



Kinetics, isotherm, and thermodynamic parameters of polyphenol adsorption from olive mill wastewater onto activated carbon

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Abstract: The aim of this paper is to study the kinetics, isotherm, and thermodynamic parameters of the adsorption of polyphenol from olive mill wastewater (OMW) produced by the two-phase extraction process on activated carbon. To better understand this adsorption process, three different isotherm models were applied: the two-parameter equations (Langmuir and Freundlich) and the three-parameter equation (Sips). Additionally, two non-linear kinetic models, namely the pseudo-first order and pseudo-second order kinetics, were employed. The results showed that the adsorption reaction was very rapid, reaching equilibrium within just two minutes. The equilibrium data were best described by the non-linear Freundlich model, which provided the highest regression coefficient ($R^2 = 0.969$). Meanwhile, the adsorption process followed the pseudo-first order kinetic model with a strong regression coefficient ($R^2 = 0.999$). The determination of thermodynamic parameters indicated that the adsorption of phenolic compounds from OMW on activated carbon is an exothermic and spontaneous process. In conclusion, the study demonstrated that activated carbon is an effective adsorbent for the removal of phenolic compounds from olive mill wastewater. The models used accurately characterized the adsorption parameters, providing a deep understanding of the mechanisms involved. This information can be essential for developing more efficient and environmentally friendly wastewater treatment systems in the olive oil industry. This research highlights the potential of activated carbon in addressing environmental challenges posed by OMW, offering a viable solution for improving wastewater management practices in the industry.

1. Introduction

In Morocco, the olive sector has a double economic and social vocation. The national arboricultural surface is largely composed of the culture of the olive tree, making it the main fruit crop of the country. Thus, the olive industry contributes to 5% of the agricultural GDP and 15% of agri-food exports. Since the launch of the Green Morocco Plan in 2008, olive growing has undergone a major expansion with a significant increase in the area devoted to olive trees, which rose from 763 000 ha in 2007/08 to 933 475 ha in 2012/13. The olive sector, for its part, has also experienced a clear growth from 765 377 tons in 2007 to 1.3 million tons with a peak of 1.5 million tons in 2010 ([Moroccan Ministry of Agriculture and Maritime Fishing, 2013](#)).

However, the olive industry is responsible for huge environmental problems. The pollution generated by these liquid discharges commonly called "Olive mill wastewater" (OMW) exceeds 30 million m³ per year (Slimani Alaoui N *et al.*, 2015, Bouknana *et al.*, 2014 & 2021). These effluents (OMW) are very rich in organic matter (phenolic compounds, lipids ...), and often spread in nature, in an uncontrolled manner, on agricultural soils or sometimes stored temporarily in tanks, exposing the water-soil-plant systems, has an unavoidable pollution

Various purification treatments are applied to them: biological, physical and chemical. Costly and still insufficient, these treatments are all aimed at reducing their impact on the environment (Arda *et al.*, 2010, Bouknana *et al.*, 2019).

The problem of evacuation of OMW has been approached in various ways by the different Mediterranean olive growing countries that have adopted specific solutions to solve this problem. Some countries have used storage and evaporation basins that have not been as successful as expected because of the problems of air pollution, slow evaporation time and infiltration of the OMW into the water table in the absence of sealing. Physical, chemical and biological treatments were also tested in order to reduce their polluting load and to minimize their high toxicity for the microflora. The biodegradability of these OMW makes the biological treatment difficult to apply and their elimination requires the use of chemical products and very expensive equipment, in addition to the waste they generate.

In order to remove phenolic compounds from vegetation water (OMW), several studies have been carried out using different methods namely: filtration on clay column and sawdust of eucalyptus wood (Ouabou *et al.*, 2014), the physicochemical process coupling ultrafiltration and a bipolar reactor (Yahyaoui *et al.*, 2013), coagulation flocculation and electrochemical way (Sbai *et al.*, 2015), the physicochemical treatment using lime and ferric chloride (Slimani Alaoui *et al.*, 2015), the physicochemical treatment of these effluents by synthesized potassium permanganoferrates of chemical formula K₃FexMnyO₈ (Zahari *et al.*, 2014).

Adsorption remains the simplest and most widely used technique for the removal of organic pollutants that are difficult to biodegrade, as well as for the decoloration of water (Meroufe *et al.*, 2015). The most commonly used and cost-effective adsorbent for the treatment of OMW is the activated carbon. The latter has demonstrated great efficiency in the decoloration and treatment of OMW while reducing their organic load. This is due to its amorphous carbon structure, which is endowed with a highly developed porosity. It can be obtained from a large number of materials containing carbon of vegetable, animal or mineral origin (Ouedrhiri *et al.*, 2018).

In the present work, the adsorption of polyphenols from OMW was investigated by studying the kinetics, isotherms, and thermodynamic parameters of the process. Additionally, various adsorption isotherm and kinetic models were applied to the experimental data.

2. Methodology

2.1 Origin of the OMW used

The OMW used were recovered from a modern oil mill equipped with a continuous two-phase extraction system located in the ben Karrich region (Province of Tetouan-North Morocco).

2.2 Adsorbent used

The adsorbent medium used in this work is a commercially available powdered activated carbon. its features are shown in **Table 1**. below.

Table 1. Characteristics of activated carbon used.

Methylene blue adsorption (0,15%) min	100 ml / g
Acidity and/or alkalinity test	According to tests
Substances soluble in C₂H₅OH 0,2%
Substances soluble in H₂O	0,2%
Substances soluble in H₂O	1%
Loss on drying at 120 ° C	10%
Calcination residue	1%
Chloride (Cl)	0,01 %
Sulfate (SO₄)	0,01%
Sulfur(S)	According to tests
Organic substances	According to tests
Ca	0,05%
Cu	0,002%
Fe	0,025%
Ni	0,002%
Pb	0,002%

2.3 Adsorption study

The adsorption of OMW on activated carbon was performed under standard conditions, at room temperature and pressure in an orbital agitator (IKA KS 4000 i control), controlling temperature and agitation. In a series of 250 ml glass bottles, 100 ml of the OMW diluted to 10% was added. The parameters studied were: contact time, solid/liquid ratio, pH, stirring speed and temperature. The solution consisting of the OMW added with a certain mass of activated carbon was stirred permanently.

The adsorption capacity is estimated using the following equation (**Eqn. 1**) (Liu *et al.*, 2012):

$$q = \frac{(C_0 - C) \cdot V}{m} \quad \text{Eqn. 1}$$

where q : the adsorbed quantity in mg/g; C_0 : the initial concentration of phenolic compounds in mg/l; V : the volume of the solution in ml; and m : the mass of activated carbon in g.

Absorbance measurements were performed by the RAYLEIGH UV-1800 spectrophotometer and the Solver XLAM tool did modeling of the experimental data.

2.4 Adsorption kinetics

Adsorption kinetics is a very important parameter that indicates the purification performance of an adsorbent. It also allows to estimate the quantity of pollutants adsorbed as a function of time. In order to study the adsorption kinetics of phenolic compounds from OMW on activated carbon, we added 4 g of activated carbon to 100 ml of OMW diluted at 10%, at room temperature and at the natural pH of

OMW (without adjustment), during a contact time of 30 minutes. Adsorption kinetics can be modeled by applying several models. In this work the pseudo-first order and pseudo-second order model were used as two kinetics models for determine the nature and kinetics order of phenolic compounds adsorption onto activated carbon.

2.4.1 Pseudo first-order kinetic model

The equation for the pseudo-first order (PFO) model is represented as follows (Malash *et al.*, 2012):

$$q = q_e \cdot e^{(-k_1 \cdot t)} \quad \text{Eqn. 2}$$

with q : the quantity adsorbed at a time t in mg/g; q_e : the quantity of polyphenols adsorbed at equilibrium in mg/g and k_1 : the kinetic constant of the adsorption reaction for the pseudo first order in min^{-1} .

2.4.2 Pseudo-second order kinetic model

The pseudo-second order (PSO) equation is often successfully used to describe the kinetics of the adsorbate binding reaction on the adsorbent (Ho *et al.*, 1999). This model is written as follows (Han *et al.*, 2009):

$$q = \frac{K_2 \cdot q_e \cdot t}{1 + (K_2 \cdot q_e \cdot t)} \quad \text{Eqn. 3}$$

with q : the amount adsorbed at a time t in mg/g; q_e : the amount of polyphenols adsorbed at equilibrium in mg/g and k_2 : the kinetic constant of the adsorption reaction for the pseudo second order in min^{-1} .

2.5 Adsorption isotherm

The behavior of an adsorbent towards an adsorbate can be explained by its adsorption isotherm. The curve of the isotherm represents the adsorbed quantity and the concentration of the adsorbate in solution, at equilibrium and at constant temperature. Adsorption isotherms are established by contacting 50 ml of the 10% diluted OMW with activated carbon masses varying between 0.025 and 2.500 g for a contact time of 30 min, at a temperature of 20°C, at pH= 2.0 and at a stirring speed of 300 rpm. To quantify the adsorption capacity of phenolic compounds on activated carbon, Langmuir, Freundlich and SIPS models were used.

2.5.1 Langmuir model

The Langmuir model is characterized by the fact that adsorption occurs in a single layer, in energetically equivalent adsorption sites. These sites can contain only one molecule per site, there are no interactions between the adsorbed molecules. The isotherm can be modeled by the following equation (Langmuir *et al.*, 1918):

$$q = \frac{b \cdot q_0 \cdot C_e}{1 + (b \cdot C_e)} \quad \text{Eqn. 4}$$

where q is the amount of polyphenols adsorbed per unit mass of charcoal in mg/g; q_0 is the maximum adsorption capacity in mg/g; b is the adsorbate-adsorbent equilibrium constant and C_e is the equilibrium adsorbate concentration in mg/l.

2.5.2 Freundlich model

The Freundlich isotherm, an empirical model, describes a multilayer heterogeneous adsorbent surface with different adsorption sites (Vázquez *et al.*, 2007). The non-linear form of the Freundlich model is expressed by the following equation (Freundlich *et al.*, 1932):

$$q = K_f \cdot C_e^{1/n} \quad \text{Eqn. 5}$$

where q is the amount of polyphenols adsorbed per unit mass of charcoal in mg/g; C_e is the equilibrium concentration of the adsorbate in mg/l and K_f and n are Freundlich constants.

2.5.3 Sips model

The Sips isotherm is a combination of Langmuir and Freundlich isotherms. It is defined by the following general expression (Sips *et al.*, 1949):

$$q_e = \frac{q_m \cdot K_S \cdot C_e^{\frac{1}{n}}}{1 + K_S \cdot C_e^{\frac{1}{n}}} \quad \text{Eqn. 6}$$

where C_e is the equilibrium concentration of the adsorbate in mg/l; q_m and K_S are respectively the maximum adsorption capacity of Sips in mg/g and the equilibrium constant of Sips in mg⁻¹ and $1/n$ is the exponent of the Sips model (it is between 0 and 1: If $1/n$ is close to unity, the Sips isotherm resembles the Langmuir isotherm predicting a homogeneous adsorption surface. If $1/n$ deviates from unity, in this case, the model resembles the Freundlich model and it describes the heterogeneity of the adsorbent surface (Nanta *et al.*, 2018).

2.6 Thermodynamic of adsorption

The effect of temperature on the phenomenon of adsorption was studied by varying this parameter from 10 to 50°C using a thermostat bath to maintain the temperature at the desired value. The tests were carried out by stirring 4 g of activated carbon in 100 ml of the OMW solution diluted to 10% with distilled water. The parameters such as free energy (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°) can be estimated from the equilibrium constants at different temperatures. The free energy change of the adsorption reaction is given by the following equations (Hsu *et al.*, 2017):

$$\Delta G^\circ = -RT \cdot \ln K_d \quad \text{Eqn. 7}$$

$$\Delta G^\circ = \Delta H^\circ - T \cdot \Delta S^\circ \quad \text{Eqn. 8}$$

where ΔG° is the free energy change in kJ/mol; R is the universal gas constant (8.314 J.mol⁻¹. K⁻¹); T is the absolute temperature in °K; ΔH° is the standard enthalpy in kJ.mol⁻¹; ΔS° is the standard entropy in J.mol⁻¹.K⁻¹ and K_d is the equilibrium constant.

The values of ΔH° and ΔS° can be calculated from the Van't Hoff equation:

$$\ln K_d = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad \text{Eqn. 9}$$

The graphical representation of $\ln K_d$ as a function of $1/T$ is a straight line with slope ($-\Delta H^\circ/R$) and intercept ($\Delta S^\circ/R$).

3. Results and Discussion

3.1 Adsorption kinetics

The kinetics of adsorption allows to evaluate the speed of retention of phenolic compounds from the liquid phase (OMW) to the solid phase (activated carbon). The experimental results of the study of this kinetics are represented in [figure 1](#).

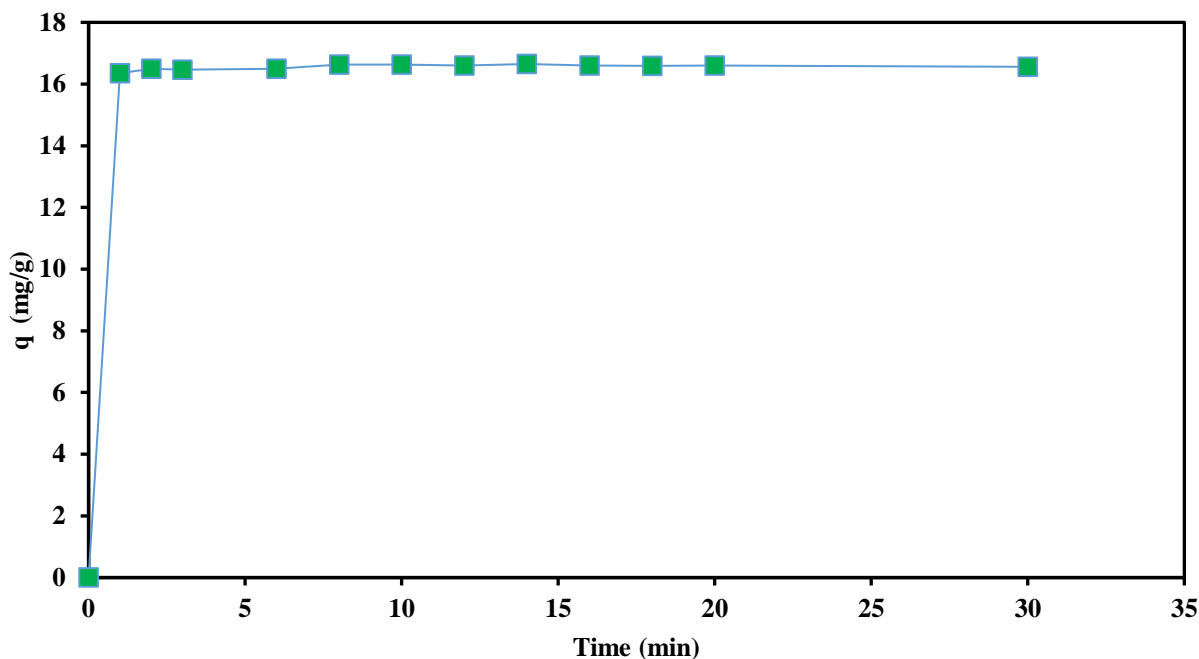


Figure 1. Adsorption kinetic of phenolic compounds on activated carbon (at $T=20^{\circ}\text{C}$, $\text{pH}=2.0$ and stirring speed of 300 rpm).

According to the results obtained, we notice that the adsorption rate of polyphenols on activated carbon is very fast so that from the first contact of the carbon with the OMW, the adsorption is triggered and the majority of phenolic compounds are adsorbed after 2 minutes. This is due to the nature of the interactions that occur between the carbon and the OMW.

3.1.1 Modelling of adsorption kinetic

Several models can be applied to exploit the results obtained during the study of the adsorption kinetic. In our case, we studied the adsorption kinetic, applying the pseudo 1st order model and the pseudo 2nd order model.

PFO model

The modelling of the experimental results of the adsorption kinetics of phenolic compounds on activated carbon by the pseudo 1st order model is shown in [Figure 2](#). The different parameters of the model as well as the regression coefficients, R^2 , are represented in [Table 2](#). By analyzing the parameters of the pseudo 1st order model, it can be seen that the regression coefficient is very good ($R^2=0.999$) and the value of the theoretical adsorbed quantity $q_{1\text{mod}}$ is very close to the experimental value $q_{1\text{exp}}$, which implies that the pseudo 1st order model can be applied for the description of the adsorption of polyphenols from OMW on activated carbon.

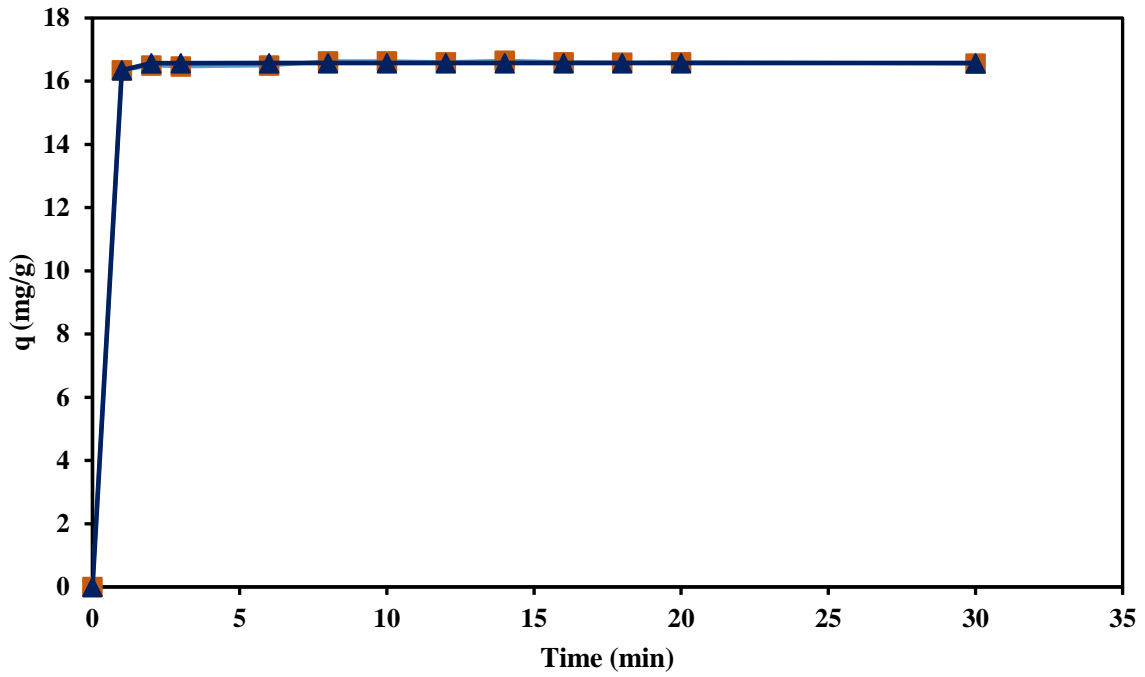


Figure 2. Modeling of adsorption kinetics by the PFO model for experimental data and modeled data

Table 2. Values of the parameters of the pseudo 1st order model.

PFO model	k_1 (min^{-1})	$q_{1 \text{ exp}}$ (mg/g)	$q_{1 \text{ mod}}$ (mg/g)	R^2
	4.28	16.55	16.57	0.999

PSO model

The modeling of the experimental results of the adsorption kinetics by the PSO model is represented on **Figure 3**.

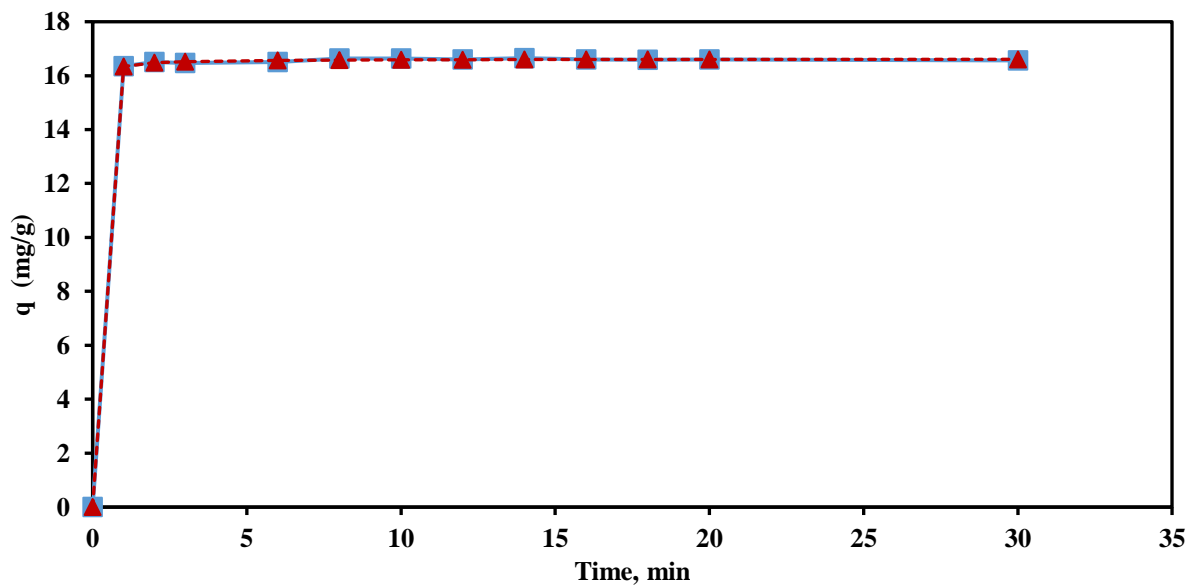


Figure 3. Modeling of the adsorption kinetics by the pseudo 2nd order model for experimental data and modeled data

The different parameters of the model as well as the regression coefficients R^2 are shown in **Table 3**.

Table 3. Parameter values of the pseudo 2nd order model.

PSO model	K_2 (min^{-1})	$q_{2 \text{ exp}}$ (mg/g)	$q_{2 \text{ mod}}$ (mg/g)	R^2
	4.28	16.55	16.57	0.999

The regression coefficient of the pseudo 2nd order model is good 0.980 and the value of adsorption capacity calculated by this model is close to the experimentally determined one, which justifies that the adsorption kinetics of polyphenols on activated carbon can be described by the pseudo 2nd order model. From the results shown in **Tables 2 and 3**, it can be concluded that the pseudo 1st order model is the closest to determine the kinetic order of adsorption of phenolic compounds from OMW on activated carbon. This model suggests that the adsorption process was controlled by physisorption.

3.2 Adsorption isotherm

The capacity of the adsorbents to adsorb the different constituents of a mixture is the most important factor determining the performance of most adsorption processes. It is therefore essential to know the adsorbate-adsorbent equilibrium properties to be able to design and dimension correctly the adsorption processes. For a given species, the adsorption isotherm expresses the quantity adsorbed per unit mass of activated carbon (q , mg/g) as a function of the concentration in the fluid phase at equilibrium (C_e , mg/l). The experimental isotherm curve plot is shown in **Figure 4**.

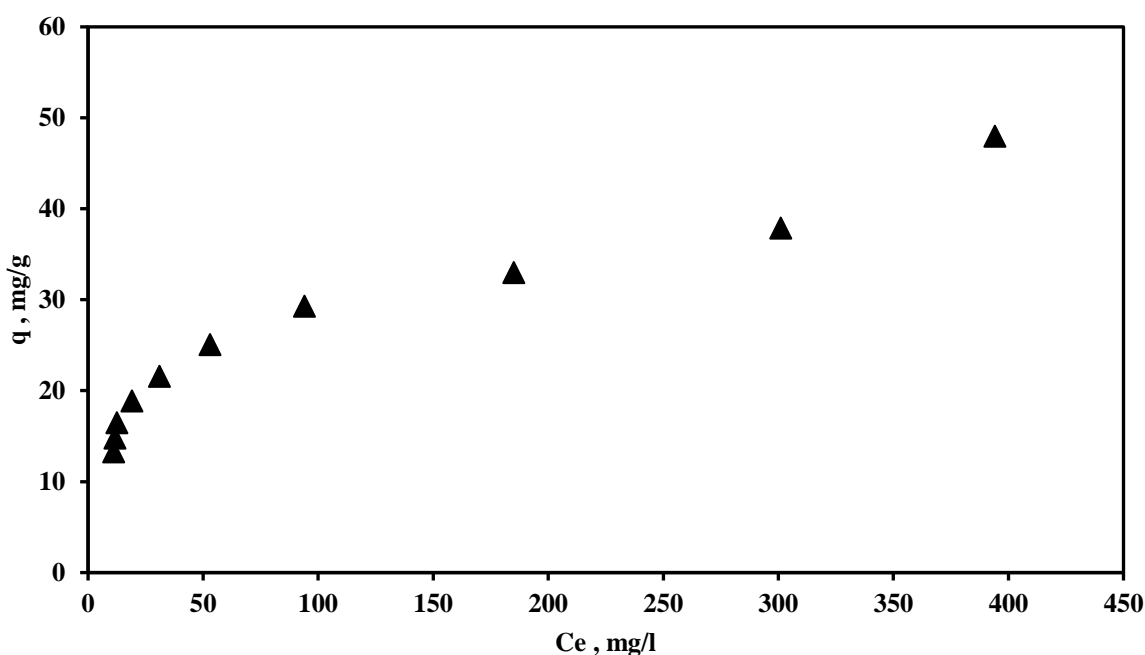


Figure 4. Isotherm of adsorption of polyphenols from OMW onto activated carbon.

According to the Brunauer isotherm classification (Beunauer *et al.*, 1644, Sing *et al.*, 1985), the adsorption isotherm of phenolic compounds by activated carbon is type II, it generally corresponds to multilayer adsorption on open surfaces.

3.2.1 Modeling of adsorption isotherm

The modeling is the mathematical description of the adsorption at the solid-liquid interface. Its objective is to find the models that accurately describe the experimental results of adsorption isotherms and to specify the parameters of each model. **Figure 5** shows our adsorption isotherms compared to the three models used (Langmuir, Freundlich and Sips).

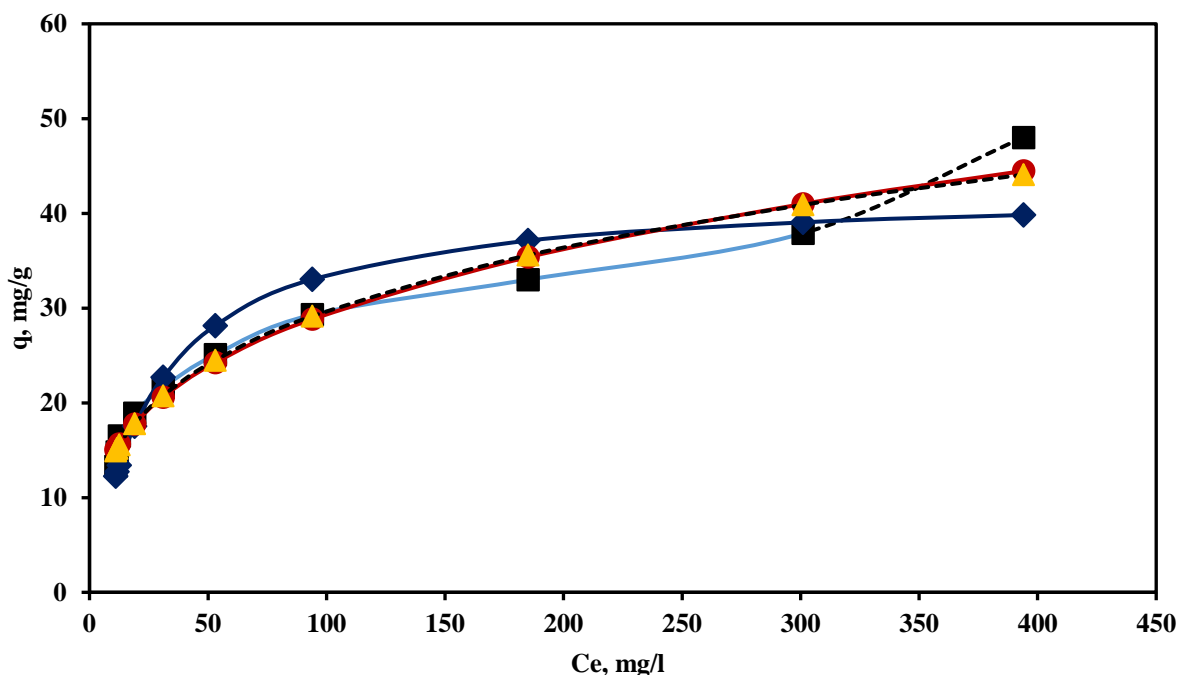


Figure 5. Adsorption isotherm of phenolic compounds on activated carbon for experimental data Langmuir data Freundlich data and sips data.

The degree of fit of the theoretical models to the experimental results is determined based on the regression coefficient R^2 . The results corresponding to the maximum adsorption capacity (q_m), the constants related to the three studied models as well as the deduced regression coefficients are presented in **Table 4**.

Table 4. Parameters of the different adsorption isotherm models

Langmuir			Freundlich			Sips			
k_l	q_m	R^2	K_f	$1/n$	R^2	q_m	K_s	$1/n$	R^2
0.036	42.57	0.888	7.28	0.3	0.969	22.5	0.031	0.34	0.967

From the results obtained (**Table 4**), it can be seen that the adsorption isotherm can be described by the Freundlich model. The value of $1/n$ of the Sips constant confirms this result, because as it has been quoted before, when $1/n$ deviates from the value of unity, the adsorption isotherm resembles the Freundlich model (Freundlich *et al.*, 1932). In this case, the adsorption occurs first in monolayer and then in multilayer. This type of isotherm is observed in the case of adsorbents with a large macroporous or mesoporous volume and this is the case of activated carbon.

3.3 Thermodynamic of adsorption

The thermodynamic parameters of the adsorption process of phenolic compounds from OMW on activated carbon are the changes in standard enthalpy, standard entropy and standard free energy. The standard entropy and standard enthalpy were obtained from graphical representation of $\ln K_d$ versus $1/T$, as shown in **Figure 6**.

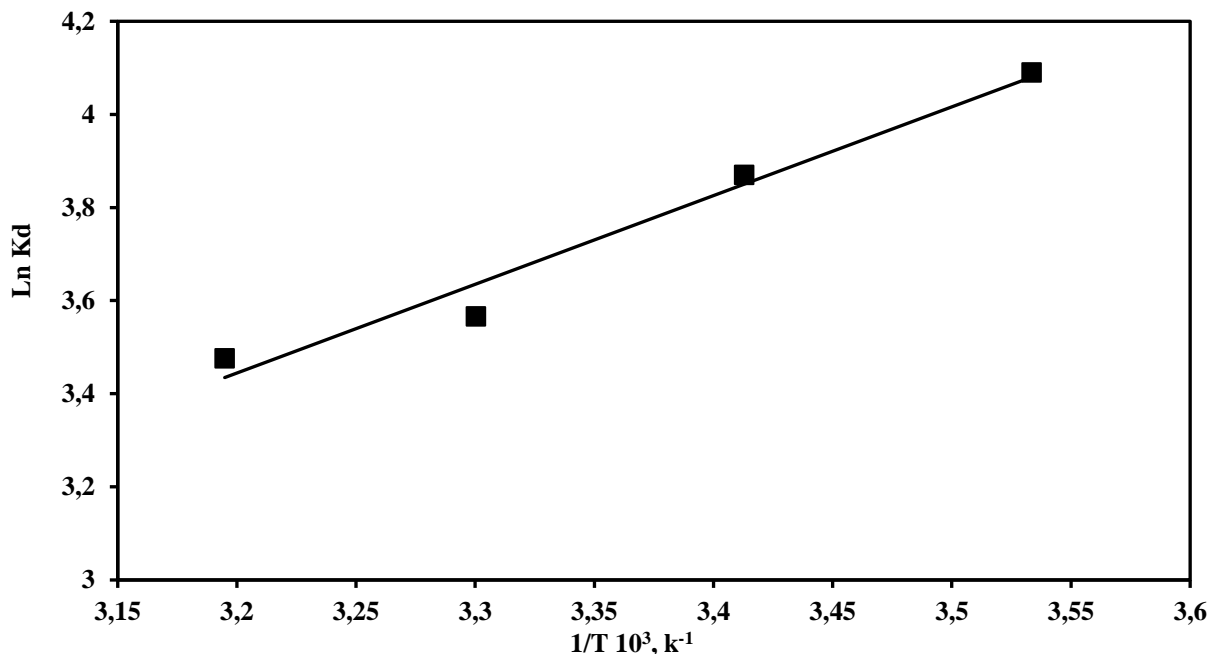


Figure 6. Variation of $\ln K_d$ as a function of temperature.

The values of these parameters were calculated using **Eqn.7, Eqn.8 and Eqn.9** at different temperatures and are shown in **Table 5**.

Table 5. Parameters of thermodynamic of adsorption

$T(^{\circ}K)$	$\Delta G^{\circ} (kJ \cdot mol^{-1})$	$\Delta S^{\circ} (J \cdot mol^{-1} \cdot K^{-1})$	$\Delta H^{\circ} (kJ \cdot mol^{-1})$
283	-140.49		
293	-116.73		
303	-89.08	-22.06	-16.21
313	-84.14		

Table 4 shows that the negative values of the three parameters ΔG° , ΔH° and ΔS° of adsorption of polyphenols on activated carbon indicate that the reaction is spontaneous and exothermic and that the order of distribution of the phenolic compounds molecules on the adsorbent is important compared to that of the solution. Furthermore, examination of the standard enthalpy values of the adsorption ($< 40 kJ \cdot mol^{-1}$) shows that it is a physisorption (Aarfane *et al.*, 2014). It is also noticed, that ΔG° increases with increasing temperature, which can be explained by the fact, that adsorption becomes difficult when the temperature becomes high (Namasiavayamer *et al.*, 1998; Deghles *et al.*, 2019;).

Conclusion

The present work demonstrated that the adsorption of polyphenols onto activated carbon is very rapid, with equilibrium being reached within 2 minutes of adsorbate-adsorbent contact. The PFO kinetic model, indicating that the process is governed by physisorption, best described the kinetic data. The experimental results were fitted to three adsorption models, with the Freundlich isotherm model providing the best fit ($R^2 = 0.969$). Thermodynamic analysis revealed that the adsorption of polyphenols from OMW onto activated carbon is an exothermic and spontaneous process.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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