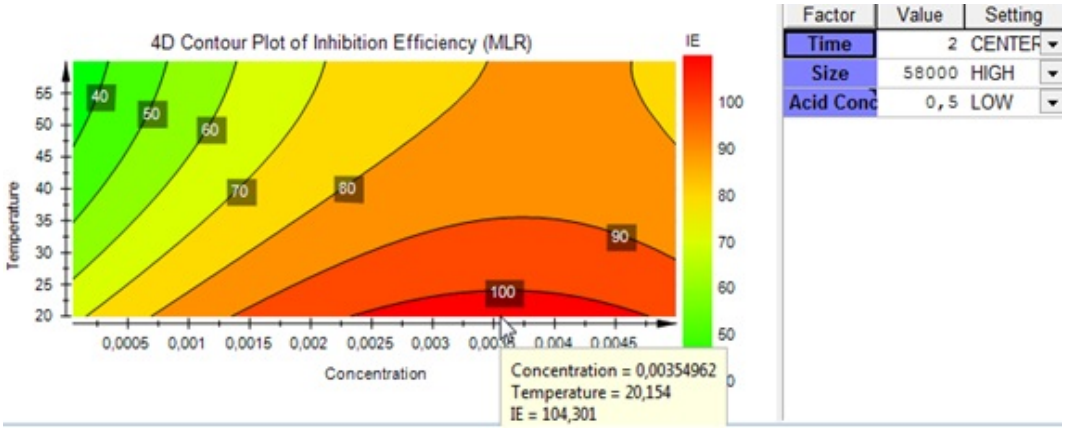

FIGURE 10. 4D Contour Plot of Inhibition Efficiencies (IE) Optimum.

a temperature of $20.154°$C. This study clearly shows that these results are in good agreement with those obtained experimentally.

5. Conclusion

The effect of five independent factors as Concentration, Temperature, Time of immersion, Different size of PVP and Acid perchloric concentration on the inhibition efficiencies (IE) in PVP polymer was investigated in this paper, using the Central Composite Face-Centered Design CCFCD, quadratic model of response surface methodology (RSM) applying Multiple Linear Regression (MLR) fit. A linear, quadratic and interaction model terms were developed. Only, the interaction effects of Con*Tim, Con*Siz, Temp*Tim and Tim*Siz were negligible while the other coefficients are significant. The correlation coefficient R^2 of 0.99 indicated a good agreement between the experimental and predicts value. RSM was effectively applied to determine the optimal condition for the maximum (IE) which is 104.301% corresponded to the optimum factors: Concentration of $3.55\,10^{-3}$ mol/L, Temperature of $20.154°$C, Immersion time of 2 h, PVP size of 58000 g/mol and perchloric acid Concentration of 0.5 mol/L. From this study, we can say that the experiments design methodology using response surface could be generally used in professional industry and chemistry. Particularly, it is approached in polymer domain for inhibition efficiencies (IE) modeling.

Acknowledgments

We would like to thank our collaborators who were involved in the original work reviewed here.

1. F. Haff, A. Sanner, F. Straub, Polymers of N-Vinylpyrrolidone: Synthesis, Characterization and Uses, *Polym. J.* **17** (1985) 143-152. https://doi.org/10.1295/polymj.17.143.
2. M. Teodorescu, M. Bercea, S. Morariu, Biomaterials of PVA and PVP in medical and pharmaceutical applications: Perspectives and challenges, *Biotechnol. Adv.* **37** (2019) 109-131. https://doi.org/10.1016/j.biotechadv.2018.11.008.
3. P. Franco, I. De Marco, The use of Poly(N-vinyl pyrrolidone) in the Delivery of Drugs: *A Review, Polymers*, **12** (2020) 1114. https://doi.org/10.3390/polym12051114.
4. I.V. Stalund, G.N. Riise, F. Leh, T.K. Bjanes, L. Riise, E. Svarstad, S. Leh, Case Report: Polyvinylpyrrolidone deposition disease from repeated injection of opioid substitution drugs: report of a case with a fatal outcome, F1000Res. 2021 Apr 19; 10:300. https://doi.org/10.12688/f1000research.51927.2. Online ahead of print. PMID: 34316359.
5. M. Kurakula, G.S.N. Koteswara Rao, Pharmaceutical assessment of polyvinylpyrrolidone (PVP): As excipient from conventional to controlled delivery systems with a spotlight on COVID-19 inhibition, *J. Drug Deliv. Sci. Techol.*, **60** (2020) 102046. https://1016/j.jddst.2020.102046
6. D. Krishnaiah, A. Bono, R. Sarbatly, R. Nithyanandam, S.M. Anisuzzaman, Optimisation of spray draying operating conditions of Morinda citrifolia L. fruit extract using response surface methodology, *J. King Saud Univ. Eng. Sci.*, **27** (2015) 26-36. https://doi.org/10.1016/j.jksues.2012.10.004.
7. E. Betiku, S.S. Okunsolawo, S.O. Ajala, O.S. Odedele, Performance evaluation of artificial neural network coupled with genetic algorithm and response surface methodology in modeling and optimization of biodiesel production process parameters from shea tree (Vitellaria paradoxa) nut butter. *Renewable Energy*, **76** (2015) 408-417. https://doi.org/10.1016/j.renene.2014.11.049.
8. T. Muppaneni, H.K. Reddy, S. Ponnusamy, P.D. Patil, Y. Sun, P. Dailey, S. Deng, Optimization of biodiesel production from palm oil under supercritical ethanol conditions using hexane as co-solvent: A response surface methodology approach,

Fuel, **107** (2013) 633-640. https://doi.org/10.1016/j.fuel.2012.11.046.

9. A. Rai, B. Mohanty, R. Bhargava, Supercritical extraction of sunflower oil: A central composite design for extraction variables, *Food Chem.*, **192** (2016) 647-659. https://doi.org/10.1016/j.foodchem.2015.07.070.
10. R. Mohammadi, M.A. Mohammadifar, A.M. Mortazavian, M. Rouhi, J.B. Ghasemi, Z. Delshadian, Extraction optimization of pepsin-soluble collagen from eggshell membrane by response surface methodology, *Food Chem*, **190** (2016) 186-193. https://doi.org/10.1016/j.foodchem.2015.05.073.
11. M.J. Ahmed, S.K. Theydan, Optimization of microwave preparation conditions for activated carbon from Albizia lebbeck seed pods for methylene blue dye adsorption, *J. Anal. Appl. Pyrol.*, **105** (2014) 199-208. https://doi.org/10.1016/j.jaap.2013.11.005.
12. E. Mastan, X. Li, S. Zhu, Modeling and theoretical in controlled radical polymerization, *Prog. Polym. Sci.*, **45** (2015) 71-101. https://doi.org/10.1016/j.progpolymsci.2014.12.003.
13. R. Kumar, P Pal, Response surface-optimized Fenton's pretreatment for chemical precipitation of struvite and recycling of water through downstream nanofiltration, *Chem. Eng. J.*, **210** (2012) 33-44. https://doi.org/10.1016/j.cej.2012.08.036.
14. A. Bono, S.M. Anisuzzaman, O.W. Ding, Effect of process conditions on the gel viscosity and gel strength of semi-refined carrageenan (SRC) produced from seaweed (Kappaphycus alvarezii), *J. King Saud Univ. Eng. Sci.*, **26** (2014) 3-9. https://doi.org/10.1016/j.ksues.2012.06.001.
15. Douglas C. Montgomery, Design and Analysis of Experiments, 10th Edition - Amazon: John Wiley and Sons, Inc., 2019.
16. K.A. Alyamac, E. Ghafari, R. Ince, Development of eco-efficient self-compacting concrete with waste marble powder using the response surface method, *J. Clean. Prod.*, **144** (2017) 192-202. https://doi.org/10.1016/j.jclepro.2016.12.156.
17. B. Simsek, T. Uygunoglu, H. Korucu, M.M Kocakerim, Analysis of the Effects of Dioctyl Terephthalate Obtained from Polyethylene Terephthalate Wastes on Concrete Mortar: a Response Surface Methodology based Desirability Function Approach Application, *J. Clean. Prod.*, **170** (2018) 437-445. https://doi.org/10.1016/j.jclepro.2017.09.176.
18. S.M. Bashar, C. K. Veerendrakumar, F.N. Muhd, Rubbercrete mixture optimization using response surface methodology, *J. Clean. Prod.*, **171** (2018) 1605-1621. https://doi.org/10.1016/j.jclepro.2017.10.102.
19. M. Tyagi, A. Rana, S. Kumari, S. Jagadevan, *J. Clean. Prod.*, **178** (2018) 398-407. https://doi.org/10.1016/j.jclepro.2018.01.016.
20. N. Bradley, The response surface methodology (M.A.) Thesis - Indiana University South Bend; 2007.
21. B. Koç, F. Kaymak-Ertekin, Response surface methodology and food processing applications, *GIDA - J. Food*, **35** (2010) 63-70.
22. A. Y. Aydar, N. Bağdatlioğ, O. Köseoğlu. Effect of ultrasound on olive oil extraction and optimization of ultrasound-assisted extracted of extra virgin olive oil by response surface methodology (RSM), *Grasas y Aceites*, **68** (2017) 189. https://doi.org/10.3989/gya.1057162.
23. T. Attar, L. Larabi, Y. Harek, Inhibition effect of potassium iodide on the corrosion of carbon steel (XC 38) in acidic medium, *Inter. J. Adv. Chem.*, **2** (2014) 139-142. https://doi.org/10.14419/ijac.v2i2.3272
24. A. Benchadli, T. Attar, E. Choukchou-Braham, Inhibition of Carbon Steel Corrosion in Perchloric Acid Solution by Povidone Iodine, *Phys. Chem. Res.*, *7* (2019) 837-848. https://doi.org/10.22036/PCR.2019.198787.1665.
25. T. Attar, A. Benchadli, B. Messaoudi, N. Benhadria, E. Choukchou-Braham, Experimental and Theoretical Studies of Eosin Y Dye as Corrosion Inhibitors for Carbon Steel in Perchloric Acid Solution, *Bull. Chem. React. Eng. Catal.*, **15** (2020) 454-464. https://doi.org/10.9767/bcrec.15.2.7753.454-464.
26. D. A. Belsley, E. Kuh, and R. E. Welsch. Regression Diagnostics: Identifying Influential Data and Sources of Collinearity. New York: Wiley, (1980).
27. A. Ghasemzadeh, H.ZE. Jaafar, E. Karimi, A. Rahmat, Optimization of ultrasound-assisted extraction of flavonoid compounds and their pharmaceutical activity from curry leaf (Murraya koenigii L.) using response surface methodology, *BMC Complementary & Alternative Medicine*, **14** (2014) 318. https://doi.org/10.1186/1472-6882-14-318.
28. Modde 9.1 User's guide; Umetrics: Umea, Sweden, (2011).
29. N. Kettaneh-Wold, Analysis of mixture data with partial least squares, *Chemometrics and Intelligent laboratory Systems*, **14**, (1992) 57-69. https://doi.org/10.1016/0169-7439(92)80092-I.
30. H. Wold. Soft Modelling by Latent Variables: The Non-Linear Iterative Partial Least Squares (NIPALS) Approach. Perspectives in Probability and Statistics, Gani, J. (ed.), (Papers in Honour of M. S. Bartlett). Academic Press, London, (1975) 117-142.
31. S. Weisberg, Applied Linear Regression, 2nd Ed., J. Wiley & Sons, Inc., New York 1985.
32. B. Chauhan, R. Gupta, Application of statistical experimental design for optimization of alkaline protease production from Bacillus sp. RGR-14, *Proc. Biochem.*, **39** (2004) 2115-2122. https://doi.org/10.1016/j.procbio.2003.11.002.
33. A. Koocheki, A.R. Taherian, S. M.A. Razavi, A.Bostan, Response surface methodology for optimization of extraction yield, viscosity, hue and emulsion stability of mucilage extracted from Lepidium perfoliatum seeds, *Food Hydrocolloids*, **23** (2009) 2369-2379. https://doi.org/10.1016/j.foodhyd.2009.06.014.
34. A. Rai, B. Mohanty, R. Bhargava, Supercritical extraction of sunflower oil: A central composite design for extraction variables, *Food Chem.*, **192** (2016) 647-659. https://doi.org/10.1016/j.foodchem.2015.07.070.

35. T. Attar, A. Benchadli, T. Mellal, B. Dali Youcef, E. Choukchou-Braham, Use of Experimental Designs to Evaluate the Influence of Methyl Green Dye as a Corrosion Inhibitor for Carbon Steel in Perchloric Acid, *Malays. J. Chem.*, **23** (2021) 60-69. http://103.6.196.91/$\sim$ikmorg/ojs/index.php/MJChem/article/view/878.

36. E. R. Rene, M. S. Jo, H. S. Park, S. H. Kim, *Int. J. Environ. Sci. Tech.*, **4** (2007) 177-182. https://doi.org/10.1007/BF03326271.

37. A. Benchadli, T. Attar, B. Messaoudi, E. Choukchou-Braham, Polyvinylpyrrolidone as a Corrosion Inhibitor for Carbon Steel in a Perchloric Acid Solution: Effect of Structural Size, *Hung. J. Ind. Chem.*, **49** (2021) 59-69. https://doi.org/10.33927/hjic-2021-08.

38. A. E. Hadi, Characterization and optimization of mechanical, physical and thermal properties of short Abaca (Musa Textile Nee) fiber reinforced high impact polystyrene composites [PhD thesis], Universiti Putra Malaysia, (2011).