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Elaboration of novel adsorbent from Moroccan oil shale using Plackett–Burman design

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A B S T R A C T

The new adsorbents were prepared from Moroccan oil shale by chemical and physical process .In this study, experimental Plackett-Burman has been used as a screening method to study six factors for the development of materials to adsorbent basis of oil shale Moroccan. The factors have been identified by two levels, To Know temperature (°C), Processing time (min), mass ratio (m precursor/m acid), Pretreatment mixture the precursor with acid, origin of the raw material and type of the activating agent (H_2SO_4, H_3PO_4).And it was chosen as a response The maximum quantity of adsorption of the molecule of Methylene blue (Q_{ads} in mg/g) and the specific surface measure by the method bet (S_{bet} in m^2/g), The predicted values were in agreement with the experimental values with a coefficient of determination (R^2) of 0.98. The model has been validated by experiments subsequent to optimized conditions. The experimental data processing by software JMP 7 showed that the processing temperature The report of oil shale on the acid and activation time were the important effect on the maximal capacity of adsorption of methylene blue. The sample prepared at 237 °C during 215 min with pre-processing has a maximal capacity of adsorption equal to 54mg/g according to model of adsorption of Langmuir and SBET equal to 143 m^2/g.

Capsule Summary: A new adsorbent based on oil shale was prepared, characterized and performance as an adsorbent was studied in present investigation.

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INTRODUCTION

Metallurgical, chemical, ceramics, electro-galvanization, textile industrial waste effluents and pollutants such as dyes

and heavy metals reaches the aqueous sources and changes the water quality i.e. made inappropriate for drinking as well as commercial usage purpose(Asfaram et al., 2015; Tounsadi et al., 2016). Among the processes for the treatment of liquid discharges, adsorption remains a relatively used technique and easy to implement. Activated carbon is the most widely used adsorbent because of its high adsorption capacity. However, this adsorbent has a high cost and remains difficult to regenerate(Agarwal et al., 2016; Asfaram et al., 2015; Sahu et al., 2010). The search for another effective and less expensive adsorb is therefore interesting. Several studies have been carried out on the investigation of new precursor less expensive and do not use the forest reserves, such as peat, chitin(Ghorbel-Bellaaj et al., 2011), silica(Givianrad et al., 2013), sawdust(Johannes et al., 2013), bagasse pith and fly ash(Esfandiar et al., 2014; Reinik et al., 2015). From this point of view, the use of bituminous shale as year adsorb is of great interest because of its efficiency, their chemical composition, rich in active functions(Chen et al., 2010; Ichcho et al., 2005). The functional sites are due to the arrangement of the natural mineral component organic and within their structure (Chen et al., 2010; Oumam, 2003). Previous work has shown that the adsorbents developed from oil shales Moroccans by chemical activation or thermal possess good fixation capacities of organic molecules and chemical elements(Ichcho et al., 2005; Reinik et al., 2014). The preparation of activated carbon is influenced by many factors. For this reason experimental designs have been used to control the different factors which influence and interfere in preparation, in order to optimize experimental conditions(ICHOU, n.d.; Khouya et al., 2005). Among the various groups of designs, the Plackett–Burman designs can be used in screening studies for the detection of influential factors on the experimental response(Abbasi and Habibi, 2016a; Garba et al., 2015). The one-factor-at-a time (OFAT) approach is laborious, time consuming and less capable of finding true optimum levels due to the interactions among factors (Agarwal et al., 2016). On the other hand, statistically designed experiments could effectively solve such issues and minimize the error in determining the effect of factors and interaction between factors. The design of experiment (DOE) offers reduced number of experiments and increased process efficiency (El-Sheekh et al., 2016; Ma et al., 2016).

Statistical experimental designs such as PBD and CCD have been successfully applied to optimize many bioprocesses(Rahimdokht et al., 2016; Sadhukhan et al., 2016), Response Surface Methodology (RSM) is a collection of mathematical and statistical techniques that is applied for the modeling and analysis of various processes in which the response of interest is influenced by several factors and the response is optimized(Ghaedi et al., 2015; Witek-Krowiak et al., 2014). The objective for the present work was to Screening the variables using statistical techniques, a Plackett–Burman design was used to study the effects of factors on the preparation of the active coal has the basis of oil shale in order to extract the most important ones.

MATERIAL AND METHODS

Samples collection and preparation

In this study we used the oil shale of Tarfaya (layer R3) and of Timahdit (layer Y), collected in June 2013, and dried in stove at 80°C during 24H. The oil shale used was taken from South of Morocco. The organic matter of the oil shale was chemically linked to the mineral matter essentially formed by calcite, dolomite, silicate and clays. To free oil shale from carbonate (RH) and (YH) dissolution with HCl were carried out. The shale was grounded (approximately 0.06-0.08 mm in diameter). 20 g of sample was added to 80 ml of HCl (7M)(Abourriche et al., 2013). The mixture was then subjected to magnetic stirring during 4 h. The formed CO_2 was trapped by bubbling.

Activation

The sample R_3H and YH was treated using 80% of phosphoric acid or sulfuric acid. The choice of activating agent is based on several tests performed in our laboratory and also according to previous studies. To optimize activation condition, various experiments were performed using different temperatures (200-450°C) and different activation time (0.5- 4 hour). After thermal treatment, the sample was washed with distilled water in a Soxhlet extractor, to eliminate the excess acid and soluble matter (Mahaninia et al., 2015; Oumam, 2003). Then it was dried at 110°C to stable moisture content and was sieved. Fig. 1 summarizes the activation process of the sample.

Characterization

The samples for the following analytical tests were prepared to ASTM standards. Duplicate experiments were performed to mesure reproducibility. The porosity have been defined by adsorption of nitrogen / isotherms of desorption of the contents, by a porosimètre, analyzer of surface (BET).

Batch adsorption

In order to perform the experiments, a stock solution containing 1000 mg / BM was prepared by dissolving in the water Distilled. The desired dye concentrations were prepared from the stock solution by diluting for each adsorption experiment(Abbasi and Habibi, 2016b, p.). The concentration of the dye in each sample was analyzed with a UV–Vis spectrophotometer (UNICO, China), measuring the absorbance at λ_{max}=664 nm. Certain amount of developed adsorbent was placed in a 100ml Erlenmeyer flask containing 50 ml of dye solution. The following equation can be used to calculate the adsorption capacities (mg/g)(Abbasi and Habibi, 2016c; Kyzas and Deliyanni, 2015).

$$Qe = \frac{(Co-Ce)}{M} V \quad (1)$$

Where, Qe (mg/g) as adsorption capacities is the amount of the BM adsorbed on the adsorbent, C_0 is concentration of

the dye (mg/L) at initial time, C_e is the equilibrium concentrations of BM (mg/L), V (ml) is the volume of BM solution and M (g) is weight of developed adsorbent (Roosta et al., 2014).

Plackett–Burman Design

The Plackett–Burman Design (PBD) is an efficient screening method to identify the important factors among large number of factors that influences a process(Pawar et al., 2016). PBD was used to select the significant factors out of six factors considered in this study which influence the quality of the adsorbent material develops from Moroccan oil shale .For mathematical modeling the following first-order polynomial model was used:

$$Y = \beta 0 + \sum \beta iXi \quad (2)$$

Where, Y is the predicted response, β0 is the model intercept and βi is the linear coefficient and xi is the level of the independent variable (Azizi et al., 2014; Olivieri and Magallanes, 2012). Six factors (Table 1), (independent variables) viz. (i) temperature (A); (ii) Processing time (B); (iii) mass ratio (m precursor/m acid) (C); (vi) Pretreatment mixture the precursor with acid (D) ; (v) origin of the raw material (E) and ; (iv) type of the activating agent (H_2SO_4 , H_3PO_4) (F) were investigated to identify the significant factors for the adsorption of BM (response 1) and the surface area of the adsorbent prepared (response 2).

Statistical and data analysis

Statistical analysis of the model was performed to evaluate the analysis of variance (ANOVA). Analysis includes Fisher test (F-test), its associated probability P (F) and the coefficient of determination (R^2) which measure the goodness of fit of the regression model(Mohammadzadeh et al., 2016; Rhazi et al., 2015). JMP 7, USA software was used for designing experiments as well as for regression analysis of the experimental data obtained(Dayana Priyadharshini and Bakthavatsalam, 2016).

RESULTS AND DISCUSSION

Matric of experience

In this study, a 12 run Plackett–Burman Design was used to evaluate the six factors. Each independent variable was evaluated at two levels: -1 for the low level and +1 for high level. The experimental design of PBD (factors and tested range) is shown in Table 2. The best adsorbents properties were observed for particular combination of the experimental parameters: Origin of oil shale, temperature, Type of acid, acid/shale weight ratio, Pretreatment mixture the precursor with acid and time of activation. The highest specific surface area is obtained for the adsorbent treated at 250°C. At 250°C, one favors the oxidation of the organic matter and the release of gases which create porosity. At 450°C, one expects reactions of recombination and insertion of heteroatom in the structure with a diminution of the porosity. The maximum S_{BET} was at tained for carbonization time of 0.5 h. For Long time (4 h) the results suggest that carbon structure pronounced reduction in surface area.

Study of response 1 (Q_m (mg/g))

The theoretical capacity of adsorption corresponding to the maximum quantity of solute which can be adsorbed by unit of mass of adsorbent costs in the operating conditions. The adsorption capacity of a coal defines its ability to eliminate impurities in our case the molecule of methylene blue.

Evaluation of the quality of the model

The linear regression analysis represented in the graph is established by the software, and will be presented as follows. Fig. 2 presents a projection of the values of the maximum quantity of adsorption provided and observed for the two models. After the graph of correlation there is a regular distribution, the experimental performance is close to the theoretical right; the graph illustrates the good correlation between the values observed and that predicted with a coefficient of determination R^2 in the order of 0.98 which remains acceptable (Table 3). It is observed that the value of R^2 =0.98 and R^2 adjusts =0.95 are very close .This translated that the observed variation is explained by the direct effects of the factors. This coefficient is very close to 1, so the quality of the adjustment of the plan of PLACKETT–BURMAN chooses for the screening of conditions is best.

Test of analysis of variance ANOVA

Analysis of the variance: Based on the comparison of the variance in the model established by report to the variance of the Residue, through test of "fisher Snedecor". For the model to be very significant at 95%, it is necessary that: Fexp >> Fα, ν_{mod}, ν_{res}, where α = 0.05. The results of the ANOVA given by JMP software are represented by the Table 4. The results of the analysis of the variance between the model established and the residue, give an experimental factor F_{exp}= variance in the model/ variance of the Residue =37.5234. And according to the table of Fisher Snedecor, (for ν_{model} = 6, and $\nu_{résidu}$ = 5, and a threshold of confidence = 5%), it was $F_{théo} = F_{(0.05, 6, 5)}$ = 4.39. After the results, it was F_{exp} = 37.5234>> $F_{théo}$= 4.39, the condition of the Fisher test Snedecor is checked, therefore the regression is significant with a confidence level of 5%.

Determination of the equation of the model

The coefficients of the equation of the model Plackett-Burman are given by JMP software and are represented in the Table 5. According to this graphical representation, we note that the effects of factors temperature, time and pretratitment are very significant with a value of p value < 0.0001. The equation of our model after elimination of all factors having a P-value superior of 0.05 is the following the response submitted by the performance of adsorption of functions of the descriptors The most relevant.

$$Y_1=54.91+ (12.75\times A) + (6.25\times B) - (5.08\times D) \quad (3)$$

Table 1: Levels of the factors tested in Plackett-Burman design

Factors	Symbol	Experimental value	
		Low (-1)	High (+1)
The processing temperature of the precursor (YH, RH)	T(°C)	200	450
Heat treatment time	T(min)	30	240
Mass ratio ($m_{precursor}/m_{acid}$)	R_m	1	3
Pretreatment mixture the precursor with acid	Pret.	Y(yes)	N(no)
Origin of the raw material (Tarfaya R or Timahdit Y	Type of OS	Y	R
Type of the activating agent (H_2SO_4 , H_3PO_4)	Type of acid	P	S

Table 2: Plackett–Burman design of factors (in coded levels) with the maximum amount of adsorption of Methylene blue molecule and Specific surface as response

Run Order	A	B	C	D	E	F	Qm(mg/g)	S_{BET} (m^2/g)
							Experimental Value	Experimental Value
1	-	+	-	+	+	+	68	125
2	+	-	-	+	-	+	52	114
3	-	+	+	+	-	-	70	188
4	-	-	+	-	+	+	32	298
5	-	+	-	-	+	-	53	131
6	-	-	+	-	-	+	35	315
7	+	-	-	-	+	-	42	140
8	+	+	+	+	+	+	78	102
9	-	-	-	+	-	-	34	180
10	+	+	-	-	-	+	65	125
11	+	+	+	-	-	-	72	134
12	+	-	+	+	+	-	58	142

A-temperature (°C); B-Processing time (min); C-mass ratio ($m_{precursor}/m_{acid}$) ; D-pretreating mixture the precursor with acid ; E-origin of the raw material and ; F- type of the activating agent (H_2SO_4 , H_3PO_4).

Response to SBET (m^2/g)

The specific surface or mass area (m^2/g) is the total surface area per unit mass of active coal accessible to molecules. The entire surface area of the particles of the active carbon is considered, open porosity understood, for the calculation of the specific surface which accumulates so the inner surface of all pores. The specific surface area includes the external surface and the internal surface of the active carbon. The inside surface is the surface microporous represented by the walls of the microspores. The external surface is the surface non-microporous which includes the walls of the mesopores and macrospores, as well as the non-porous surface of the sample. In our case the surface varies between 102 and 315 m^2/g approximately. From a physical point of view, the difference between the internal surface and the external surface is that the value of the energy of adsorption may be up to two times greater on the walls of the microspores that on the external surface. This phenomenon is explained by the presence of two opposite walls close to creating a double interaction for a adsorbed molecule in a microspore.

Table 3: Adjusted regression of the model

Adjusted regression	Values
R square	0,978274
R Square Adjusted	0,952203
The root of the mean square error	3,533176
Average of the response	54,91667
Observations (or are weighted)	12

Linear regression of the model

Table 4: Analysis of the variance is determined by the software JMP

Source	Degrees of Freedom	Sum of squares	Average Square	Report F
Model	6	2810,50	468,41	37,52
Residues	5	62,41	12,48	Prob. > F
Total	11	2872,91		0,0005

Table 5: Interaction between the various factors

Term	Estimate	SD*	Report t	Report t	Prob.>|t|	Symboles
Constant	54,91	1,02	53,84		<,0001*	Y_0
T (min)	12,75	1,02	12,50		<,0001*	A
T (°C)	6,25	1,02	6,13		0,0017*	B
Pret	-5,08	1,02	-4,98		0,0042*	D
Rm	2,58	1,02	2,53		0,0523	C
Type of OS	-0,25	1,02	-0,25		0,8161	E
Type of acid	-0,08	1,02	-0,08		0,9381	F

*SD = Standard deviation

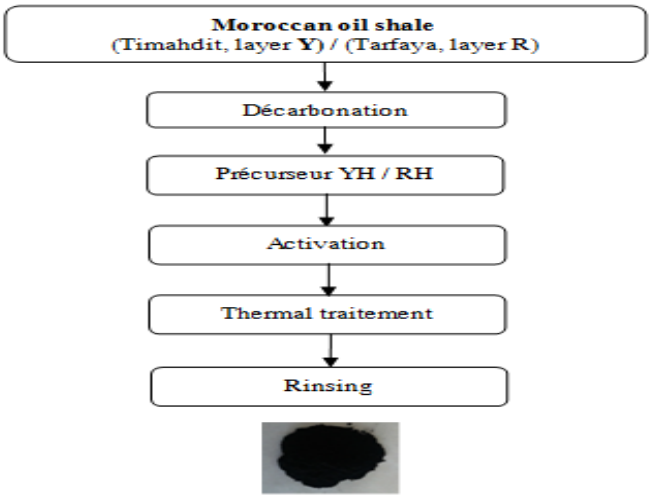

Fig. 1: Diagram for activation process

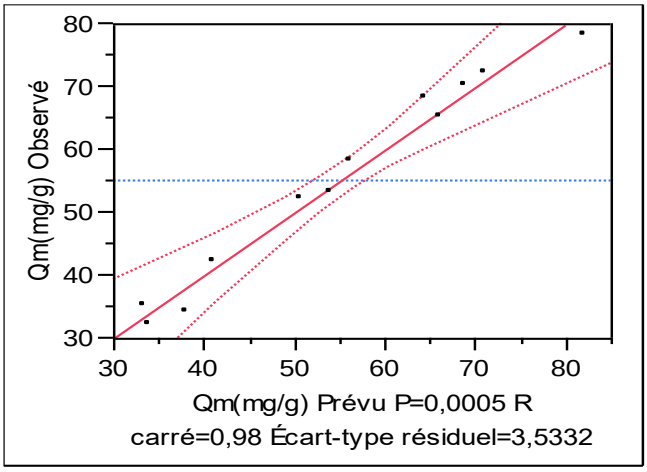

Fig. 1: Graphical representation of observed values as a function of predicted values

Table 6: Summary of the adjustment

Adjusted regression	Values
R square	0,982743
R Square Adjusted	0,962034
The root of the mean square error	13,64795
Average of the response	166,1667
Observations (or are weighted)	12

The graphic representation of the linear regression analysis is established by the software, presented in the Fig. 3. R^2 is equal 0.98 therefore the model is validated and can exploit this result to meet the objective. According to the graphic representation, the coefficient of linear regression R^2 =0.98 is

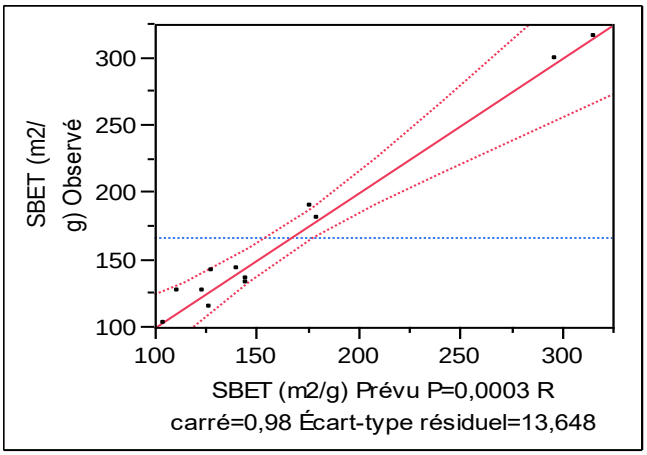

Fig. 2: Graphical representation of observed values as a function of predicted values

Table 7: Analysis of the variance determined by the JMP software

Source	Degrees of Freedom	Sum of squares	Average Square	Report F
Model	6	53036,33	8839,39	47,45
Residues	5	931,33	186,27	Prob. > F
Total	11	53967,66		0,0003

Table 8: Graph of the effect of different factors

Term	Estimate	SD*	Report t	Report t	Prob.>|t|	Symboles
Constant	166,16	3,94	42,18		<,0001	Y_0
T (°C)	-40	3,94	-10,15		0,0002	A
T (min)	-32	3,94	-8,12		0,0005	B
R_m	30,33	3,94	7,70		0,0006	C
P_{ret}	24,33	3,94	6,18		0,0016	D
Type of acid	-13,66	3,94	-3,47		0,0179	F
Type of OS	9,83	3,94	2,50		0,0548	E

greater than 0.80 what shows that the good choice of the plan of screening Plackett-Burman. It is observed that the value of R^2 =0.98 and R^2Adjusts =0.96 are very close .This reflects the fact that more than 96% of the variation observed are explained by the direct effects of factors. This coefficient is very close to 1, so the quality of the adjustment of the plan Plackett-Burman, chooses for the screening of factors in order to have a good performance is best (Table 6).

Test of analysis of variance ANOVA

Based on the comparison of the variance in the model established by report to the variance of the Residue, through test of "fisher Snedecor". For the model to be very significant at 95%, it must be that: $F_{exp} >> F_{\alpha}$, ν_{mod}, ν_{res} where α = 0.05.The results of the ANOVA given by JMP software are represented by the Table 7. The results of the analysis of the variance between the model established and the residue, give an experimental factor F_{exp}= variance in the model/ variance of the Residue = 47.45. And according to the table of Fisher Snedecor, (for ν_{model} = 6, and $\nu_{résidu}$ = 5, and a threshold of confidence = 5%), it was $F_{théo}$ = F 0 05, 6, 5 = 4,39 . According to the results, it was Fexp = 47.45>>$F_{théo}$= 4,39 the condition of the Fisher test Snedecor is checked, therefore the regression is significant with a confidence level of 5%

Determination of the equation of the model

The coefficients of the equation given by JMP software and are represented in the table 8. According to this graphical representation, one finds that all the factors are significant except the effect of the type of acid used their P valu is greater than 0.05.So after the results of the JMP software, the equation of the model used for the screening of factors influence on the process of development is written as follows:

$$Y_2 = 166.16 - (40\times A) - (32\times B) + (30.33\times C) + (24.33\times D) - (13.66\times F) \quad (4)$$

Prediction Profiler for the two responses

The Prediction Profiler provides by the diagram below confirms that result in effect, the factors that appear to be more influential are the temperature, duration, the report and the mode of processing an analysis of the diagram also allows to conclude that its factors affect the responses of the way antagonist. It is clear that an increase of the temperature and the duration of treatment causes an increase in the adsorption capacity of the material; on the other hand it can produced a decrease in the specific surface, for pretreatment, it may be favorable for the specific surface but not for the adsorption capacity.

CONCLUSIONS

The screening of the conditions of preparation of materials adsorbents to basis of bituminous shales of Morocco had been achieved by the methodology of the plans of experience in the study of the effect of certain operating parameters, temperature, Processing time, mass ratio ($m_{precursor}/m_{acid}$), pretreating mixture the precursor with acid origin of the raw material (E) and type of the activating agent (H_2SO_4, H_3PO_4), On the process of development. We have assessed the performance of the adsorbents prepared by studying their capacity of adsorption of the molecule of Methylene blue and the specific surface area of the adsorbents developed.

The results obtained and the adsorption capacity of the Methylene blue and the values of the specific surface area have led to the conclusion that the factors most influence are the temperature the time and the percentage of acid and for the qualitative factors The best adsorbent developed from the oil shales of the region of Tarfaya by the activation of the precursor by the pretreatment with phosphoric acid.

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