

Journal of Chemical, Biological and Physical Sciences



An International Peer Review E-3 Journal of Sciences

Available online at www.jcbps.org

Section A: Chemical Sciences

CODEN (USA): JCBPAT

Research Article

Application of Full Factorial Design for single super phosphate production from Abu Tartur Phosphate Rocks

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Received: 30 April 2018; **Revised:** 16 May 2018; **Accepted:** 22 May 2018

Abstract: The multivariate 2⁴ full factorial methodologies is used to study the effect of different parameters on the single super phosphate production using Abu Tartur Phosphate ore and sulfuric acid solutions. The full factorial design has been performed to improve the P₂O₅ Conversion %, for single super phosphate production. Four factors were taken into consideration in the experimental planning: acid concentration, time reaction, particle size and solid/ liquid ratio. The analysis of variance (ANOVA) has been used to determine the main effects and interactions between the studied factors. According to The optimum conditions have been determined as, acid concentration of 70 %, time reaction of 5 min, particle size of 80% finance and solid/ liquid ratio, g /ml of 1:2. Under these optimum conditions, the P₂O₅ Conversion is 80.7 %.

Keywords: Statistical modeling, full factorial design, Phosphate ore, single super phosphate.

INTRODUCTION

Phosphorus (P) is one of essential macro nutrients for a higher plants and animals. Phosphates material is insoluble in alkali soils like those of Egypt. Therefore in original state it is practically unavailable as plant

nutrient source^{1, 2}. The appropriate and sound utilization of phosphate rocks (PRs) as source of P can contribute to sustainable agricultural intensification. Particularly in developing countries endowed with Phosphate Rock (PR) resources. Phosphate Rock (PR) is a general term used to describe phosphate - bearing minerals. Phosphate Rock (PR) is a finite, nonrenewable natural resource. Geological deposits of different origin are found throughout the world³.

Currently, Phosphates, in the form of fertilizers, are essential in the agricultural sector. They are also very important constituents in animal feed stocks and in food and other chemical industries. About 95 % of the world phosphate rock production is consumed in fertilizer industry. Most of the balance is processed, in electric furnaces, into elemental phosphorus which is the main raw material for manufacturing various phosphate compounds^{4, 5}. Different types of phosphate rocks have widely differing mineralogical, chemical and textural characteristics. While there are more than 200 known phosphate minerals, the main mineral group of phosphates is the apatite group. Calcium- phosphates of the apatite group are mainly found in primary environments (in sedimentary, metamorphic and igneous rocks) but also in weathering environments. Three quarters of the world's phosphate deposits are of sedimentary origin and about 75%-80% of those include carbonate gangue⁶. These deposits consist mainly of calcite, fluorapatite, quartz, and kaolinite. During the last decade, there have been numerous published articles which indicate that phosphate deposits reserves are expected to be depleted in 50-100 years and peak phosphate production is expected to be reached^{7, 8} in 2033-2034, while others are more optimistic⁹.

Phosphate ore is an important economic deposit in Egypt. Phosphate deposits in Egypt are a part of the Middle East to North African phosphogenic province of Late Cretaceous-Palaeogene age. Phosphate ores occur in Egypt in three main provinces, Western desert, Nile valley, and Red sea. Added to these are some phosphate- bearing sediments are present in Bahariya Oasis, and Sinai but with limited extent and valueless as shown^{10, 11} in **Figure 1**.

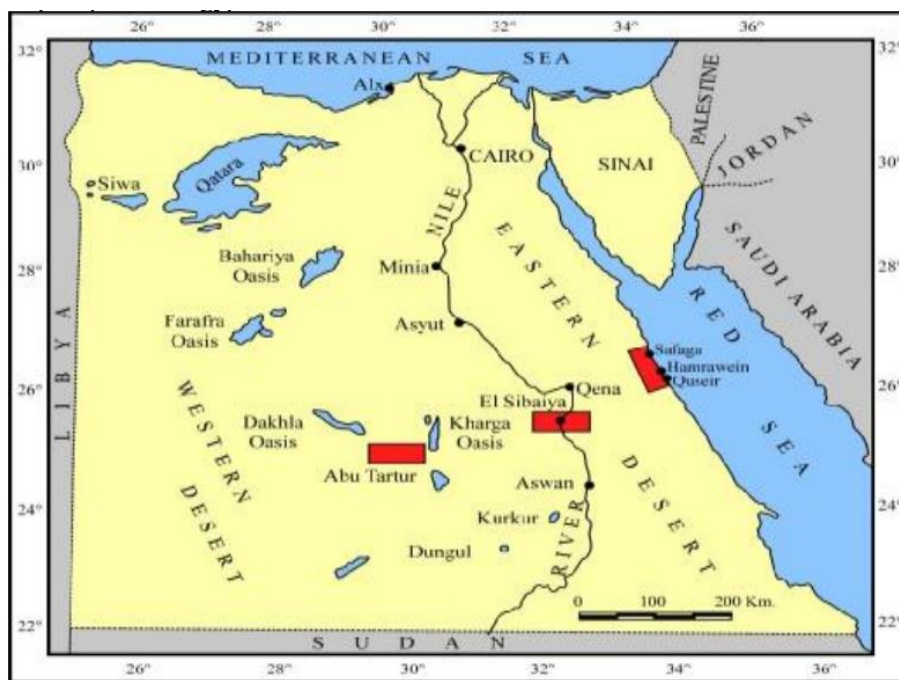
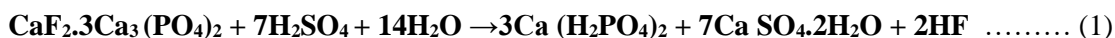


Figure 1: location map of the phosphorite deposits in Egypt^{10, 11}.

Single superphosphate (SSP) is the earliest type of phosphatic fertilizer produced, also known as normal or simple superphosphate. The manufacturing process of this fertilizer involves the reaction of diluted sulphuric acid with beneficiated phosphate^{12,13} rock as shown in *equation 1*



The fertilizer contains about (16 – 20 %) P₂O₅. Calcium phosphate produced by this reaction remains in the fertilizer. The global production of this type of fertilizer is declining due to environmental and economic considerations.

The present work aims to Application of Full Factorial Design for single super phosphate production from Abu Tartur Phosphate Rocks. Statistical design and analysis of experiments were used in order to determine the main effects and interactions of sulfuric acid concentration, solid-to-liquid ratio, particle size and reaction time in terms of the maximum P₂O₅ conversion %. Therefore, the full factorial design involving 16 treatment combinations and 4 replications of the central point has been applied to investigate the effect of different factors on the P₂O₅ conversion %.

EXPERIMENTAL

Raw Materials: The working sample of phosphate ore was collected from the under study mine of Abu Tartur plateau located in the Western Desert of Egypt. The ore sample was pretreated crushed then washed with water and dried at 105⁰C then cooled and treated to upgrading of Low grade phosphate ore by Using Gravity Separation Technique¹⁴. The data of chemical analysis of the ore sample are listed in **Table 1**.

Table 1: Chemical analysis of upgraded Abu-Tartur sample phosphate ore

Constituent, %			
P ₂ O ₅	30.84	SO ₃	1.50
CaO	46.67	MgO	0.90
Fe ₂ O ₃	3.8	Al ₂ O ₃	0.46
F	2.8	Na ₂ O	0.28
SiO ₂	2.3	L.O.I.*	5.1
Constituent, mg			
U	30	∑ REEs	900

L.O.I. = Loss of ignition

XRD of the sample indicated that: the main mineral of the phosphate rock is Francolite together with minor amounts of Dolomite and Calcite as shown in **Figure (2)**.

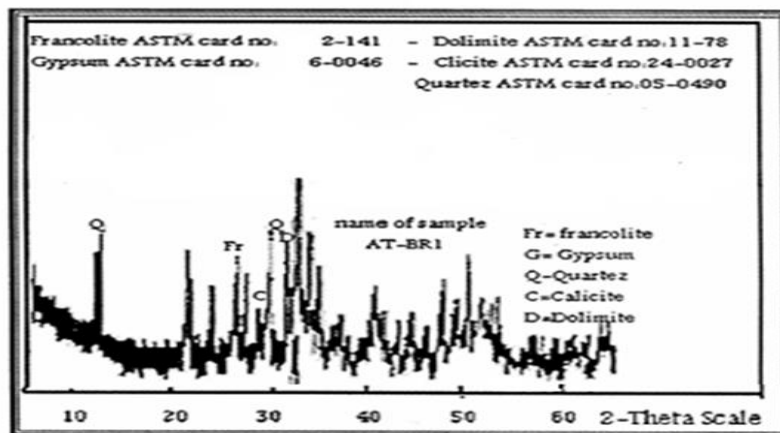


Figure (2): XRD pattern of Abu-Tartur phosphate working sample.

Apparatus: The reaction was carried out in a cylindrical 1 L reactor of 10 cm diameter. It was fitted with Teflon-coated stirrer with 4 cm diameter and placed in thermostatically controlled water bath. The impeller tip speed was adjusted at 200 rpm.

Procedure: The single super phosphate production is carried out by mixing of grinded phosphate rock with diluted sulfuric acid in a cylindrical reactor with stirring; the process can be divided into two stages as follows:

- The first stage represents the diffusion of sulfuric acid to the rock particles accompanied by a rapid chemical reaction on the particle surface, which continues until the acid is completely consumed, and crystallization of calcium sulphate.
- The second stage represents the diffusion of the formed phosphoric acid into the pores of the rock particles which did not decompose. This stage is accompanied by a second reaction.

After the desired reaction time, The product was weighed and P_2O_5 content was determined by Atomic Absorption Spectrometer type GBC 932 AA (UK) while P_2O_5 content was determined by a colorimetric method (spectrophotometer type Shimadzu UV 1208, ammonium molybdate and ammonium metavanadate were used for P_2O_5 analysis)¹⁵. P_2O_5 conversion, % was calculated by the following equations:

$$P_2O_5 \text{ conversion, \%} = \frac{P_2O_5 \text{ concentration, Water soluble, \%}}{\text{total } P_2O_5 \text{ concentration, \%}} \times 100$$

Design of experiments: The statistical design of experiments is a systematic approach to determine the mathematical relationship between factors and responses. DoE is used to plan experiments so that the maximum amount of information can be extracted from the performed experiments. The factors in a DoE investigation are independent of each other in a statistical sense which makes it possible to evaluate the effect on the response of each factor separately (main effects). In addition, interaction effects between factors can be evaluated.

A commonly used type of DoE is full factorial design, which is used both for screening and optimization purposes. A great advantage with the full factorial design is that all main effects and interaction effects are independent of each other and therefore their effect on the response can be resolved in the evaluation. The simplest factorial is the 2^n factorial design in which each variable is investigated at two levels, where each variable (X_i , $i = 1 - n$) is investigated at minimum two levels^{16, 17}. As the number of factors (n) increases, the number of runs for a complete replicate of the design also increases rapidly. Center points are important for the DoE. The center point is usually replicated and will give information on the variation in the responses. The center points will also provide information on possible curvature in the data¹⁸. Curvature is detected when the average mean response at the center points is significantly greater or less than the average mean response of the factors at their low and high settings. Modeling can be performed using the first order model, defined by the equation:

Modeling can be performed using the first order model, defined by the equation:

$$Y = b_0 + \sum_{i=1}^n b_i x_i + \sum_{i=1}^n \sum_{j>1}^n b_{ij} x_i x_j$$

or the second order model, which is:

$$Y = b_0' + \sum_{i=1}^n b_i x_i + \sum_{i=1}^n b_{ii} x_i^2 + \sum_{i=1}^n \sum_{j>1}^n b_{ij} x_i x_j$$

The statistical design of experiments is procedure for planning experiments so that the data obtained can be analyzed to yield valid and objective conclusions. This procedure was used to estimate the interaction between factors and decrease the number of experiments, experiments time, and process cost. To obtain a reliable statistical model, prior knowledge of the procedure is generally required¹⁹. The two main applications of experimental design are screening, in which the factors that influence the experiment are identified, and optimization, in which the optimal settings or conditions for an experiment can be found^{20, 21}.

RESULTS AND DISCUSSIONS

Optimization of process conditions is usually one of the most important factors to reduce the production cost. The conventional method, which involved varying one variable at a time while keeping the other variables constant is lengthy and often does not produce the effect of interaction of different variables. Laboratory scale confirmation of the products was then used to validate the feasibility of the derived optimum conditions. In the light of pre-experiments, four factors, namely, acid concentration, reaction time, particle size, % and solid / liquid ratio g / ml were chosen as independent variables in order to optimize the favor single super phosphate production from Abu Tartur Phosphate Rocks.

The experimental variables are evaluated at two levels, low (denoted as -1), high (denoted as $+1$), and midlevel denoted as 0), as shown in Table 2. The experiment order was randomly to avoid systematic errors. The results are analyzed with the Design Expert 11.0.3.0 software, and the main effects and interactions between factors were determined.

Table 2: The levels of experimental factors for the full factorial design

Factors	Coded variables	Low level (-)	High level (+)	Mid-level (0)
Sulfuric acid concentration, %	A	50	70	60
Time, min.	B	5	15	10
Particle size %	C	80	96	88
Solid /liquid ratio, g /ml	D	1.5	2	1.75

The significant parameters: For 2^n factorial designs, it is assumed that the response is close to linear over the range of the factor levels. However, linearity assumption is often violated in practice. In this case, it is necessary to include one or more runs where all factors are set at their midpoint. The addition of center points to design allows the researcher to check whether the linearity of the effects is a reasonable assumption or whether quadratic terms should be added to the model. The designed operating conditions are summarized in **Table 3** showing that all factors are varied simultaneously. According to experimental plan, a total number of 16 runs and 4 replications of the center points have been designed.

The results obtained from **Table (3)** show that 80.68 P_2O_5 conversion, % was successfully produced of single super phosphate from Abu Tartur Phosphate ore regards to the following conditions (experiment number 11); 70 % sulfuric acid, reaction time 5 min , 80% particle size and solid/ liquid ratio, gm/ ml, of 1/2. However, .Moreover, experiments number 18 also represents the second highest P_2O_5 conversion, % efficiency (79.65). On the other hand, experiments number 10 and 12 represent the lowest P_2O_5 conversion, % efficiency (55.61and 52.72% respectively).

The effects of the experimental factors and their interactions influence the, P_2O_5 Conversion % is summarized in **Tables 4 and 5**. The positive values of these effects reveal that the increase of these parameters increased leaching efficiency. Conversely, negative values of the effects decreased the response (reaction conversion %).

From the obtained results it is clear that, the solid/ liquid ratio had the greatest effect on reaction P_2O_5 conversion %, followed by sulfuric acid concentration. The obtained results confirm the main advantage of the 2^n factorial design compared to the One-factor-at-a-time, where it shows the effects main variable and also the effect of variables interactions

The obtained results showed that, the solid/ liquid ratio, g/ ml had the greatest effect on P_2O_5 Conversion, % (6.7525), followed by sulfuric acid concentration (2.545). This means that, the increase in these factors level leads to increase on P_2O_5 Conversion %. While time interaction have negative effect on P_2O_5 Conversion, % (-9.91), and Particle size, %(-5.0025) which means that these factors level should be at the lowest value

On the other hand, acid concentration- particle size, acid concentration- reaction time -particle size, acid concentration- time - solid/ liquid ratio ,and acid concentration- - particle size - solid/ liquid ratio ,and acid concentration- time - particle size - solid/ liquid ratio have negative effect on P_2O_5 Conversion, % (-5.635, -1.6175, -1.4525, -1.745, -2.4075).

Table 3: Design matrix of the 2⁴ full factorial design

run#	conc H ₂ SO ₄ , %	time, min	Particle size, %	ratio, g/ ml	Water soluble, %	total P ₂ O ₅ , %	H ₂ O, %	P ₂ O ₅ Conversion, %
1	70	15	96	1/2.00	15.01	21.54	13.6	69.68
2	70	15	96	1/1.50	10.18	18.07	14.86	56.33
3	70	15	80	1/2.00	13.08	17.57	14.7	74.44
4	70	5	96	1/2.00	13.03	18.48	15	70.50
5	50	15	96	1/2.00	13.93	18.91	13.55	73.66
6	50	5	80	1/1.50	15.04	19.3	15.25	77.92
7	70	15	80	1/1.50	10.02	14.96	15.2	66.93
8	50	5	96	1/2.00	14.41	19.85	13.96	72.59
9	70	5	96	1/1.50	14.59	24.15	15.26	60.41
10	50	15	80	1/2.00	7.87	14.15	14.9	55.61
11	70	5	80	1/2.00	13.66	16.93	13.8	80.68
12	50	15	96	1/1.50	6.88	13.05	13.85	52.72
13	50	5	80	1/2.00	12.68	16.67	15.1	76.06
14	50	5	96	1/1.50	11.52	16.38	15.34	70.32
15	50	15	80	1/1.50	8.02	14.03	16.75	57.16
16	70	5	80	1/1.50	13.06	16.88	19.89	77.36
17	60	10	88	1/1.75	13.58	17.3	13.44	78.49
18	60	10	88	1/1.75	13.9	17.45	12.94	79.65
19	60	10	88	1/1.75	13.33	17.53	12.82	76.04
20	60	10	88	1/1.75	13.49	17.14	13.48	78.70

Table 4: Estimated Effects for P₂O₅ conversion %

	Term	Stdized effect	Sum of Squares	Contribution, %
	Intercept			
m	A - H ₂ SO ₄ conc, %	2.545	25.9081	1.6793
m	B - time, min	-9.91	392.832	25.4625
m	C - Particle size,%	-5.0025	100.1	6.48824
m	D - ratio, gm/ml	6.7525	182.385	11.8218
m	AB	4.5275	81.993	5.31459
m	AC	-5.635	127.013	8.23267
m	AD	1.805	13.0321	0.844709
m	BC	4.55	82.81	5.36755
m	BD	3.3	43.56	2.82345
m	CD	4.9075	96.3342	6.24415
m	ABC	-1.6175	10.4652	0.678331
m	ABD	-1.4525	8.43902	0.546997
m	ACD	-1.745	12.1801	0.789485
m	BCD	2.18	19.0096	1.23216
m	ABCD	-2.4075	23.1842	1.50275
	Curvature	8.89396	316.41	20.5089
e	Lack of fit		0	0
e	Pure Error		7.1347	0.462454
	Lenth's ME	9.08958		0
	Lenth's SME	16.181		0

The four observed recoveries at the center were 78.5 %, 79.7 %, 76 % and 78.7 % (**Table 3**). The average of these four center points is 78.22 %. The average of the 16 runs for base design (**Table 3**) is 68.28 %. Since these two averages are not similar, it is assumed that there is a curvature present. The test for nonlinearity, however, does not tell which factor (s) contains the curvature, only that it exists²². Although center points can detect curvature, they don't provide enough information to model the curvature. To model the curvature, square terms are needed, which requires adding more points to the design. In fact, since the model equation contains some interaction terms, it means that the model is, therefore, capable of representing some curvature in the response function¹⁸. In this regard, the regression model equation with interaction terms can be written as²³:

$$Y = b_0 + b_1A + b_2B + b_3C + b_{12}AB + b_{13}AC + b_{23}BC + b_{123}ABC \dots \dots (\text{Eq1})$$

Where Y = P₂O₅ Conversion, %; b = model coefficients; and A, B, C, and D = dimensionless coded factors for sulfuric acid concentration, time reaction, particle size, solid/ liquid ratio, respectively. The coefficients of Equation (1) are presented in **Table (5)**.

Table 5: Estimated Coefficients for P₂O₅ Conversion, %

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	68.28	1	0.3855	67.05	69.51	
A-H ₂ SO ₄ Conc	1.27	1	0.3855	0.0455	2.50	1.0000
B-Time	-4.96	1	0.3855	-6.18	-3.73	1.0000
C-Particle size	-2.50	1	0.3855	-3.73	-1.27	1.0000
D-Ratio	3.38	1	0.3855	2.15	4.60	1.0000
AB	2.26	1	0.3855	1.04	3.49	1.0000
AC	-2.82	1	0.3855	-4.04	-1.59	1.0000
AD	0.9025	1	0.3855	-0.3245	2.13	1.0000
BC	2.27	1	0.3855	1.05	3.50	1.0000
BD	1.65	1	0.3855	0.4230	2.88	1.0000
CD	2.45	1	0.3855	1.23	3.68	1.0000
ABC	-0.8087	1	0.3855	-2.04	0.4182	1.0000
ABD	-0.7262	1	0.3855	-1.95	0.5007	1.0000
ACD	-0.8725	1	0.3855	-2.10	0.3545	1.0000
BCD	1.09	1	0.3855	-0.1370	2.32	1.0000
ABCD	-1.20	1	0.3855	-2.43	0.0232	1.0000
Ctrl Pt 1	9.94	1	0.8621			

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-co linearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

Final Equation in Terms of Coded Factors

$$\text{P}_2\text{O}_5 \text{ Conversion, \%} = 68.2812 + 1.2725 * A - 4.955 * B - 2.50125 * C + 3.37625 * D + 2.26375 * AB - 2.8175 * AC + 0.9025 * AD + 2.275 * BC + 1.65 * BD + 2.45375 * CD - 0.80875 * ABC - 0.72625 * ABD - 0.8725 * ACD + 1.09 * BCD - 1.20375 * ABCD \quad \dots \text{Equation (2)}$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. Equation (2) in terms of actual factors:

Final Equation in Terms of Actual Factors

$$\begin{aligned} \text{P}_2\text{O}_5 \text{ Conversion, \%} = & -493.63 + 11.1655 * \text{H}_2\text{SO}_4 \text{ conc} + 100.214 * \text{Time} + 7.83781 * \text{Particle size} + \\ & 336.98 * \text{Ratio} - 1.5289 * \text{H}_2\text{SO}_4\text{Conc} * \text{Time} - 0.149312 * \text{H}_2\text{SO}_4\text{Conc} * \text{Particle size} - 5.812 * \\ & \text{H}_2\text{SO}_4\text{Conc} * \text{Ratio} - 1.2765 * \text{Time} * \text{Particle size} - 68.344 * \text{Time} * \text{Ratio} - 4.46812 * \text{Particle size} * \\ & \text{Ratio} + 0.0190437 * \text{H}_2\text{SO}_4\text{Conc} * \text{Time} * \text{Particle size} + 1.0012 * \text{H}_2\text{SO}_4\text{conc} * \text{Time} * \text{Ratio} + 0.07675 \\ & * \text{H}_2\text{SO}_4\text{Conc} * \text{Particle size} * \text{Ratio} + 0.83125 * \text{Time} * \text{Particle size} * \text{Ratio} - 0.0120375 * \text{H}_2\text{SO}_4\text{Conc} \\ & * \text{Time} * \text{Particle size} * \text{Ratio} \end{aligned} \quad \dots \text{Equation (3)}$$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

The Statistical Analysis for the Proposed Model: The statistical analysis of the model was performed in the form of analysis of variance (ANOVA) as shown in **Table 6**. This analysis included the Fisher's F-test (overall model significance), its associated probability P(F), correlation goodness of fit of regression model. The analysis also includes the Student's t-value for the estimated coefficients and associated probabilities P(t). For each variable, the quadratic models were represented as contour plots.

From the table it is clear that, the model f-value of 34.18 implies the model was significant. There is only a 0.01% chance that a 'model F-value' of this magnitude would occur due to noise. The prob> F values for the models which were less than 0.05 (<0.0001) indicate model terms are significant with a confidence interval of 95%. In addition, A, D, AB, AC, BC, BD and CD are significant model terms while B, C, AD, ABD, ABC and ACD are not significant. The value of the adjusted determination coefficient (R^2 -adj) is 96.52 %, which means that only about 3.47 % only of the total variation was not explained, this proves the high significant of the model. The 'adequate precision' measures the signal to noise ratio. A ratio greater than 4 is desirable

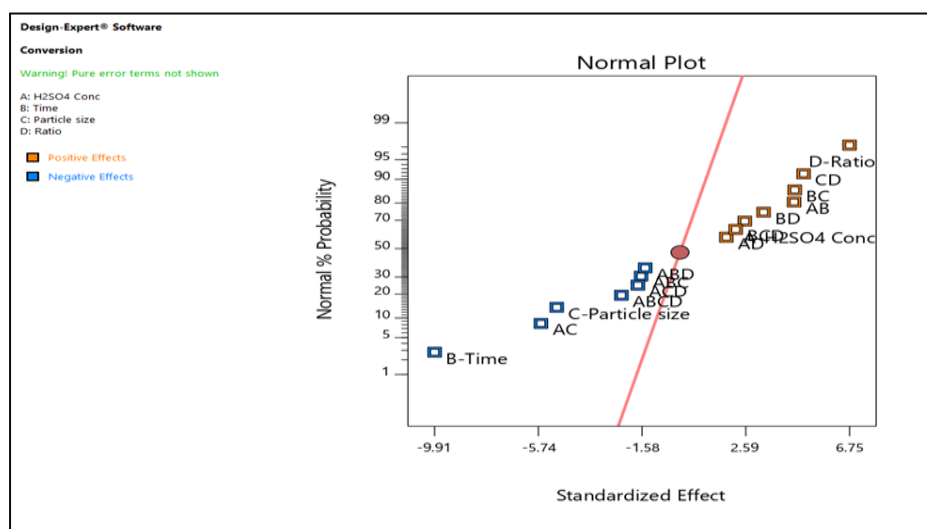
The process of screening involves the ordering of estimates of the effects according to their magnitudes and calculating their cumulative probabilities. These effects are then plotted on a normal probability. The normal probability plot often provides an effective way of helping with the selection¹⁸. In this regards, the estimates of main effects of factors, at the same time with their interaction terms, are shown on a normal probability plot of effects for P_2O_5 conversion % (**Figure 3**). All effects which are insignificant are normally distributed with mean zero and variance σ^2 , and tend to fall along a straight line on the plot. In contrast significant effects have non-zero means and are located far away from the straight line. The greater the significant effect, the most away it is from the straight line. The results obtained in Figure 2 clear that, the main effects A, D, and the interactions AB, AD, BC, BD, CD, and BCD are statistically significant for P_2O_5 conversion % because they stray farther from the line while, the main effect B, C and other interactions AC, ABC, ABD, ACD and ABCD are insignificants because they fall along the line.

The same indication is given clearer in the Pareto chart of the selected effect in Figure 4. Pareto chart is a special type of bar chart where the values being plotted are arranged in descending order, therefore it helps to set the priority levels while designing the process²⁴. The chart gives the factors which have a standardized effect beyond 99% confidence level beyond the line at 2.77. **Figure 4** shows that sulfuric acid concentration and solid/ liquid ratio are the most significant factors for P_2O_5 conversion %. Also, it shows the descending order of the effects of different factors and their interactions.

Table 6: Analysis of variance for P₂O₅ Conversion,

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1219.25	15	81.28	34.18	0.0070	significant
A-H ₂ SO ₄ Conc	25.91	1	25.91	10.89	0.0457	significant
B-Time	18.76	1	18.76	7.88	0.0654	Not significant
C-Particle size	18.43	1	18.43	7.75	0.0643	Not significant
D-Ratio	182.39	1	182.39	76.69	0.0031	significant
AB	81.99	1	81.99	34.48	0.0099	significant
AC	127.01	1	127.01	53.41	0.0053	significant
AD	13.03	1	13.03	5.48	0.1011	Not significant
BC	82.81	1	82.81	34.82	0.0097	significant
BD	43.56	1	43.56	18.32	0.0234	significant
CD	96.33	1	96.33	40.51	0.0079	significant
ABC	10.47	1	10.47	4.40	0.1268	Not significant
ABD	8.44	1	8.44	3.55	0.1561	Not significant
ACD	12.18	1	12.18	5.12	0.1086	Not significant
BCD	19.01	1	19.01	7.99	0.0663	significant
ABCD	23.18	1	23.18	9.75	0.0524	significant
Curvature	316.41	1	316.41	133.04	0.0014	
Pure Error	7.13	3	2.38			
Cor Total	1542.79	19				

*Std. Dev. =1.54, R²= 0.9942, Mean=70.27, Adjusted R²=0.9651, C.V. %=2.19, Adeq Precision=19.6723

**Figure 3:** Normal probability plot of the standardized effects for P₂O₅ conversion %

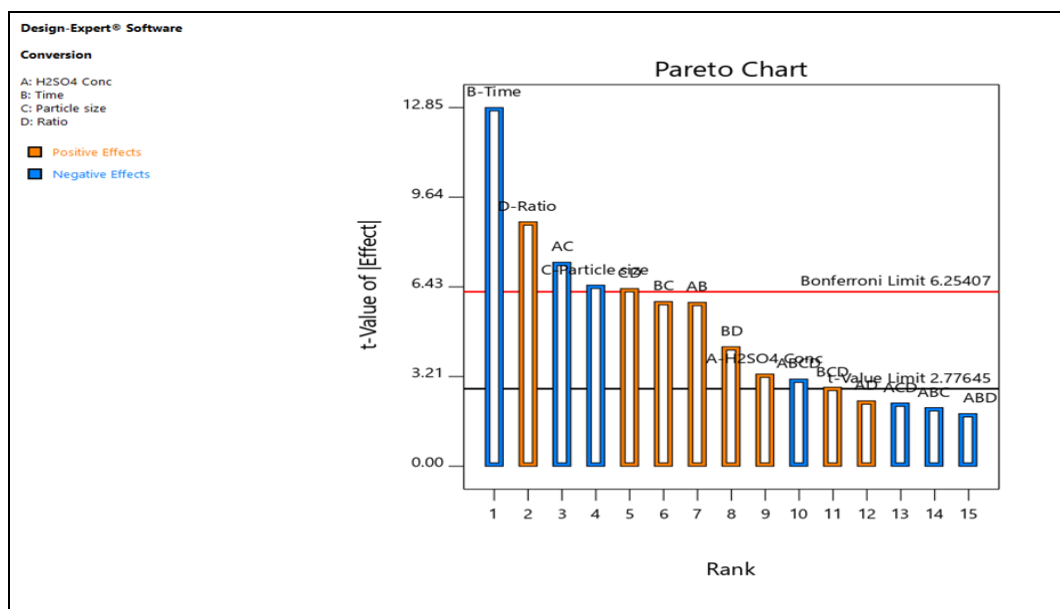


Figure 4: Pareto Chart of the Standardized Effects

The normal plot of residuals is a graphical representation for determining if the data is distributed normally or not¹⁴. The normal probability plots of the residuals for the data tests the hypothesis that the residuals have a normal distribution, where if the plot gives straight line, this means that the residuals have a normal distribution. From **Figure 5**, it is clear that the points were almost distributed in a line, which indicated that the model fitted moderately well.

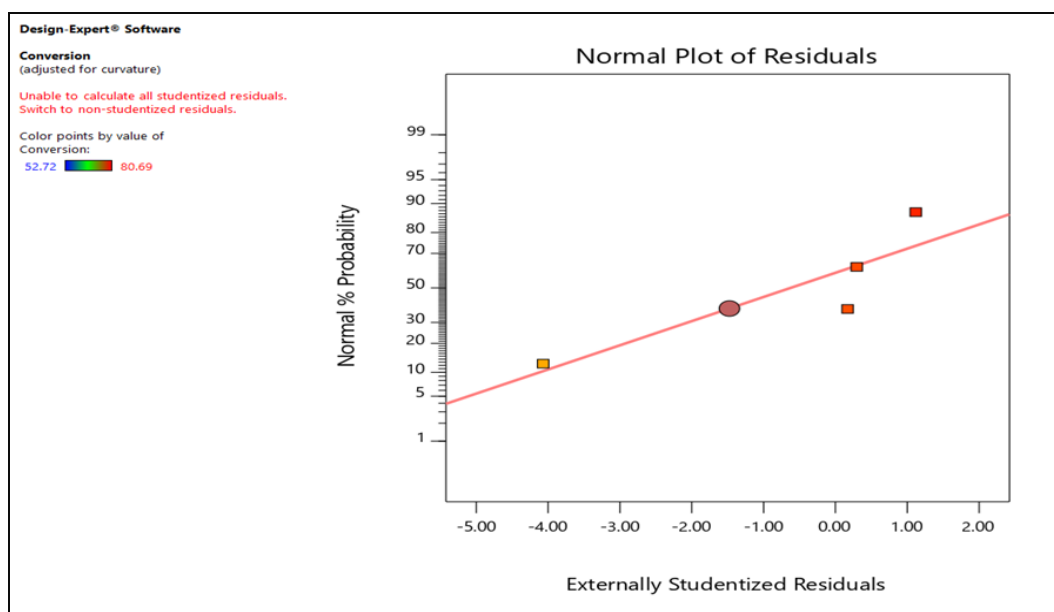


Figure 5: Normal Plot of Residuals.

The plot of residuals versus the predicted response values was presented in **Figure (6)**. This plot tests the hypothesis of constant variance i.e., the random errors are distributed with mean zero and constant variance. From the Figure, it is observed that there was no definite increase in residuals with predicted levels, which show that Equation (2) was in excellent agreement with the experimental data and supporting the underlying assumptions about the errors.

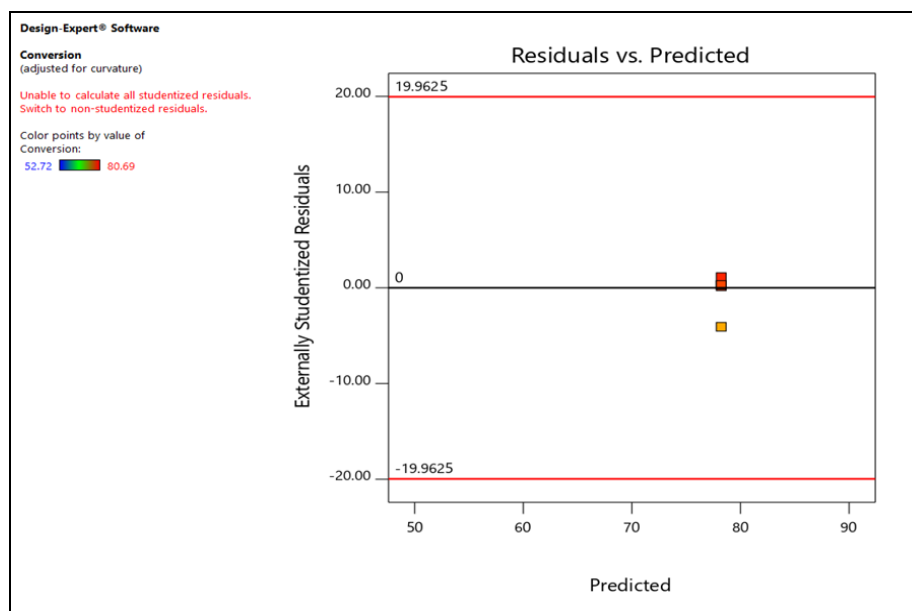
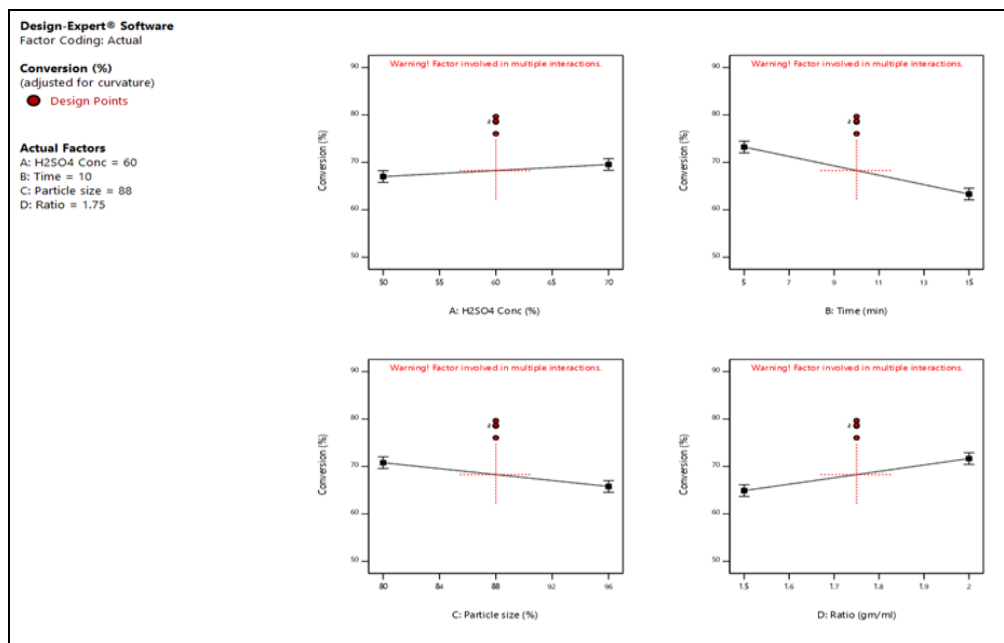
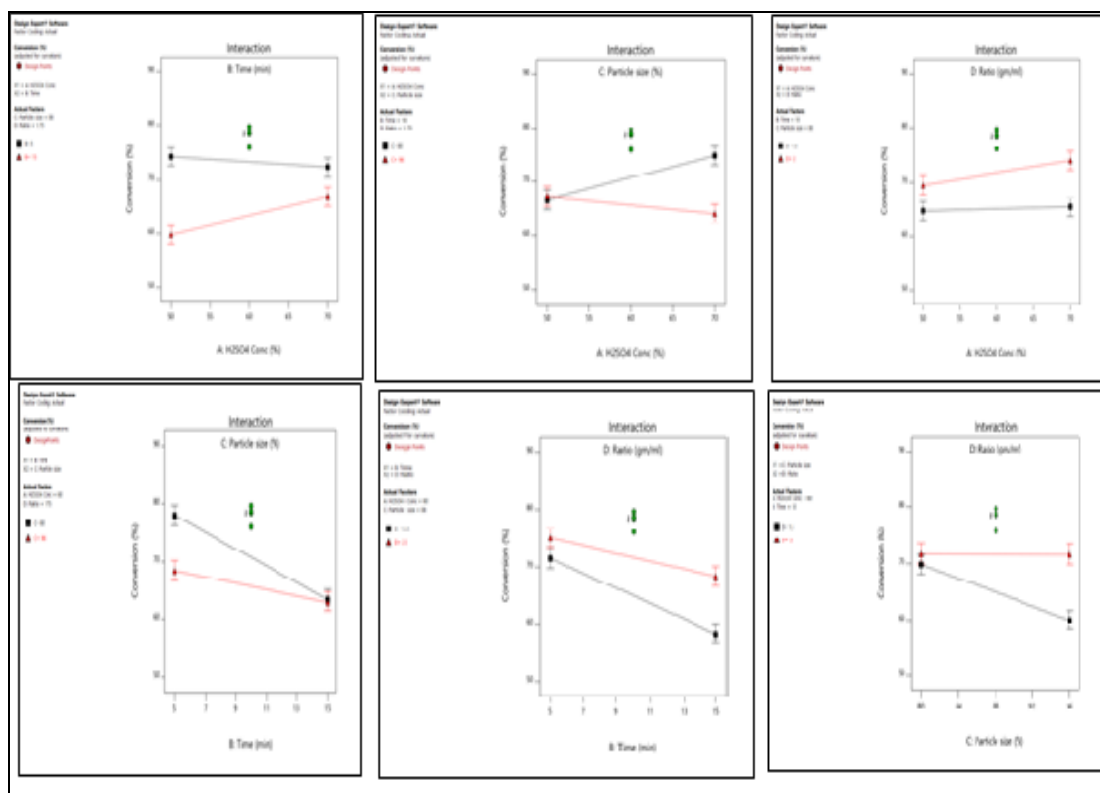


Figure 6: Plot of Residuals versus Predicted Recoveries

The effects of the all individual factors used for P_2O_5 conversion % investigation are presented in **Figure 7**. Solid/ liquid ratio, gm/ ml, (D) effect plot shows that the increase in Solid/ liquid ratio is enhancing effectively the P_2O_5 conversion % .sulfuric acid concentration (A) effect plot clarify that by increase sulfuric acid concentration, the P_2O_5 conversion % increased.

The same effect was observed for the reaction time (B), where the plot of time effect indicates that increase in reaction time has slightly effect on the P_2O_5 conversion %. The particle size (C) effect plot shows that, the increase in the particle size is improving has slightly effect on the P_2O_5 conversion %. One of main advantages of factorial design procedure is the ability to show the interaction between the different factors under the investigation.

The interaction effect for P_2O_5 conversion % is shown in **Figure 8**. Where if the lines of two factors are parallel, this means that there is no interaction. On the contrary, when the lines are far from being parallel, the two factors do interact. From the Figure, it is clear that the two lines is intersect in all the interaction plots, which indicated that the interaction of the main effects have statistically significant The experimental results and the predicted results for P_2O_5 conversion % were presented graphically in **Figure 9**. From the Figure, it is clear that there is a good agreement between experimental results and those predicted by the model.

Figure 7: Main Effects Plots for P₂O₅ conversion %Figure 8: Interaction effect plot for P₂O₅ conversion %

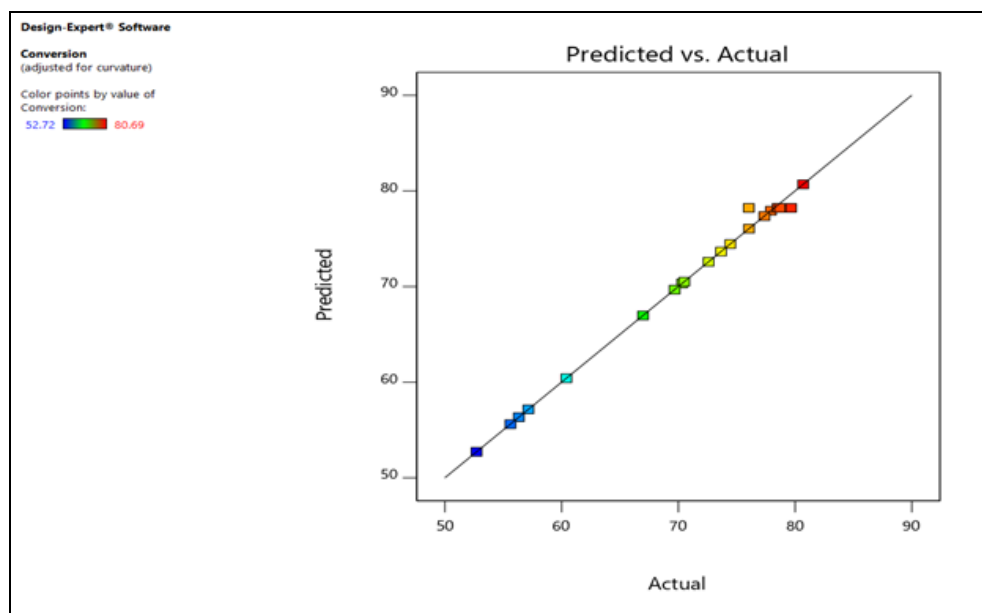


Figure 9: Scatter diagram of experimental values versus predicted values by Equations (2).

According to the statistical modeling performed, the optimal conditions for the P_2O_5 conversion % were : sulfuric acid concentration 70%, , and stirring time of 5 min. particle size 80%, and solid/ liquid ratio, g/ ml, 1/2. Regards to these optimal conditions, about 80.68 %, P_2O_5 conversion % was successfully produced to single super phosphate . The validation of the model was achieved by performing additional experiments under the predicted optimal conditions. The two experiments yielded a P_2O_5 conversion % of 80.68 %, which clear that there is an agreement between the predicted and experimental results confirmed the experimental adequacy of the model and the existence of the optimal conditions. This means that, the model developed was considered to be accurate and reliable for P_2O_5 conversion %.

CONCLUSION

The P_2O_5 conversion % has been investigated using the full factorial design methodology. The studied factors were sulfuric acid concentration, time, particle size %, solid/ liquid ratio. The obtained results were statistically analyzed by using analysis of variances (ANOVA) to determine the main effects and interactions between the investigated factors. The obtained results indicate that, both sulfuric acid concentration and solid/ liquid ratio, are effectively enhance the P_2O_5 conversion %, while both of time and particle size %, has slightly effect on the conversion efficiency 80.68 %, under the following optimum conditions; : sulfuric acid concentration 70%, , and stirring time of 5 min. particle size 80%, and solid/ liquid ratio, g/ ml, 1/2.

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Online publication Date: 22.5.2018