International Journal of Scientific Research in Chemical Sciences **Research Paper**
Vol.7, Issue.3, pp.11-19, June (2020) **E-ISSN:** 2455-3174

Adsorption of Nylosan Red onto Grapes Cores-based Activated Carbon

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Available online at: www.isroset.org

Received: 27/May/2020, Accepted: 05/June/2020, Online: 30/June/2020

***Abstract*—** The aim of this study is to evaluate effectiveness of activated carbons prepared from grapes cores chemically activated using Calcium chloride ($CaCl_2$), Phosphoric acid (H_3PO_4) and Potassium hydroxide (KOH) as oxidizing agents representing GC-1, GC-2 and GC-3 adsorbents respectively in order to remove Nylosan Red (NR) dye from simulated wastewater. Both linear and non-linear forms were used for the evaluation of two common isotherm models, Langmuir and Freundlich isotherms parameters.
In order to justify the validity of the values, statistical aspects for both forms were checked by considering R^2 for best fit linear isotherm and P-Values and other important errors functions such as Chi-square statistic & Root mean square error and Average percentage error for non-linear regression method.
High removal efficiencies of 90.5 % and 98 % NR at adsorbent dose (4 g/L) and initial dye concentration (100 mg/L) were obtained by GC-2 and GC-3 respectively. However, 88.31 % removal was obtained by GC-1 using adsorbent dose (8 g/L).
The best fit for Freundlich model in its linear form, was well represented by NR adsorption onto GC-3 with a capacity of 28.146 mg/g, with very low errors function values, while GC-1 and GC-2 were moderate.
Non-linear Freundlich isotherm models present a good results compared to linear models in terms of adsorption energy, the most important is related to the adsorption of NR by GC-3 with a value equal to 42.57 mg/g, also as a finding the adsorption of NR by GC-1 presents a low but acceptable adsorption capacity compared to the adsorption isotherm using GC-2 and GC-3.

***Keywords*—** Modals; Dyes; Adsorbent; Langmuir; Freundlich; Isotherms

I. INTRODUCTION

The performance and the effectiveness of the adsorption techniques depend deeply of the nature of the adsorbent, particularly: the cost, the availability and the regeneration. Part of the sustainable development and based on Lavoisier's law in matter of conversation of mass "nothing is lost, nothing is created, everything is transformed", this theorem can also be projected in the waste management system, thus we have prepared an adsorbent from agro-wastes for dye removal from textile industry effluents.

Among a huge variety of wastes, grape marc an agricultural waste, initially rich in carbon can be considered as a potential precursor for activated carbon preparation was chosen in this study as adsorbent.

Chemical or physical treatments for this type of waste are usually needed to enhance its adsorption capacity with respect the organic compound to be removed.
Sorption processes such as reverse osmosis, electro dialysis, precipitation and adsorption techniques have been frequently employed to achieve required degree of removal [20, 21, 22].

Due to the complexity of solving isotherm models involving three or several parameters, the majority of adsorption studies are based on isotherm models having one or two-parameters especially those linear.

In this study, we have assessed the adsorption processes using Langmuir and Freundlich isotherm models in their linear and non-linear forms in order to make a comparative study of an azo dye adsorption onto the grape cores-based activated carbons chemically treated by different oxidizing agents.

The paper is organized as follows, Section I contains the introduction of the adsorption techniques and mathematical models which were used, Section II contains the related work of dye capture by using agriculture waste, Section III contains the dye specification, the process of the preparation of the adsorbent and the experiments; models which have been used, Section IV describes results, discussion and comparison of the adsorption of NR by different grape cores-based activated carbons, separation factors and coefficient of distribution and all the linear and non-linear isotherms are represented in figures, and Section V concludes research work with future directions.

II.RELATED WORK

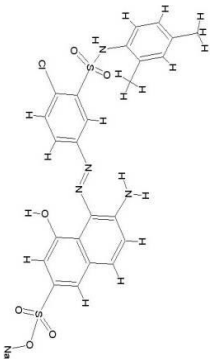
Different conventional methods exist for wastewater treatments such as precipitation, oxidation, floatation-coagulation and electro-coagulation but their use is limited due to many constraints.
In this research we have focused on the adsorption as a method of wastewater treatment using an activated carbon prepared from grape cores as a waste.

III. METHODOLOGY

III. 1 Stock solutions preparation

Nylosan Red belongs to the azo anion acid dyes family, it is very toxic substance, Table 1 shows chemical structure and characteristics of Nylosan Ref-dye.

Table 1. Chemical structure and characteristics of Nylosan Red-dye

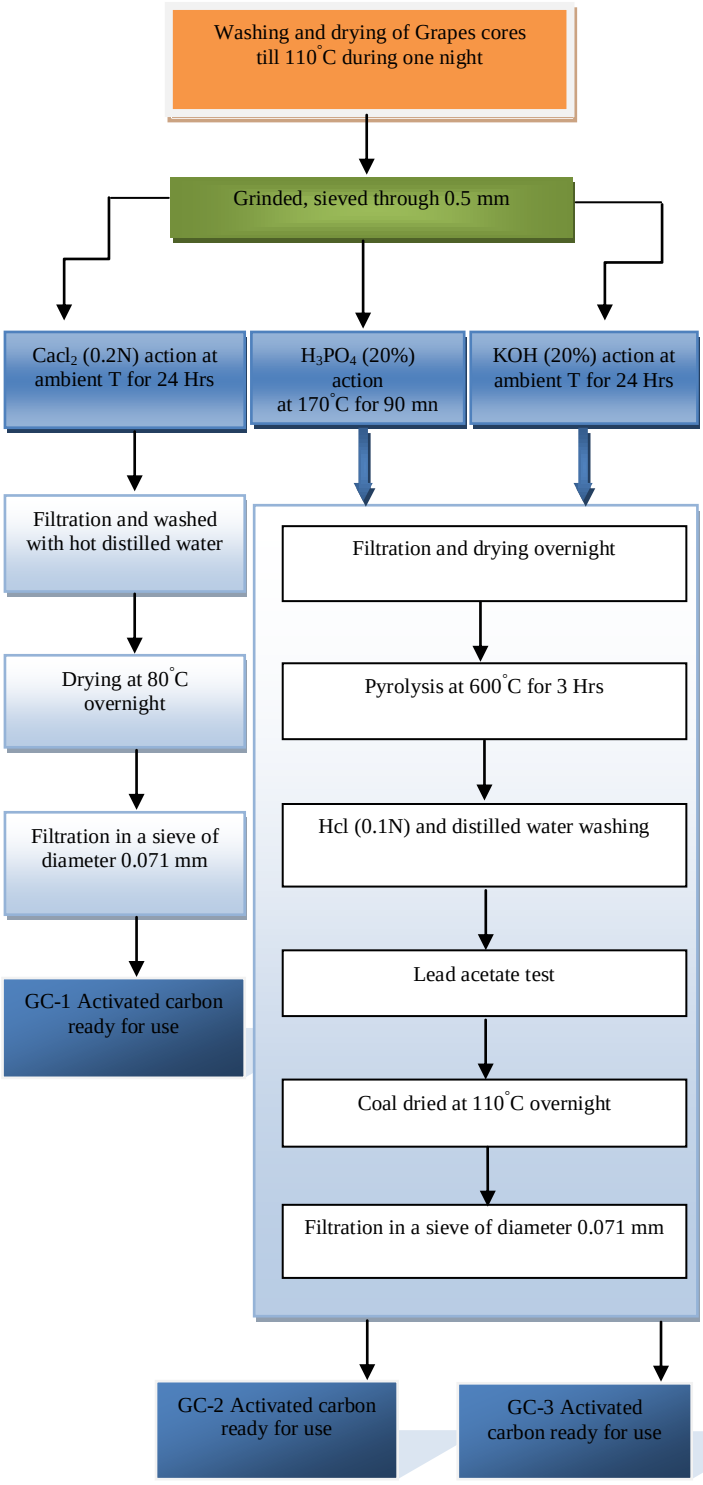
	Characteristics	Chemical structure
Molecular formula	$C_{24}H_{21}ClN_4O_6N_2Na$	
Molecular weight	587.97 g/mol	
Max wave length $(\lambda_{max})^*$	500 nm	
LD_{50}(mice)	5000 mg/Kg	
LC_{50}	90 mg/g	
Solubility in H_2O	80 g/L at 90°C	

** Experimentally obtained value*

III. 2 Adsorbents preparation

In order to eliminate impurities, dust and to reduce moisture, grapes cores samples were washed several times with distilled water then dried in an oven overnight at 110 °C. The obtained samples were then grinded, sieved through 0.5 mm for intimate chemical agent-particle contact using a Vierzen Crosshop grinder before activation. Depending on the chemical agent chosen, three grape cores-based activated carbons were prepared in a two-steps process involving a chemical impregnation using phosphoric acid, hydroxide potassium and calcium chloride separately, followed by pyrolysis at 600 °C for 3 hours for each case as shown in scheme 1.

GC-1: Grapes cores chemically activated with $CaCl_2$
GC-2: Grapes cores chemically activated with H_3PO_4
GC-3: Grapes cores chemically activated with KOH
NR: Nylosan Red-dye



Scheme 1. A two steps-process carried out on activated process on grapes cores (Cases: GC-1/GC2/GC-3 preparation)

III. 3 Adsorption experiments

Time, dose and pH, these parameters were chosen to establish adsorption isotherms for different systems, 25 mL solution of initial concentrations ranging between 100-2400 mg/L were mixed with an optimal dose of each adsorbent, the resulting suspension was then agitated at a speed of 200 rpm until equilibrium has been established. After the adsorption process had occurred, the resulting

solution in each flask was centrifuged at 4000 rpm and the supernatant analysed using a UV-Visible 2121 Optizen spectrophotometer at wave value of 500 nm. The following relationships were used to evaluate adsorbent-adsorbate interactions.

Dye uptake $q_e(mg/g)$ and the coefficient of distribution $K_d(L/g)$ are calculated according to the equations (1) and (2)

$$q_e = \frac{(C_0 - C_{eq})}{m*1000} * V \quad (1)$$

$$K_d = \frac{(C_0 - C_{eq})}{C_{eq} * m * 1000} * V \quad (2)$$

Where, $C_0(mg/L)$ is the initial dye concentration; $C_{eq}(mg/L)$ is the equilibrium concentration; $m(g)$ the mass of the adsorbent; $V(mL)$ the volume of the adsorbate.

The performance of the adsorption is evaluated by using the removal efficiency or elimination rate $R_E(\%)$, its formula is as indicated below

$$RE(\%) = \frac{(C_0 - C_t)}{C_0} * 100 \quad (3)$$

The equilibrium parameter R_L is considered as an essential characteristic of the Langmuir isotherm which is interpreted as follow and defined by equation (4)
The nature of the adsorption process can be unfavourable $R_L \rangle 1$; linear $R_L = 1$; favourable $0 \langle R_L \langle 1$ or irreversible $R_L = 0$.

$$R_l = \frac{1}{1 + bC_0} \quad (4)$$

$b(L/mg)$ is the Langmuir equilibrium constant related to the energy of adsorption

III. 4 Linear and non-linear Langmuir and Freundlich isotherms models

The isotherms data were analysed using two of the most commonly used equilibrium models namely Langmuir and Freundlich isotherms.

The Langmuir isotherm is based on the assumption that it predicts monolayer coverage of the adsorbate on the outer adsorbent surface. It also suggests that there is no lateral interaction between the sorbed molecules [1]/[3].

The Freundlich isotherm is based on multilayer adsorption on the heterogeneous surface [2]/[3].
Where, n is a Freundlich constant indicating the intensity of adsorption
$K_F(mg/g)$ is a Freundlich constant corresponding to the energy of adsorption
$q_m(mg/g)$ Maximum adsorption capacity

The adsorption intensity "n" gives an indication on the possibility of adsorption, values in the range of 2 to 10 indicate favourable adsorption, for $1 \langle n \langle 2$ the adsorption capacity is moderated and for $n \langle 1$ it means the capacity of adsorption is low.

Unlike the linear analysis, a different isotherm would significantly affect the R^2 value and impact the final determination of parameters, where the use of the non-linear method would avoid such errors [4].
The expressions of models are listed in table 2.

Table 2. Linear and non-linear forms of the Langmuir and Freundlich isotherms models

Isotherms	Non-linear	Linear	Plot
Freundlich	$q_e = K_F C_{eq}^{1/n}$	$\ln(q_e) = \ln K_F + (1/n) * \ln(C_e)$	$\ln(q_e)$ VS $\ln(C_e)$
Langmuir-I	$q_e = \frac{q_m b C_e}{1 + bC_e}$	$C_e/q_e = (C_e/q_m) + (1/b*q_m)$	C_e/q_e VS C_e
Langmuir-II		$1/q_e = (1/b*q_m)*(1/C_e) + (1/q_m)$	$1/q_e$ VS $1/C_e$
Langmuir-III		$q_e = q_m - (1/b)*(q_e/C_e)$	q_e VS q_e/C_e
Langmuir-IV		$q_e/C_e = b*q_m - b*q_e$	q_e/C_e VS q_e

III. 5 Error functions analysis

Error function such as, Chi-square (χ^2), Root mean square error ($RMSE$) and Average percentage error (APE),are used in order to evaluate models, if data from the model are similar to the experimental data, χ^2 will be a small number, if they are different, χ^2 will be a large number and the smaller the *RMSE* value, the better is the curve fitting. In general, more lower are errors function values, good agreement between the experimental and calculate and best model convergence becoming then favourable. Their functions are listed below:

$$Chi - SquareStatistic = x^2 = \sum \frac{(q_{e,\exp} - q_{e,cal})^2}{q_{e,cal}} \quad (5)$$

$$RMSE = \sqrt{(1/N-2) * \sum_1^N (q_{e,\exp} - q_{e,cal})^2} \quad (6)$$

$$APE = (\sum_1^N (|q_{e,\exp} - q_{e,cal}| / q_{e,\exp}) / N) * 100 \quad (7)$$

With, N is the number of observations in the experimental data.

It has been reported that it is inappropriate to use the coefficient of determination of linear regression analysis for comparing the best-fitting solution of different isotherms [4-5], that is why it is very important to study other error functions.

The non-linear and linear isotherm models having two parameters were used, non-linear regression analysis gives valid values for the intervals or missing observations and it

offers a chart easy to understand but it is necessary to justify the validity of every model linear and or non-linear by checking the statistical aspect by taking into consideration values of coefficients of determination, P-Values and other important errors functions such as Chi-square statistic & Root mean square error and Average percentage error.

IV. RESULTS AND DISCUSSION

IV. 1 Adsorption of NR by GC-1

Results of the adsorption isotherm by using an amount of adsorbent equal to 0.2g are:

-We have found that the highest values of K_d (Figure 7) and R_E (Figure 8) are equal to 1.668 L/g and 93.03 % respectively and correspond to an initial concentration C_0 equal to 200 mg/L and 23.257 mg/g of dye uptake q_e.

-Equilibrium factors R_L values (Figure 1) are inversely proportional to the initial and equilibrium concentrations.

-The only Langmuir isotherm model which is valid and can describe the equilibrium data is the linear model type-II, its coefficient of determination is equal to 0.874; it has a maximum adsorption capacity and adsorption energy equals to 339.753 mg/g and 0.0035 L/mg respectively, in the other hand non-linear Langmuir isotherm model is invalid due to higher values of errors functions and low coefficient of determination.

-Linear Freundlich isotherm model has low values of errors functions, the coefficient of determination is equal to 0.9453 but the model is moderate due to the low value of the adsorption intensity of 1.133 the adsorption energy found is equal to 1.5706 mg/g, same findings for the non-linear isotherm, the coefficient of the determination is high equal to 0.9766, the adsorption capacity and intensity values are very low equal to 0.9097 mg/g and 0.9896 respectively.

All isotherm models fittings are represented in Figure-2

IV. 2 Adsorption of NR by GC-2

-For all initial NR concentrations (100-1000) mg/L and an absorbent dose of 0.1 g, the removal efficiencies values are greater than 90%.

-By using 0.1 g of GC-2 and for all initial dye concentrations ranking between (100-1000) mg/L, the removal efficiency (Figure 8) is above 90 %, at C_0 =100 and 2400 mg/L $R_E(\%)$ is equal to 90.5 and 75.16 respectively.

-The coefficient of determination from linear Langmuir isotherm model type-I is slightly higher than type-II, the *APE* calculated are equal to 15.918 % and 38.535 % respectively.

-Non-linear Langmuir isotherm model is favourable because the separation factor value is ranging between 0 and 1 and its coefficient of determination and the Langmuir equilibrium constant are slightly higher than that obtained from linear Langmuir isotherm model type-I, their values are equal to 0.9553 and 0.01 L/mg respectively, the maximum adsorption capacity is equal to 470.6 mg/g and the *APE* is about 17.829 %.

-The coefficient of determination from Freundlich linear model is equal to 0.859, the adsorption energy found is equal to 12.304 mg/g, the adsorption capacity is moderate because the intensity is equal to 1.696.

-Regarding the non-linear Freundlich isotherm model it has a coefficient of determination high equal to 0.9392 and an *RMSE* equal to 33.936, the adsorption energy is equal to 2.302, which mean that the process is favourable, its adsorption capacity is 27.5 mg/g

All isotherm models fittings are represented in Figure-4

IV. 3 Adsorption of NR by GC-3

-By using 0.1 g of adsorbent, it is apparent that more the initial concentration decrease more the removal efficiency and coefficient of distribution increase, all initial dye concentrations less than 1000 mg/L gave an elimination rate more than 90 %, therefore C_0 is inversely proportional to R_E and K_d (Figure 8).

-Separation factors values are between $0\langle R_L \langle 1$ and the parameters found from different Langmuir isotherm models are valid, the best one is model type-I, the adsorption capacity found is 419.13 mg/g and R^2 is equal to 0.983. Non-linear Langmuir isotherm model is favourable too, the *APE* is about 16.197 %, R^2 =0.9585 and the energy and maximum adsorption capacity are equal to 0.0203 L/mg and 410.4 mg/g respectively.

-Linear Freundlich isotherm model presents a coefficient of determination equal to 0.943; the adsorption intensity is about 2.128, which means that the adsorption capacity is good, values of errors function are low, therefore this model is favourable.

-The coefficient of determination obtained from the non-linear Freundlich isotherm model and the adsorption intensity are higher than those obtained from the linear Freundlich isotherm model, they are equal to 0.9787 and 2.65 respectively and those obtained from the linear model are equal to 0.943 and 2.128 respectively, both models are favourable with an advantage to the non-linear because the adsorption energy is the greatest, it is equal to 42.57 mg/g,

All isotherm models fittings are represented in Figure-6

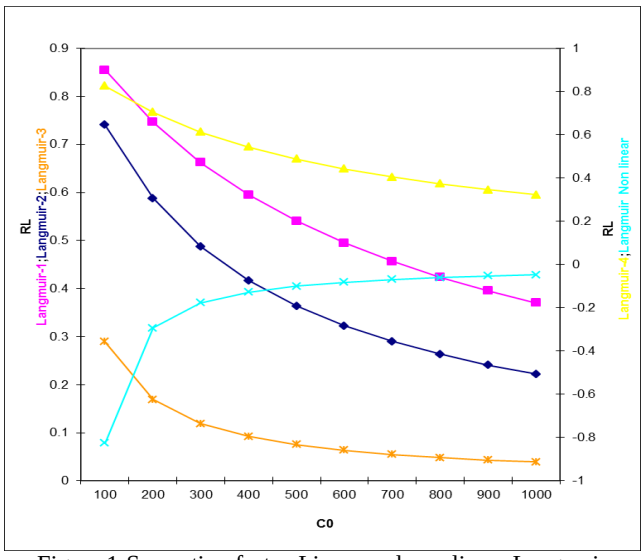


Figure 1-Separation factor-Linear and non-linear Langmuir isotherms models (GC-1 case)

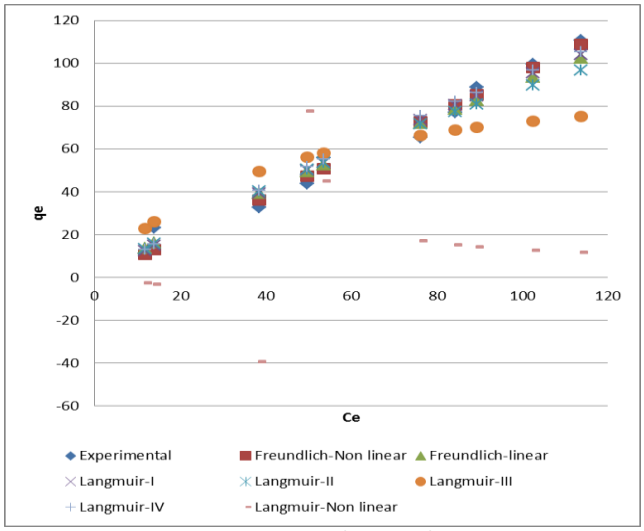


Figure 2-Linear and non-linear fittings of the Langmuir and Freundlich isotherm models for GC-1

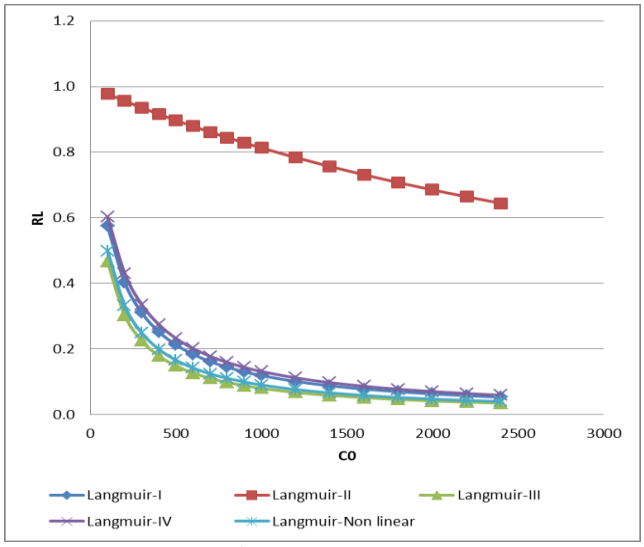


Figure 3-Separation factor-Linear and non-linear Langmuir isotherms models (GC-2 case)

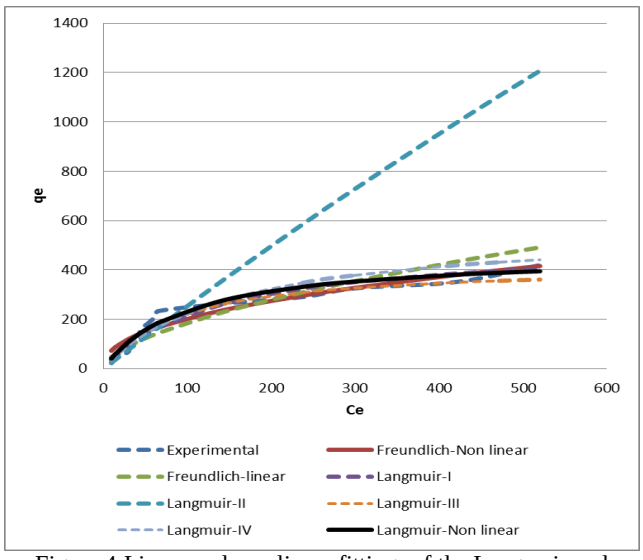


Figure 4-Linear and non-linear fittings of the Langmuir and Freundlich isotherm models for GC-2

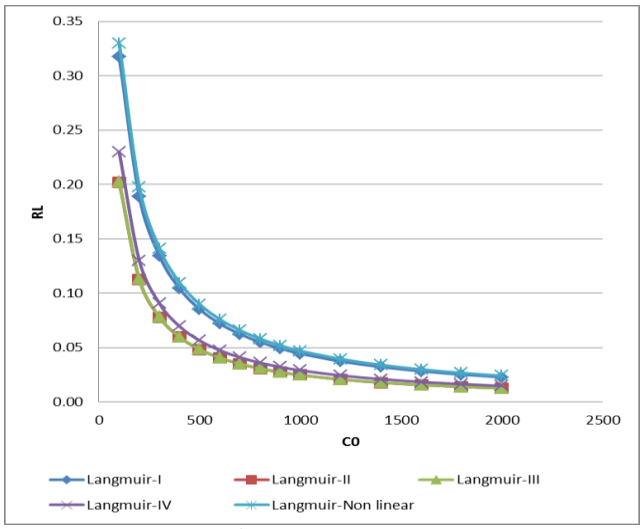


Figure 5-Separation factor-Linear and non-linear Langmuir isotherms models (GC-3 case)

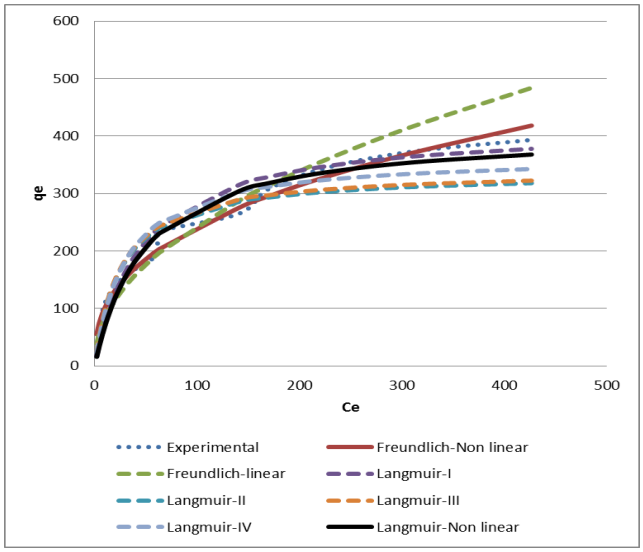


Figure 6-Linear and non-linear fittings of the Langmuir and Freundlich isotherm models for GC-3

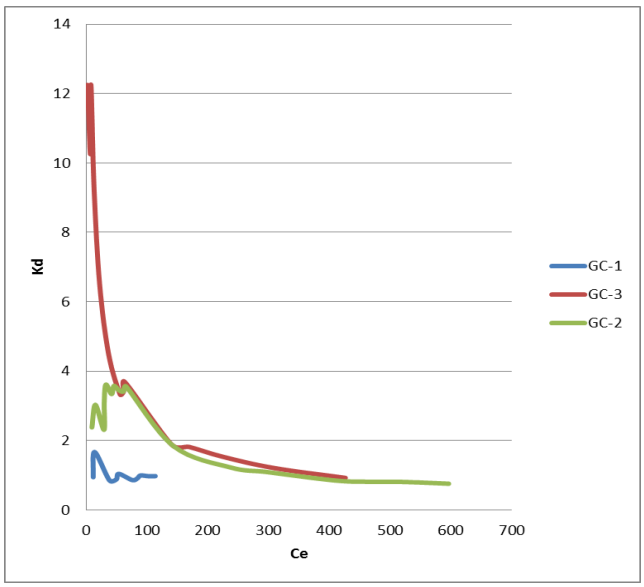


Figure 7-Coefficient of the distribution curves (GC-1/GC-2/GC-3 cases)

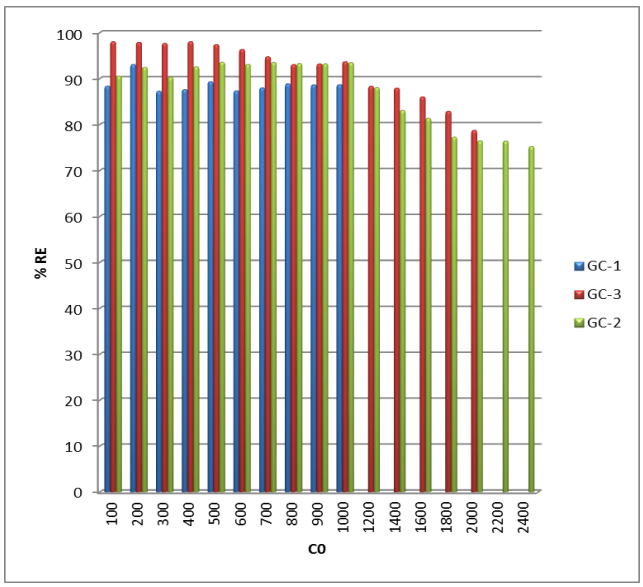


Figure 8-Evolution of RE for the adsorption isotherms of NR (GC-1/GC-2/GC-3 cases)

Table 3. Parameters of the Linear and non-linear forms of the Langmuir isotherms models

Models	Type	R^2	$b(L/mg)$	$q_m(mg/g)$	χ^2	*RMSE*	*APE*
Adsorption of NR by GC-1 ($CaCl_2$ activation)							
$q_e = 0.93 * C_e + 1.888$	*Experimental*	0.977	-	-	7.539	5.344	10.909
$\frac{C_e}{q_e} = 0.926 + 0.0016 * C_e$	*Linear* – I	0.112	0.0017	642.396	0.224	0.165	13.448
$\frac{1}{q_e} = 0.0029 + 0.8457 * \left(\frac{1}{C_e}\right)$	*Linear* – II	0.874	0.0035	339.753	0.012	0.0094	14.534
$q_e = 101.82 - 40.606 * \left(\frac{q_e}{C_e}\right)$	*Linear* – III	0.084	0.0246	101.824	146.908	33.944	81.504
$q_e/C_e = 1.136 - 0.0021 * q_e$	*Linear* – IV	0.084	0.0021	545.929	0.439	0.243	13.803
$q_e = \frac{-0.155 * C_e}{1 - 2.21E - 02 * C_e}$	*Non – Linear*	0.03	–0.0221	7.025	1811.51	67.49	96.719
Adsorption of NR by GC-2 (H_3PO_4 activation)							
$q_e = 0.613 * C_e + 110.67$	*Experimental*	0.8585	-	-	281.78	51.782	51.984
$\frac{C_e}{q_e} = 0.2627 + 0.002 * C_e$	*Linear* – I	0.957	0.0074	509.172	0.25	0.086	15.918
$\frac{1}{q_e} = 9E - 05 + 0.378 * \left(\frac{1}{C_e}\right)$	*Linear* – II	0.954	0.00023	11297.3	0.02	0.002	38.535
$q_e = 421.78 - 87.164 * \left(\frac{q_e}{C_e}\right)$	*Linear* – III	0.575	0.0114	421.78	676.49	89.693	94.589
$q_e/C_e = 3.761 - 0.0066 * q_e$	*Linear* – IV	0.575	0.0066	569.883	3.72	0.78	30.813
$q_e = \frac{4.723 * C_e}{1 + 1.003E - 02 * C_e}$	*Non – Linear*	0.9553	0.01004 ± 0.00149	470.6 ± 24.09	67.98	29.1	17.829
Adsorption of NR by GC-3 (KOH activation)							

$q_e = 0.836 * C_e + 113.21$	*Experimental*	0.8215	-	-	215.58	51.833	49.567
$\frac{C_e}{q_e} = 0.1107 + 0.0024 * C_e$	*Linear* – I	0.983	0.0215	419.137	0.094	0.041	18.757
$\frac{1}{q_e} = 0.003 + 0.075 * \left(\frac{1}{C_e}\right)$	*Linear* – II	0.994	0.0395	336.951	0.0012	0.0008	10.548
$q_e = 341.79 - 25.498 * \left(\frac{q_e}{C_e}\right)$	*Linear* – III	0.851	0.0393	341.787	243.308	47.376	20.846
$\frac{q_e}{C_e} = 12.283 - 0.0335 * q_e$	*Linear* – IV	0.851	0.0335	366.756	–7.742	1.72	48.613
$q_e = \frac{8.35 * C_e}{1 + 2.034E - 02 * C_e}$	*Non – Linear*	0.9585	0.0203 ± 0.0034	410.4 ± 21.6	80.78	24.994	16.197

Table 4. Parameters of the Linear and non-linear forms of Freundlich

Models	R^2	n	$K_F(mg/g)$	χ^2	*RMSE*	*APE*
Adsorption of NR by GC-1 ($CaCl_2$ activation)						
$q_e = 0.909 * C_e^{1.0105}$	0.9766	0.9896 ± 0.0797	0.9097 ± 0.3317	10.154	5.43	9.561
$\ln(q_e) = 0.451 + 0.882 * \ln(C_e)$	0.9453	1.1331	1.5706	0.0869	0.1806	3.8146
Adsorption of NR by GC-2 (H_3PO_4 activation)						
$q_e = 27.503 * C_e^{0.434}$	0.9392	2.302 ± 0.185	27.5 ± 5.49	136.44	33.936	31.497
$\ln(q_e) = 2.509 + 0.589 * \ln(C_e)$	0.859	1.696	12.304	0.3491	0.323	5.499
Adsorption of NR by GC-3 (KOH activation)						
$q_e = 42.573 * C_e^{0.377}$	0.9787	2.65 ± 0.13	42.57 ± 4.26	38.17	17.907	17.128
$\ln(q_e) = 3.337 + 0.469 * \ln(C_e)$	0.943	2.128	28.146	0.119	0.197	3.153

IV 4 Comparison between linear and non-linear Langmuir and Freundlich isotherm models for the adsorption of NR by all adsorbents

-Except for the adsorption of NR by GC-1, all coefficients of determination are very important and converge to the unit. Isotherm Models type-I and II are the most favourable according to the linear Langmuir isotherm models. The important maximum adsorption capacities were found in the case of GC-2. Adsorption energies are ranking between [0.0002-0.04] L/mg, the highest value it concerns the case of GC-3.

-Non-linear Langmuir isotherm model related to the elimination of NR by GC-1 has the lowest coefficient of determination and highest errors functions values, contrary to the other non-linear models. The maximum adsorption capacity of NR by GC-2 and GC-3 reached 470.6 and 410.4 mg/g respectively.

-Concerning the linear Freundlich isotherm models, the coefficients of determination which are higher than 0.9 are obtained for the cases GC-1 and GC-3, and the *APE* is around 3 %. The only favourable isotherm model concerns the case of GC-3, for the 2 others, they are moderate. The lowest adsorption energy is obtained from the adsorption isotherm of NR by GC-1.

-Regarding the non-linear Freundlich isotherm models, the coefficients of determination were found acceptable and the highest values which tend to the unit concern the adsorption of NR onto GC-2 and GC-3. Value of the adsorption intensity obtained from the adsorption isotherm model applied for the elimination of NR by GC-1 is less than 1, it means that the adsorption capacity is low, in parallel the adsorption energy is low also, the other models are favourable and their adsorption energies are good.

V. CONCLUSION AND FUTURE SCOPE

- A kind of perfect interaction and capture occurred between the acid dye and the grapes cores based activated carbon GC-3 case (base activation), 98% of NR was

adsorbed. In the other hand 90.5 % was removed in case of GC-2 (acid activation) and 88.31 % in case of GC-1(salt activation), the effect of salt is to remove dust and impurities and moisture so this kind of activation will ameliorate crystalline structure and will create capillaries.

- The activation of grapes cores-based activated carbons by H_3PO_4 or by KOH presents good results in the non-linear and linear Langmuir isotherm models. The linear Langmuir isotherm model type-II applied for the adsorption of NR by GC-3, shows the best correlation between the experimental and calculated data, the maximum adsorption capacity is found equal to 336.951 mg/g and the adsorption energy is about 0.0395 L/mg.

- Linear Freundlich isotherm models for the adsorption of NR by GC-2 or by GC-1 are moderate because the adsorption intensities are less than 1, in the other hand, the adsorption of the same dye by GC-3 is good.

- The linear and non-linear Freundlich also the non-linear Langmuir isotherm models for the adsorption of NR by GC-1 presents the lowest maximum adsorption capacity. The highest maximum adsorption capacity obtained from the linear and non-linear Freundlich isotherm model is related to GC-3 and it is equal to 28.146 and 42.57 mg/g respectively. GC-3 adsorbs also well as indicated by the linear and non-linear Langmuir and Freundlich isotherm models.

- By comparison between the grapes cores-based activated carbons ($CaCl_2$ activation) and those activated by H_3PO_4 or KOH, these 2 last adsorbents present good results especially in the matter of dye uptakes and maximum adsorption capacities, therefore the pyrolysis of grapes cores and chemical activation will ameliorate much better the adsorption capacity, so it has an impact on the adsorption process by creating free adsorption sites.

-Therefore, Grapes cores-based activated carbons chemically modified with base KOH or acid H_3PO_4 can be used for the capture of azo anion acid dyes such as Nylosan Red, option to use base activation of grapes cores activated carbon to adsorb acid will be very appropriate and recommended, this is an in house process done in the laboratory which can be projected at the industry level, and contribute in the protection of the environment in a matter of waste management and recovery, and produced water treatment especially those issued from the textile industry.

Future scope of improvement is to compare the adsorption of Nylosan Red by the same adsorbent without chemical activation and study and compare the adsorption process by using an industrial adsorbent.

ACKNOWLEDGMENT

Authors would like to express thank to the financial support from National CNEPRU project (Mostaganem University) E02220070003-01/01/2010 of the Ministry of Higher Education and Scientific Research-Algeria

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