

Synthesis activated carbon from tires and its application for the elimination of phenol

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ABSTRACT/RESUME

Abstract: This work is part of the recovery of a synthetic waste (tire) by preparing a highly carbonated activated carbon. The effectiveness of this activated carbon was been examined in the adsorption of phenol in the aqueous phase at room temperature with constant stirring. A comparative study was been made on a commercial activated carbon (CAC).

The prepared activated carbon (CAP) has undergone a modification by chemical activation for the following conditions (ratio of activating agent 2 ml / g of activated carbon, activation temperature of 550 ° C., activation time of 1 hour and diameter 300 µm particles). The material was characterized by measuring the point of zero charge (pHpzc), specific surface, DRX, SEM and some parameters which govern the kinetics of adsorption (humidity rate, ash rate, pH and apparent density). The results of the adsorption of phenol on the two synthetic and commercial active carbon show that the adsorbed amount obtained for the two types of carbon is almost identical. Kinetic model allowed us to show that the pseudo second order model is the most representative. The adsorption isotherms are of type L and they are well adjusted by the Freundlich model with correlation coefficients of 90%.

I. Introduction

Industrial effluents from different activities often have a significant pollutant load that is difficult to biodegrade. Among these, we note the phenols, which are organic compounds of great environmental interest; their determination has increased in recent years due to their toxicity. These phenolic compounds are often obtained from various manufacturing processes such as pharmaceuticals, petroleum refineries, coke factory... etc.

Because of this threat to the environment, much work has been carrying out. Conventional methods for the removal of phenolic pollutants in aqueous solutions can be divide into three broad categories: chemical, physical and biological [1]. The techniques most often used are solvent extraction, filtration, adsorption, precipitation, coagulation, ion exchange, destructive techniques such as ozonation and chemical oxidation [2-4]. Adsorption is one of

the widely used techniques for removing pollutants from gas and liquid phases using activated carbons.

The use of the latter has remained for a long time and until today the most effective adsorbent thanks to its high absorption capacity, mainly due to its large specific surface. This therefore encouraged researchers to direct their work towards inexpensive and available materials [5,6] such as waste olive pomace, cocoa shells and date stones, etc. [7].

This work presented in this manuscript consists of the application of our activated carbon prepared from synthetic waste and more particularly the tire for the elimination of an organic pollutant, phenol.

II. Methods and materials

II.1 Preparation of activated carbon

For the synthesis of activated carbon, we followed an experimental protocol based on recent bibliographic research [8]

The base material (tire) was been washed several times with distilled water and then dried in an oven

at 105 ° C for 24 hours, then cut, ground and sieved to retain only the fraction of 300 µm. This retained fraction underwent a pretreatment before being carbonized at 550 ° C.

The pretreatment consists of introducing the material of the selected fraction with the activating agent for H₃PO₄ (concentrated phosphoric acid) in an impregnation ratio of 2.91g / g [8.9].

The reaction mixture is continued and maintained at 85 ° C. for a contact time of 2 hours under reflux. Once activated, it is stored away from air in tightly closed bottles until charring tests.

Carbonization is been carried out in a programmable tubular furnace at 550 ° C for one hour under an inert gas to obtain a dry residue. The latter is wash with distilled water under reflux until the rinsing water is neutralized by checking the pH regularly. This protocol, used by Anundo Polania [10], made it possible to clean the microporosity of activated carbon based on coconut. The charcoal thus treated is dry in an oven at 105 ° C for 48 hours.

II.2 Characterization technics

II.2.1 Physic-chemical characterization of prepared activated carbon

II.2.1.1 Determination of the point of zero charge: The pH_{pzc} or pH of the point of zero or zero charge corresponds to the pH value for which the net charge of the adsorbent surface is zero. This parameter is very important in adsorption phenomena, especially when electrostatic forces are involved in the mechanisms.

A quick and easy way to determine the pH_{pzc} is to place 50 ml of Na₂CO₃ (0.1mole / l) in closed flasks, the pH is adjusted from 2 to 12 by adding NaOH or HCl solution (0.1M). 0.05g of activated carbon is be then added to each flask. The suspensions must be kept stirring, at room temperature, for 24 h, and the final pH is then determined. We draw on a graph ΔpH as a function of the initial pH where ΔpH = (pH_f-pH_i), the intersection of the curve with the axis which passes through zero gives the isoelectric point [11].

II.2.1.2 / Specific surface area by the BET method

The BET method requires pre-treatment of the samples (degassing and dehydration between 150 ° C and 300 ° C) in order to evacuate all the gases previously adsorbed.

The device used is of the ASAP 2010 micrometrics type. It is useful to indicate that by this method; only the external specific surface area accessible to the molecules of the adsorbate is taking into account. The nitrogen adsorption isotherm (at -195

° C) provides access to the SBET specific surface area (m² / g) of the materials studied.

The specific surface can be calculate directly from the isotherm of linearized BET in the application area where P / P₀ is between 0.05 and 0.3.

Using experimental data from the adsorption isotherm and the Brunauer-Emmett-Teller equation. (BET) are used to determine the volume of gas required to form a monomolecular layer on the sample surface.

The adsorbed quantity corresponding to a monomolecular layer when the coverage is total makes it possible to determine the specific surface (m² / g) [12].

II.2.1.3 X-ray diffraction spectrum (XRD)

X-ray diffraction is a technique that determines the structure of complex natural substances. It is also a means of qualitatively identifying compound [13]. X radiations have the property of passing through a material and being diffracted by atoms. The technique makes it possible to determine interatomic distances and the arrangement of atoms in crystal lattices. As the X-rays are diffracted in a different way by the elements of the lattice according to the construction of these, the irradiation of the material by X-rays makes it possible to know its crystallographic nature. The degree (2 thetas) of diffraction depends on the energy of the incident radiation and the spatial distribution of the atoms (crystal structure).

II.2.1.4 Scanning electron microscopy (SEM)

Scanning microscopy is a technique for the local textural and chemical characterization of materials based on the interaction of these materials with a beam of focused electrons, with an energy between 0.5 and 35kV. The different radiations emitted under the impact of the electron beam (secondary electrons, backscattered electrons and X-rays) are used to form images expressing different properties of the material (topography, heterogeneity of composition and local elemental composition) [14].

II.2.1.5 Determining the humidity level

The humidity level is a ratio expressed as a percentage and is determined by the drying of the adsorbent. A mass of adsorbent is placed in a crucible and weighed. It is then dry in an oven at 105 ° C. The sample is weighed every 5 hours. The weighing operation is repeated until the weight of the sample becomes constant. On leaving the oven, it is cool in a desiccator and then reweighed [15].

The weight of the sample before and after heating is P_i and P_f, respectively. The moisture content is calculated by the following relation (1).

$$HL\% = \frac{P_f - P_i}{P_i} * 100 \dots \dots \dots (1)$$

II.2.1.6 Determining the rate of volatile matter

The sample is placed in a closed crucible. It is then heat in a muffle furnace at 700 ° C for 10 minutes. The initial weight of the sample P_i and the final weight P_f are using to determine the level of volatiles. The greater the weight loss, the richer the sample in volatile matter [16, 17]. The rate of volatile matter is calculate by the relation (2):

$$RVM = \frac{P_i - P_f}{P_i} * 100 \dots \dots \dots (2)$$

II.2.1.7 Determination of the fixed carbon rate

The fixed carbon rate is determined by difference:

$$100\% - \sum(HL + RVM + RC)$$

Where: HL: humidity level
 RVM: Volatile matter rate
 RC: Ash rate

II.2.1.8 Apparent density

A quantity of the carbon (m) is introduced into a graduated cylinder and tamped for one to two minutes to compact the activated carbon. The precise volume (V) occupied by the coal is determined [17]. The apparent density is giving by the following formula (3):

$$\rho(\text{app}) = m/v \dots \dots \dots (3)$$

II.2.1.9 The real density

A quantity of the charcoal is placed in an oven at 105 ° C. for 24 hours. A mass (m) of this carbon is introduce into a pycnometer. The volume is then complete with a known volume (V) of methanol. The real density is calculated as follows (4):

$$\rho(\text{real}) = m/v \dots \dots \dots (4)$$

Where: v = v pycnometer –v methanol

II.3 Application to adsorption

II.3.1 Adsorption kinetics

The adsorption kinetics are carrying out according to the same experimental protocol. Determination of the time corresponding to equilibrium allowed

the establishment of adsorption isotherms for the adsorbent.

The study of the contact effect is carried out for a mass of adsorbent of 0.05 g, at a pH equal to 6, a phenol concentration which is equal to 50 mg / l, at a temperature of 25 ° C and with stirring of 200 rev. / min. Samples are taken at different time intervals of (5, 10, 20, 30, 60, 240,360)

Knowledge of this time is essential for the calculation of the maximum adsorption capacity, which is giving by equation (5).

$$Q_e = \frac{(C_0 - C_e)}{m} V \dots \dots \dots (5)$$

Where: Q_e is the quantity adsorbed at time t (mg / g), C_0 and C_e the initial concentrations and at time t respectively in pollutant (mg / l), V is the volume of the solution (ml) and m the mass of the adsorbent in g.

The determination of the rate of elimination of phenol, noted R expressed in (%) is given by equation (6):

$$R = \frac{(C_0 - C_t)}{C_0} * 100 \dots \dots \dots (6)$$

Where: C_0 and C_t , the initial concentrations and at time t respectively

The effect of some parameters affecting adsorption has been studied namely influence of the effect of the initial mass of the adsorbent varying from (0.02, 0.03, 0.06, 0.09, 0.12 and 0.15), the effect of the concentration initial (25.50 and 100) and finally the effect of pH (2, 4, 6, 8, 10 and 12).

II. Results and discussions

III.1 Physico-chemical characterization of activated carbon

Knowledge of the physicochemical and structural properties of a material is necessary to understand many phenomena such as adsorption. The most important characteristics as well as the results of the removal of phenol on prepared activated carbon and commercial activated carbon are showing in Table No. 1.

Table 1. Different physicochemical characteristics of prepared activated carbon (CAP) and (CAC)

Activated carbon	Rate of humidity (%)	Real density (g/cm ³)	Apparent volumetric mass (g/cm ³)	Ash rate (%)	Total pore volume (cm ³ /g) $V_{PT} = 1/\rho_{app} - 1/\rho_{real}$
CAP	4.70	0.82	0.546	3.35	0.605
CAC	5.65	0.98	0.514	2.98	0.925

The values obtained for humidity levels below 5% and those for ash levels below 10% show that they

are within the standards and can tell us about the quality of our activated carbons.

III.1.1 Point of zero charge

Understanding the mechanism can be explained by determining the pH_{pzc} of the adsorbent as well as its acid-base character depending on the pH of the solution. Thus for the pH = 6 (pH of the medium), the CAs whose pH_{pzc} > 6 will have the positively charged surface (pH < pH_{pzc}) while the CAs whose pH_{pzc} < 6 will have a negatively charged surface (pH > pH_{pzc}) and consequently these values inform us if the adsorption is favored or not.

In our case the pH_{pzc} of the CAP is 6.2 (pH < pH_{pzc}) the surface of the carbon being positively charged and the substance to be removed being negatively charged, there is therefore an attraction, therefore an increase in the absorption capacity. For CAC the pH_{pzc} is 8.36, (pH > pH_{pzc}), the surface is negatively charged, the adsorption of negatively charged substances is unfavorable. The representation of the point of zero charge ΔpH = f (pHi) for the two activated carbons is illustrated in figure N° 1.

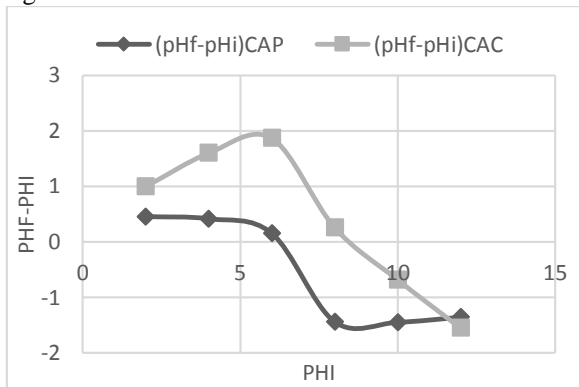


Fig 1. Point of zero charge $\Delta pH = f(pHi)$ of CAP and CAC

III.1.2 Analysis by scanning electron microscopy (SEM)

The images (figure 2) represent the images produced by scanning electron microscopy for the surfaces of CAP and CAC carbon (Merck)

The SEM images obtained at 50 μm (for the CAC) and 10 μm (for the CAP) show that the surface of the latter is well developed. A better development characterizes the commercial activated carbon its structure shows a more homogeneous and more developed porous structure in comparison with the prepared activated carbon CAP which presents irregularities on its surface and which reveals a uniform porosity

III.1.3 X-ray diffraction spectrum of prepared activated carbon (CAP).

The DRX profile shows an amorphous structure of the prepared activated carbon (Figure 3)

The figure below (3) shows an amorphous structure of activated carbon. The XRD profile of the latter shows a diffraction peak $2\theta = 27^\circ$ which is attributed to the presence of the carbon / graphite corresponding to the graphite structure of the highly organized a layer of graphite [18].

This result can be explain by the fact that the carbonization reaction of organic compounds consists of breaking chemical bonds with temperature and further condensing into active compounds. These compounds form layers of typical graphitic planes during carbonization.

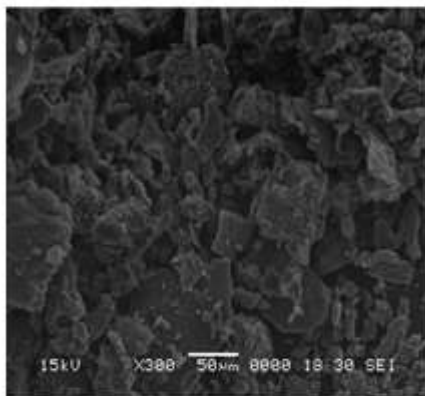
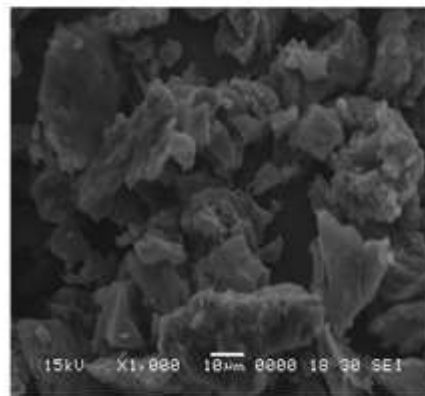


Fig 2. SEM of CAC (50 μm)



SEM of CAP (10 μm)

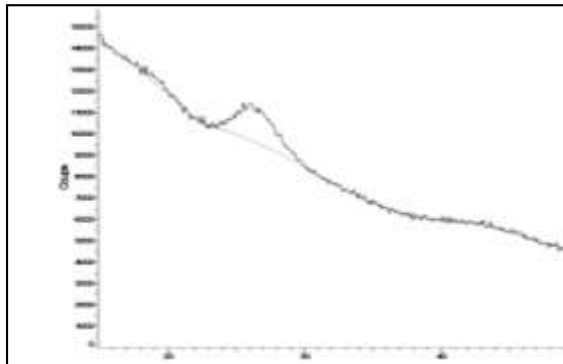


Fig 3. DRX Spectrum of CAP

III.4 Results of adsorption kinetics

III.4.1 Adsorption kinetics

The tests of the kinetics of the elimination of phenol by adsorption on prepared activated carbon (PAC) were carrying out at room temperature. The objective is to determine the adsorbent pseudo-adsorbate equilibrium time. This study found that about 60 minutes of stirring is sufficient to achieve equilibrium. The curve $Q = f(t)$ of phenol on CAP and CAC is shown in Figure 4.

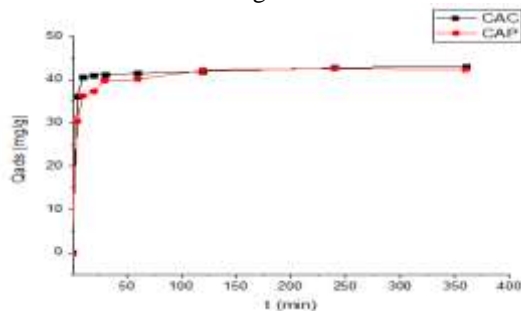


Fig 4. Adsorption kinetics $Q=f(t)$ of phenol of CAP and CAC ($m=0.05$, $V=50ml$, $pH=6$, $C=50mg/l$, $T=25^{\circ}C$)

The plotted curves show that equilibrium is quickly reached for the two activated carbons (CAP, CAC). The adsorption is very fast at the start of the contact between the adsorbate and the adsorbent this is due to the existence of a large number of free sites, which explains the steep slope of the curve. The activated carbons show good performance with an increase in the quantity adsorbed until saturation is obtained corresponding to a time of 60 min is of the order of 41, 411 mg / g and 40, 176 mg / g with elimination yields corresponding to 82.823 % and 80.117% respectively for CAC and CAP.

III.4.2 Effect of the initial concentration on the adsorption of phenol on activated carbon

The initial concentration effect is carrying out at different concentrations of phenol (25, 50, 100 mg /

l). The mass of adsorbent $m_{opt} = 0.05g$, $t_{eq} = 60min$

3, $pH = 6$ at room temperature.

According to the results obtained, we can see that the phenol adsorption capacity for CAC and CAP adsorbents increases with increasing initial concentration of phenol.

The increase in the concentration causes the increase in the driving force of the concentration gradient, therefore the increase in the molecular diffusion of phenol in solution in the surface of the adsorbent (figure 5).

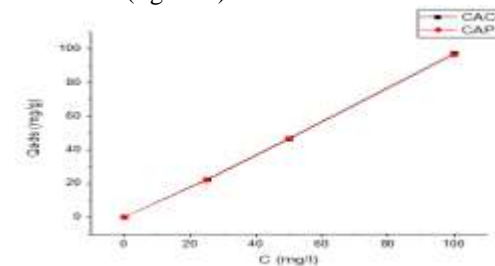


Fig 5. Effect of the initial concentration of phenol on the adsorption capacity of activated carbon (CAC, CAP), (Contact time=1h, $pH=6$, $m=0.05g$, $V=50ml$, $T=25^{\circ}C$)

III.4.3 Effect of the mass of the adsorbent on the adsorption capacity

The initial mass effect is carried out with different masses of activated carbon varying between 0.02 and 0.15g with a phenol concentration of 50mg / l, contact time of 60 minutes, $pH = 6$ and at a temperature of $25^{\circ}C$. The results obtained are showing in Figure 6.

The phenol elimination capacity is giving in the form of a yield curve, which is around 96.35% for CAC and 96.74% for CAP for a mass of 0.15g. This yield is all the more important when the quantity of the adsorbent is high, this can be explained by the availability of the number of active sites on the surface of the material.

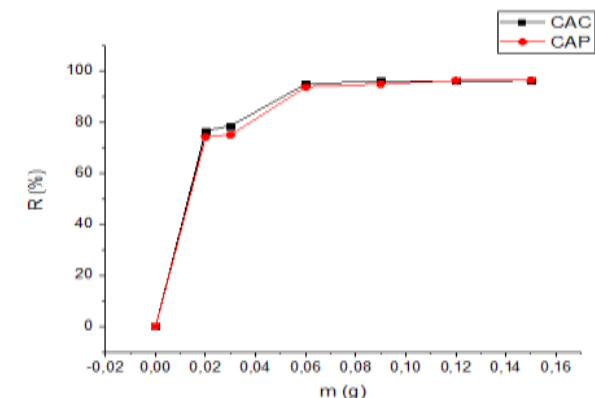


Fig 6. Phenol elimination yield on CAP and CAC, (Contact time 1h, $pH=6$, $V=50ml$, $C=50mg/l$, $T=25^{\circ}C$)

III.4.4 Effect of the initial pH of the solution

Masses equal to 0.05 g of adsorbent were placed in solutions of volume equal to 50 ml containing the adsorbate at a dose of 50 mg / l. The pH of the solutions was adjusting to values of 2, 4, 6, 8, 10, and 12. In addition, stirred for 1 hour at a temperature of 25 ° C. The results of the variation of the phenol adsorption rate as a function of pH are showing graphically in figure 7.

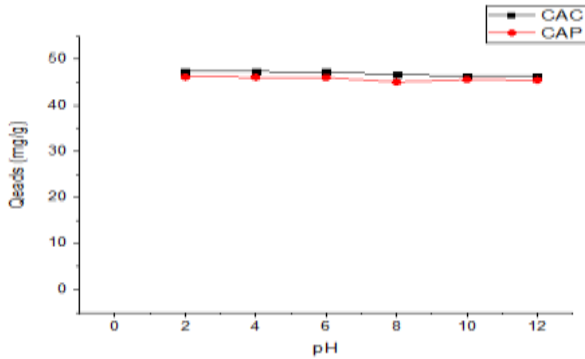


Fig 7. Absorption capacity of phenol on activated carbon (CAP, CAC), (Contact time 1h, V=50ml, C=50ml/g, T=25°C)

III.4.5 modeling of adsorption kinetics

III.4.5.1 Model of pseudo first order kinetics

The relation can describe this system:

$$\frac{dq_t}{dt} = K_1(q_e - q_t) \dots \dots \dots (7)$$

Where: q_e : amount adsorbed at equilibrium (mg / g)
 q_t : quantity adsorbed at time t (mg / g)
 k_1 : Lagergren speed constant

The integration of equation (1) gives the linear form of Lagergren. The plot of the curve $\ln(q_e - q_t) = f(t)$

makes it possible to determine k_1 and the quantity adsorbed at equilibrium q_e .

The various parameters have been grouped together in table 2 and the curves obtained are represented in figure 8 (a, b).

III.4.5.2 Pseudo-second order kinetics model

This model is express according to equation (8):

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \dots \dots \dots (8)$$

Integrating this equation gives us the linear form equation (9)

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} \dots \dots \dots (9)$$

Where: Adsorption rate constant for the pseudo-second order (g.mol⁻¹.min⁻¹).

q_e : amount of adsorbate at equilibrium per gram of adsorbent (mg / g).

The pseudo-second order equation is often used successfully to describe the kinetics of the reaction of pollutants binding to the adsorbent. This model gives a better description of the kinetics of the adsorption reaction compared to the pseudo-first order model.

All the kinetic parameters of the two activated carbons (CAC and CAP) determined from these straight lines are collated in Table 2. The latter indicates that the values of the correlation coefficient (R²) for the second order model is clearly higher than the first order model.

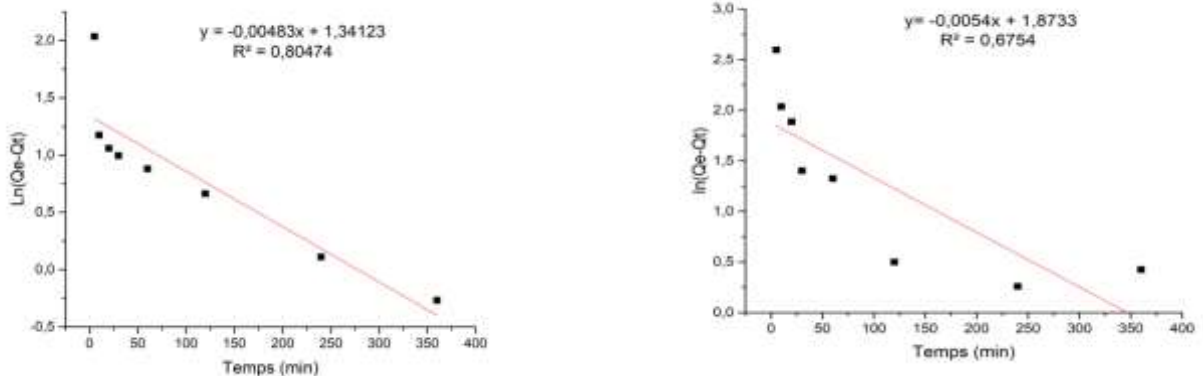


Fig 8. Pseudo first order kinetics (a): CAC and (b): CAP

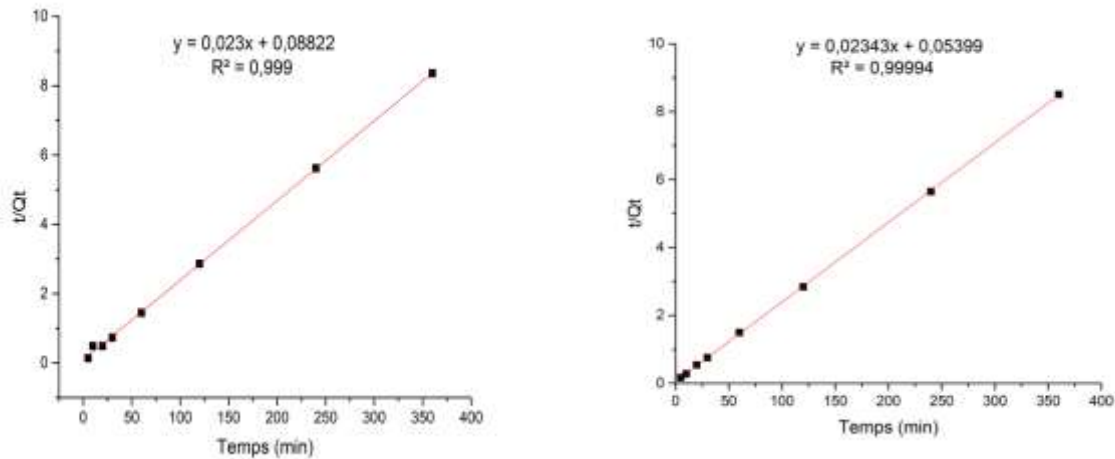


Fig 9. Pseudo second order kinetics (a): CAC, (b): CAP

Table 2. Constants and coefficients of first and second order correlations

Concentration mg/l	Activated carbon	Pseudo-order 1			Pseudo-ordre 2		
		Qe (mg/g)	K ₁ (L . mn ⁻¹)	R ²	Qe (mg/g)	K ₂ (L . mn ⁻¹)	R ²
50	CAP	44,700	0,0054	0,6754	18,521	0,0234	0,999
	CAC	43,01	0,043	0,8047	11,36	0,023	0,999

We can see that the adsorption kinetics are correctly described by the pseudo-order kinetic model 2 and follow the Freundlich model which has correlation coefficients all greater than 0.99 for a concentration of 50 mg / L.

III.4.5.3 modeling of isotherms

Adsorption isotherms are often used to determine maximum pollutant binding capacities and to identify the type of adsorption. The results processed according to the mathematical models of Langmuir, Freundlich and, it are allows to calculate the maximum adsorption capacity as well as the adsorption parameters.

The adsorption isotherms were studied by stirring a mass of the adsorbent 0.50 g in solutions of the phenol of the same concentration of 50 mg / l. The adsorbent and the adsorbate were contact for 1 hour with stirring at 200 rpm. After determining the residual concentrations, we followed, on the one hand, the evolution of $\ln q_t = f(\ln C_e)$ according to the Freundlich model and on the other hand, the evolution of $C_e / q_t = f(C_e)$ according to the model of Langmuir and the evolution of q_t according to \ln . The results obtained are illustrated in figure (10).

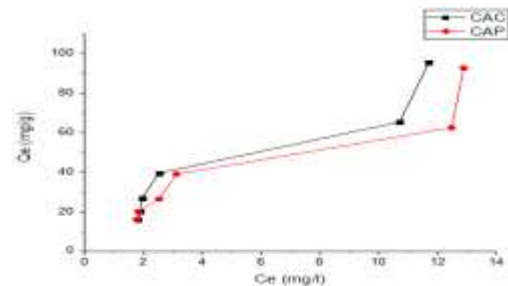


Fig 10. Adsorption isotherms $Q_e = f(C_e)$ for CAC and CAP

Examination of the two curves for the two activated carbons CAC and CAP on phenol shows that the shape of these isotherms is of type L.

The shape of the curve shows a strong increase in the pollutants on the two activated carbons depending on the concentration of phenol until it reaches a plateau, beyond the adsorption becomes constant this can be explained by the fact that from a certain quantity of phenol, the sites of the adsorbent will be saturated.

a / Freundlich isotherm

The representative curve of the Freundlich isotherm

is give in figure 11.

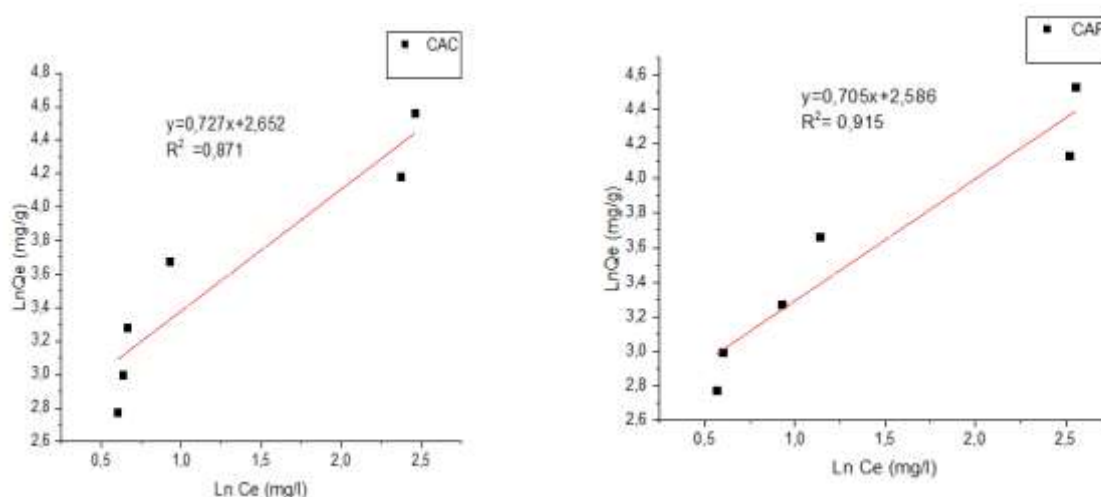


Fig 11. Isotherme de l'adsorption du phénol par l'équation de Freundlich.

The results of the correlation coefficients and absorption parameters are giving in the table below.

Table 3. Correlation coefficients and adsorption parameters deduced from the Langmuir model and the Freundlich model.

Acivated carbon	Langmuir			Freundlich		
	Q _{max}	K _L	R ²	K _F	n	R ²
CAC	333,33	0,036	0,806	14,18	0,727	0,871
CAP	250	1	0,855	13,27	0,705	0,915

According to these results, it can be concluded that Freundlich is most likely to model the adsorption of phenol on prepared activated carbon with a correlation coefficient of 90%.

VI. Conclusion

The tire is a waste, which is very responsive in nature and little exploited in several countries around the world. This waste can be recovered thanks to the implementation of an experimental activation protocol in order to obtain a high-performance activated carbon with the aim of eliminating a certain number of phenolic compounds, including phenol. Based on the results obtained, we can draw some essential points:

The chemical activity of the activated carbon from the tire is very effective
 Adsorption is clearly influence by the presence of active sites present on the surface of our activated carbon

Activated carbon prepared from the tire is a new material, which can compete with commercial activated carbon (Merck)

Finally, the adsorption kinetics are correctly described by the pseudo-order kinetic model 2 and follow the Freundlich model with a correlation coefficient all equal to 0.999 for a concentration of 50 mg / L.

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