Removal of Clofibric, Salicylic and Gallic Acids from Aqueous Solutions by Adsorption on a Commercial Activated Carbon

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Abstract: The adsorption behavior of three pharmaceutical compounds and widespread used drugs, namely, clofibric acid, salicylic acid and gallic acid from aqueous solutions was investigated using an activated carbon, as adsorbent. This study aims to evaluate the performance efficiency of the proposed adsorbent commercial activated carbon for eliminate these organic compounds.

The Freundlich, Langmuir, Temkin and Toth models were applied to the equilibrium data and in order to describe the adsorption behavior. It was found that the experimental data fitted well to the Langmuir model. It is also revealed that the adsorption of this compounds from the aqueous solutions on the activated carbon refer to the S-type by the Giles's classification.

In order to investigate the mechanism of adsorption, kinetic data were modelled using the pseudo-first- and pseudo-second-order kinetic model. The results showed that kinetic data followed closely to the pseudo-second order model.

Keywords: Adsorption, kinetics, equilibrium, Activated Carbon, Pharmaceutical compounds.

1. INTRODUCTION

The presence of pharmaceutical compounds in surface waters has become a subject of public concern due to it several environmental and health problems. Indeed, such product they have potential toxicity towards aquatic life and human [1,2]. Although they are developed with the intention of performing a biological effect, they are not easily degraded [3,4] and generally toxic to aquatic environment [5]. There are several studies that have described organic substances degradation. Benitez *et al*. [6] studied gallic acid degradation using UV/H_2O_2 treatment, Fenton's reagent and the photo-Fenton system. Adan *et al*. [7] used hydrogen peroxide over TiO_2/SiO_2 fibres in photochemical and photocatalytic processes to degrade salicylic acid. Kim *et al*. [8] reported the degradation of diclofenac and clofibric acid during UV photolysis and UV/H_2O_2 reactions. Thus, many investigations have shown that substances of pharmaceutical are found as contaminates in surface waters throughout the world [1,9]. Some are known to be toxic even in very small doses [10,11]. Moreover, many of these compounds quickly diffuse into the environment and are resistant to current treatment processes. Therefore, they are frequently found in sewage treatment plants and pharmaceutical wastewater [12]. For all these reasons, their presence in the environment, in particular in water, should be controlled [13-15]. Most of such organic substances are soluble in water. Thus, they can easily contaminate the aquatic ecosystem which has a harmful effect on most of the organism.

There are several technologies and processes for the removal of highly toxic organic compounds from water aqueous solution such as coagulation/ flocculation [16], ozonation [17], precipitation, ion exchange [18], electrochemical reduction, biological degradation [19] and adsorption procedures [20,21]. Therefore, adsorption process seems to be an effective technology for the removal of harmful, pollutants emerging substances from aqueous medium. Moreover, it has been described as reliable and economic separation process for treating industrial and domestic effluents. It is also the best useful tool for protecting the environment [22,23], because of their design simplicity, initial cost, ease of operation and insensitive to toxic substances [24,25].

Several types of adsorbents are classified into mineral, organic or biological origin. Review of literature indicates that the adsorption behavior of pharmaceuticals compounds with various adsorbents like activated carbons, zeolites and clay has been reported [26-28]. Each adsorbent has its own characteristics such as porosity, pore structure and nature of its adsorbing surfaces. In addition, Behera

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et al. [29] have investigated sorption of non-steroidal anti-inflammatory drug onto various soil minerals and activated carbon through batch experiments. Further photocatalytic degradation of different analgesics compound has also been discussed [30]. Adsorption study was also investigated by studying removal of different emerging adsorbates include solvents (pentachlorophenol) [31], pesticides (bentazone, atrazine) [32], additive (bisphenol A) [33], analgesics (paracetamol, ibuprofen) [29,34] and anti-inflammatory agents antibiotic (ofloxacin) [35] by using activated carbons and clays.

The aim of the study was to investigate the adsorption behavior of Salicylic Acid (SA), Clofibric Acid (CA) and Gallic Acid (GA) from aqueous solutions on an activated carbon. The experiments were designed in order to optimize the effect of operating factors as adsorbent doses, concentration of pollutants and pH. Equilibrium data were fitted to several isotherm equations. Adsorption kinetics data were also analyzed by using pseudo-first-order and pseudo-second-order models to data.

2. METHODOLOGY, REAGENTS AND EQUIPMENTS

2.1. Materials and Methods

Organic solutions were prepared by dissolving the appropriate masses of each compound in ultra-pure water. The pH of the solutions, ranging from 3 to 6, was adjusted by HCl (0.1mol/dm^3) or NaOH (0.1 mol/dm^3). All solutions were prepared with ultra-pure water using Milli-Q plus 185 equipment (Millipore) water purification system.

2.2. Adsorbent and Adsorbates

The adsorption capacity of an adsorbent towards the removal of pollutants is directly related with its physicochemical properties. Therefore, to a deeper understanding of the adsorption process it becomes basic to determine the porous texture and the physicochemical features of the carbons. The adsorbent used in this study is a commercial activated carbon obtained from Norit Hydrodarco 3000 and used without further purification.

2.2.1. Determination of pH of the Point of Zero Charge (pHpzc) of the Activated Carbon

An important property of the activated carbon in relation to adsorption studies is the pHpzc [36]. Surface charge density is an important factor in determining the adsorption characteristics of the activated carbon. It is determinated by the pHpzc which is the net surface charge, resulting from the adsorption of the potential determining, H$^+$ and OH$^-$, is zero [37]. Therefore, it is very important to determine the pHpzc for the adsorbent. It is known that the net charge on carbon surface is positive at a solution pH lower than that corresponding to the point of zero charge (pHpzc) of the surface and is negative at a solution pH higher than pHpzc [37,38]. The procedure of pHpzc determination is described as follows: 50 cm^3 of 0.01 mol/dm^3 NaCl solutions were filled in closed Erlenmeyer flasks under agitation at room temperature, the pH of which was adjusted from 2 to 10 by addition of 0.01 mol/dm^3 HCl or NaOH. Then, 0.15 g of solid adsorbent was added to each flask. The content of the flasks was filtered through 0.45 µm Durapore membrane filters after 48 h of agitation and the final pH was then measured [36]. In this method pHpzc is localized at the point where the curve pH_{final} versus $pH_{initial}$ intersects the first bisector and shown in Figure **1**. The fined value is pHpzc = 4.29 showing that the activated carbon is anionic.

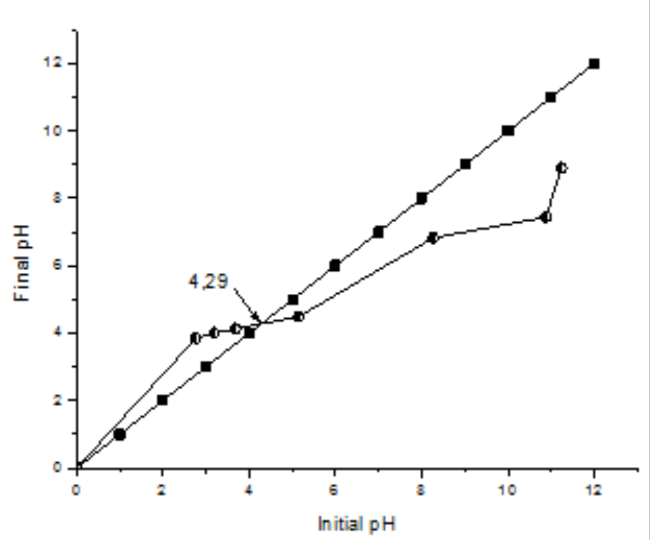


Figure 1: Determination of the point zero of charge of the activated carbon.

2.3. Adsorption Procedure

The equilibrium sorption of molecules was carried out at room temperature. In the isotherm experiment essay, the mass of adsorbent was fixed as 0.05 g AC with 10 cm^3 of solution. In all cases, samples were stirred by magnetic stirrers at for suitable time intervals. The solutions were filtered through a 0.45 µm Durapore membrane filter and the filtrate analyzed for all organic molecules using UV spectrophotometer (Jasco V-730). The data was fitted using the following isotherms: Freundlich, Langmuir, Temkin and Toth. The amount of organic acid adsorbed per unit mass of the activated carbon (mg/g) at equilibrium time was calculated using the equation:

$$q_e = (C_0 - C_e)V / W \quad (1)$$

where C_o and C_e (mg/dm^3) are the initial and equilibrium concentrations of molecule, V (dm^3) is the volume of the solution, and W (g) is the mass of adsorbents used.

Absorbance data were converted into concentration data using calibration relations pre-determined at the wavelength of maximum absorbance for each organic acid species.

2.4. Kinetic Experiments

Sorption kinetics experiments were carried out by contacting solutions of each molecules at several initial concentrations ranging from 5 to 20 mg/dm^3 with activated carbon stoppered conical tubes. Samples were then filtered out at various times from 0 to 130 min and the concentration in the supernatant solution was analyzed.

2.4.1. Preparation of Standard CA, SA and GA Solutions

Adsorption kinetics must be investigated in order to determine the time necessary for reach equilibrium, and to elucidate the mechanism of the adsorption process. To study the adsorption kinetics, 10 cm^3 of CA, SA and GA solutions, with an initial concentration of 50 mg/dm^3, were mixed with different mass of activated carbon in glass vials at a constant agitation speed. The main characteristics of the adsorbates are included in Table **1**.

2.4.2. Analytical Measurement of CA, SA and GA Solutions

The standard calibration curves of known concentrations of CA, SA and GA were plotted by finding out the absorbance at the characteristic wavelength of λ_{max}= 227 nm, λ_{max} = 230 nm and λ_{max} = 268 nm, respectively.

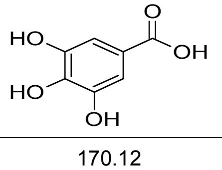
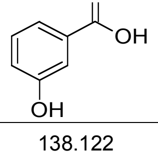
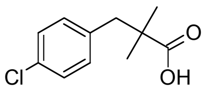
3. RESULTS AND DISCUSSION

The adsorption study processes were carried out by varying the pH of the solution, the adsorbate concentration and the adsorbent dose. In all the cases, the solution volume (⍰) was kept constant. Adsorption kinetics must be investigated in order to determine the time necessary for reach the equilibrium, and to elucidate the mechanism of the adsorption process.

3.1. Effect of the Initial pH

Surface chemistry will therefore play a fundamental role in CA selectivity in adsorption processes. The surface charge of activated carbon, mostly acidic. It depends mainly on the pH of the medium. The surface of a AC can develop either positive charges or negative charges. These charges are responsible for the presence of electrostatic forces attraction or repulsion between the solute and the adsorbent. A negative charge on the surface of the coal results from the presence of groups phenolic. Organic pollutants require high acidity and when pH < pHpzc the adsorbed amount increases with the pH of the solution (promotes the adsorption of organic molecules, increases electrostatic forces between the positive charge of the adsorbent and the negative charge of the phenolic molecule.

Table 1: Characteristics of the Adsorbates

Characteristic	Gallic Acid (GA)	Salicylic Acid (SA)	Clofibric Acid (CA)
IUPAC name	3,4, 5-Trihydroxybenzoic acid	2-Hydroxybenzoic acid	2-(4-Chlorophenoxy)-2-methylpropanoic acid
Chemical formula	$C_7H_6O_5$ or $C_6H_2(OH)_3COOH$	$C_7H_6O_3$ or $C_6H_4(OH)$ COOH	$C_{10}H_{11}ClO_3$
Molecular structure			
Molecular weight	170.12	138.122	214.65
Solubility in water (g/L, 25 ºC)	15	1.8	0.583
pKa at 25 ºC	3.13	2.97	2.8
λ_{max} (nm)	268	230	227

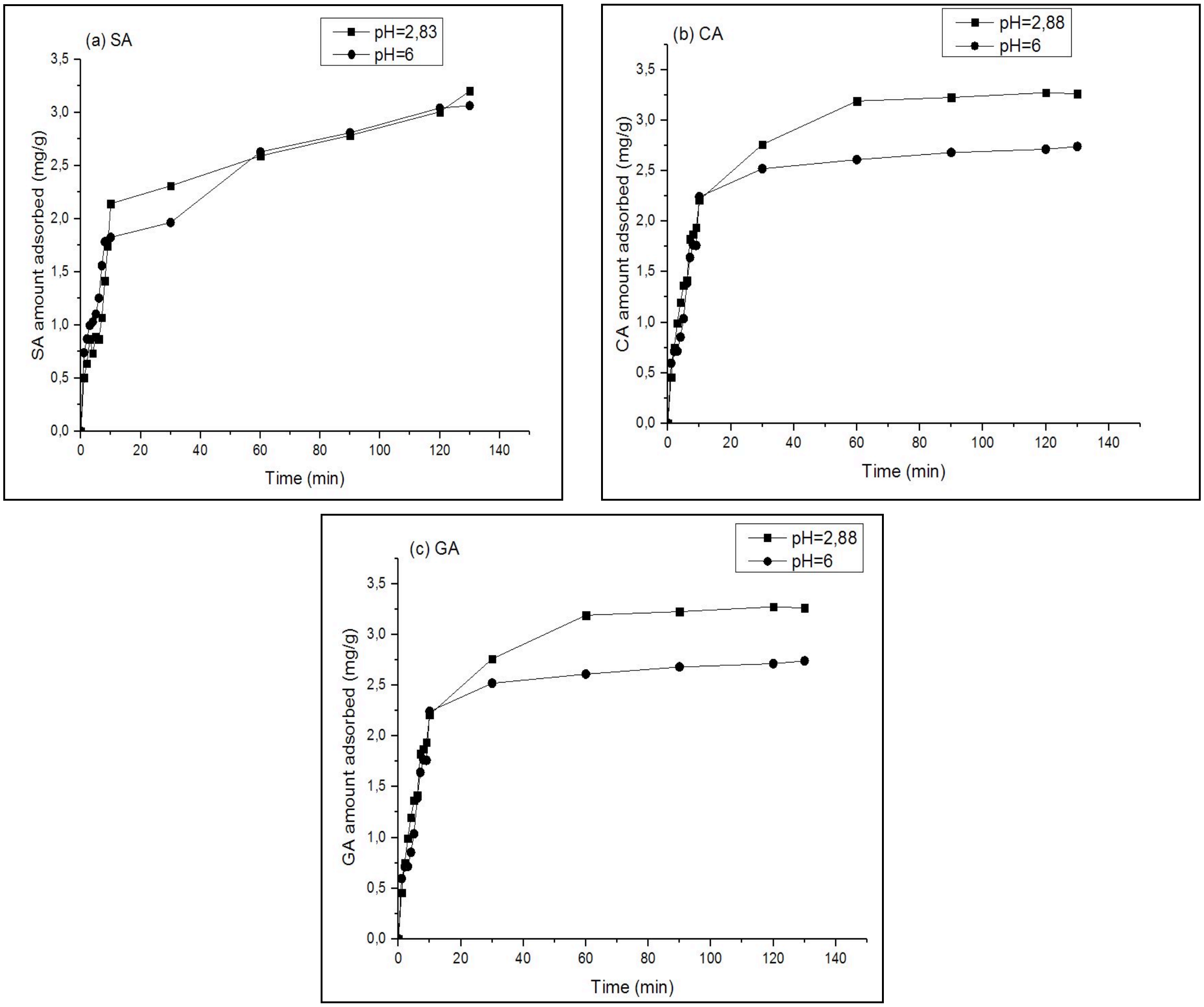


Figure 2: Kinetics results (**a**) SA, (**b**) CA and (**c**) GA for pH = 3 and 6. Conditions: adsorbent dosage = 0.05g, adsorbent concentration = 15 mg/dm^3, T = 24±2°C.

Two initial pHs were considered, 3 and 6, and the results are presented in Figure **2**. The activated carbon surface is negatively charged at pH values above pHpzc and becomes more negatively charged as the pH increases. For this reason the pHs considered in this work, 3 < pHpzc = 4.29 and 6 > pHpzc = 4. The initial concentration of each molecule was 15 mg/dm^3. Samples were taken at various time intervals and after adsorbent-adsorbate separation, the residual concentrations of the molecules were determined by UV-Visible spectrophotometry. The results revealed that the removal of organic compounds uptake decreased with increasing the pH. This might be due to the weakening of electrostatic force of attraction between the oppositely charged adsorbate and adsorbent. At lower pH, the amount adsorbed was found to increase because the surface area was more protonated and competitive adsorption occurred between H$^+$ protons and the organic molecules, the adsorbent surface is able to attracted cations in solution. H$^+$ ions as well as organic anions are adsorbed on the surface. Since the concentration of H$^+$ rapidly exceeds than the weak electrolyte anions, the former adsorb on the carbon surface far in excess of the anions and subsequently enhance the anion adsorption.

3.2. Effect of the Adsorbent Mass

The effect of the adsorbent mass, from 0.025 to 0.1 g, on the adsorption of the pharmaceutical compounds was studied at room temperature and at pH = 3. The results are shown in Figure **3**. The amount of CA, SA and GA adsorbed was found to decrease with increasing adsorbent amount. The amount of acids adsorbed decreased from 3.80 to 1.51 mg.g^{-1}, in the case of CA, from 5.76 to 1.47 mg.g^{-1}, in the case of SA, and from 5.33 to 1.42 mg.g^{-1}, in the case of GA. The

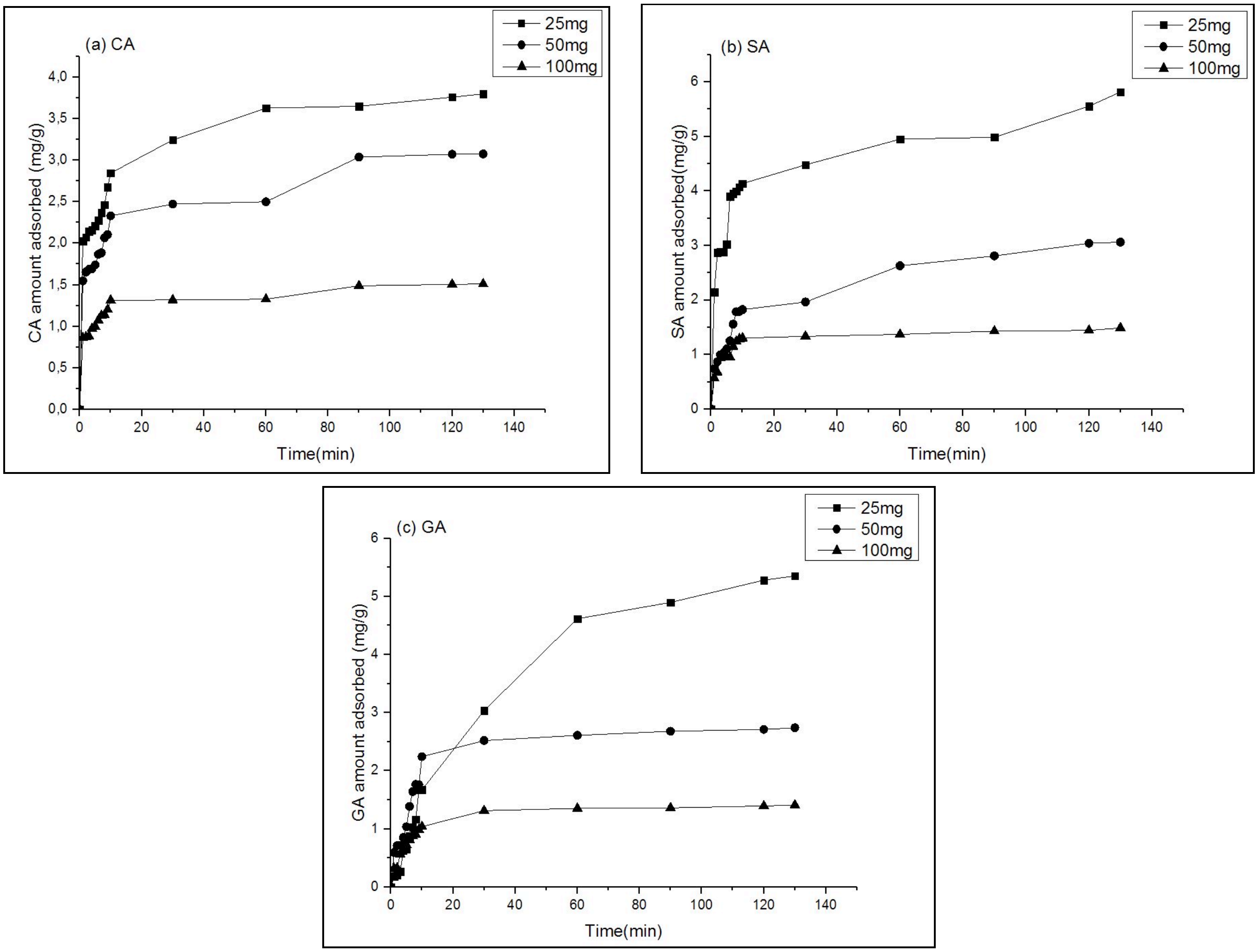


Figure 3: Effect of the activated carbon mass on the adsorption capacity of (**a**) Clofibric acid, (**b**) Salicylic acid and (**c**) Gallic acid. Conditions: adsorbate concentration = 15 mg/dm^3, T = 24±2°C, pH = 3.

decrease in adsorption density with increase in adsorbent amount can be related to the high number of unsaturated adsorption sites. During the adsorption of the acids the molecules reached the boundary layer, then they had to diffuse into the adsorbent surface, and finally, they had to diffuse into the porous structure of adsorbent. Hence, this phenomenon will take a relatively longer contact time.

3.3. Effect of the Initial Concentrations

The percent removal of the organic molecules by the activated carbon, as a function of initial concentration, is shown in Figure **4**. The removal percentage increases with the initial concentration until equilibrium is reached. These results indicate that an increase in initial concentration resulted in a stronger driving force to overcome the mass transfer resistances between liquid and solid phases. The quantity removal of CA and SA increases with the initial concentration, where it is seen that the adsorption of CA increased gradually from 0.73 to 3.05 mg.g^{-1}. It was observed that the adsorption of emergent molecules increases with initial concentration. These results also indicate that the sorption process can be considered very fast because a significant amount of SA, CA and GA were adsorbed on the activated carbon within the first 10 min of adsorption.

3.4. Equilibrium Studies

An adsorption isotherm is characterized by certain constant values, which express the surface properties and the affinity of the adsorbent, and can also be used to compare the adsorptive capacities of the adsorbent for various pollutants. The analysis of equilibrium adsorption data by fitting them to several isotherm models is an important step to find design the adsorption systems. There are many models that can explain the adsorption equilibria. Freundlich, Langmuir and Temkin models are the most frequently employed models used for evaluating the experimentally acquired

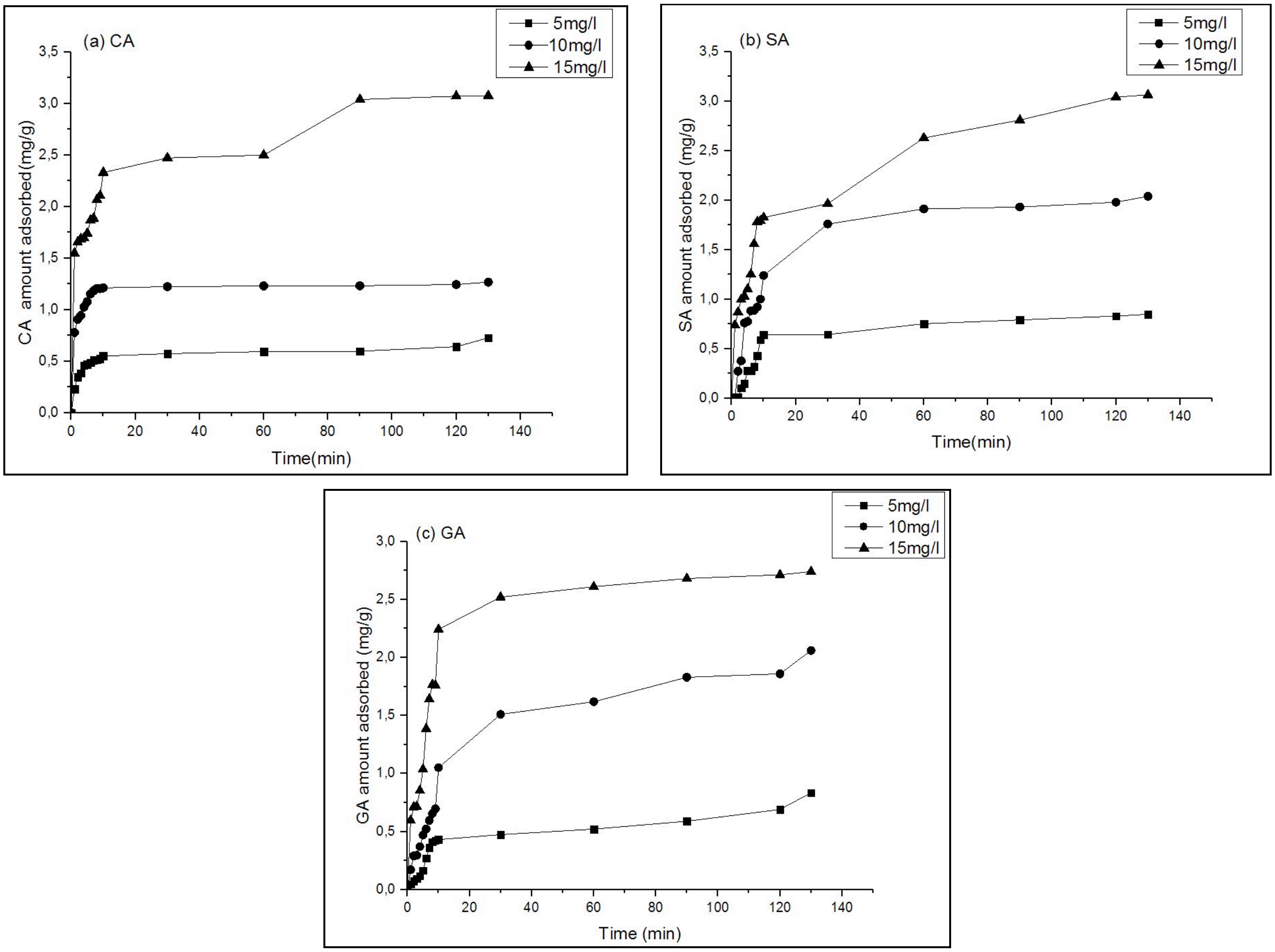


Figure 4: Effect of the initial concentration of (**a**) CA, (**b**) SA and (**c**) GA on the adsorption efficiency. Conditions: T = 24±2°C, adsorbent amount = 0.050 g, pH = 3.

adsorption isotherms [39]. In this work, these models were used to describe the relationship between the amount of pharmaceutical compounds adsorbed and its equilibrium concentration.

It was observed that the adsorption isotherm of SA, CA and GA from the aqueous solution with activated carbon refer to the S-type isotherms according to the Giles's classification [37], see Figure **5**. S-type isotherm is the adsorption described, at which the molecules of solute are adsorbed vertically as is the case with molecules having a single functional group, or, when the molecules are in competition with strong adsorption with the molecule solvent [40]. It is therefore possible to observe a cooperative adsorption of molecules. By the S-type isotherm the adsorption is described, at which the strength of the interaction between the solute and the adsorbent is less than the force of interaction between the adsorbed molecules, which can explain the formation of hydrogen bonds. In general, the adsorption occurring in a solid-liquid is complex, simply because there is always competitive adsorption between molecules and solution. The situation is more complex because water itself a reactive solvent that is present in various forms.

The Freundlich isotherm model is the earliest known relationship describing the non-ideal and reversible adsorption, which can be applied to multilayer adsorption, on the basis of an assumption concerning the energetic surface heterogeneity. This model is applicable to adsorption processes that occur on heterogonous surfaces. The Freundlich isotherm is introduced as an empirical model, where q_e represents the amount adsorbed per amount of adsorbent at the equilibrium (mg/g), Ce represents the equilibrium concentration (mg/dm^3), and k_F and n are parameters that depend on the adsorbate and adsorbent interaction. The $1/n$ indicates the degree of non-linearity between the solution concentration and the adsorption as follows: for values in the range of $0.1 < 1/n < 1$, the adsorption is favorable. Hence, the empirical equation can be written as:

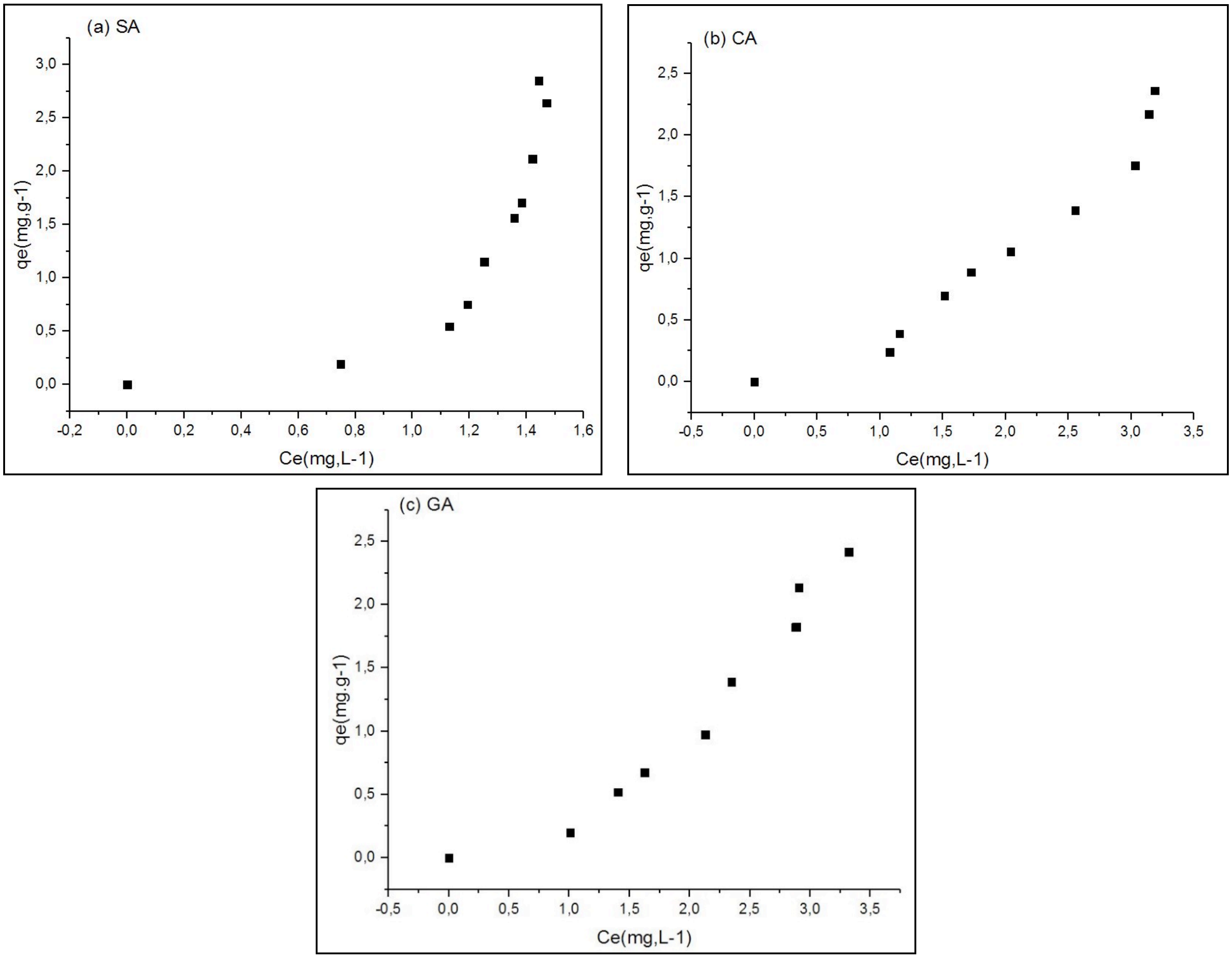


Figure 5: Equilibrium adsorption isotherms, measured at temperature 24±2°C. (**a**) SA, (**b**) CA and (**c**) GA.

$$q_e = k_F.C_e^{\frac{1}{n}} \tag{2}$$

The equation can be linearized as (see Figure **6**):

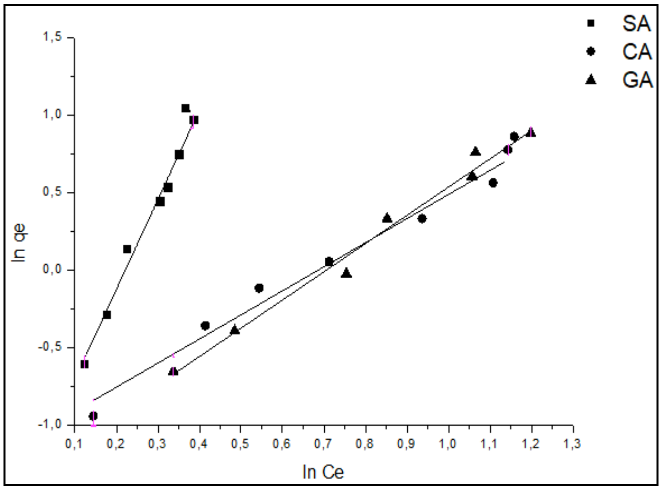


Figure 6: Adsorption isotherms from the Freundlich model. Conditions: T = 24±2°C, adsorbent amount = 0.050 g, pH = 3.

$$\ln q_e = \ln k_F + \frac{1}{n}\ln C_e \tag{3}$$

The Temkin isotherm contains a factor that explicitly take into account the account of adsorbent–adsorbate interactions. The model assumes that the heat of adsorption of all molecules in the layer would decrease linearly rather than logarithmic with coverage. The model is given by the following equation:

$$q_e = \frac{RT}{b}\ln(AC_e) \tag{4}$$

The equation can be linearized as (see Figure **7**):

$$q_e = \frac{RT}{b}\ln(A) + \frac{RT}{b}\ln(C_e) \tag{5}$$

where $\frac{RT}{b} = B$

The Langmuir isotherm is applied to describe the formation of a monolayer adsorbate on the outer surface of the adsorbent, and after that no further adsorption takes place [41]. The model is given by the following equation:

$$q_e = \frac{k_L \cdot q_L \cdot C_e}{1 + k_L \cdot C_e} \tag{6}$$

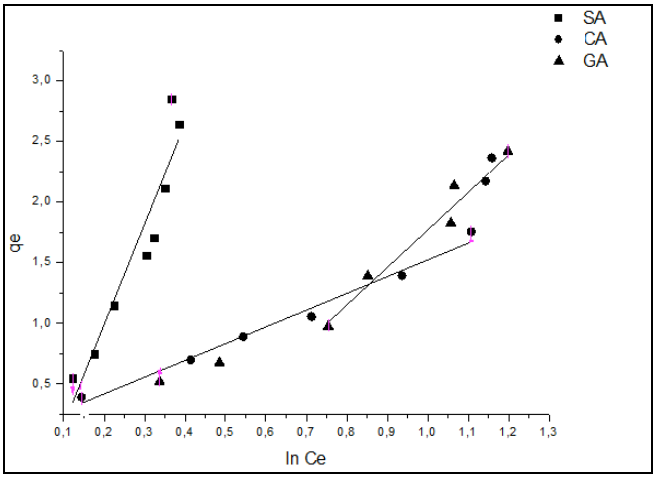


Figure 7: Adsorption isotherms from the Temkin model. Conditions: T = 24±2°C, adsorbent amount = 0.050 g, pH = 3.

The equation can be linearized as (see Figure **8**):

$$\frac{q_e}{C_e} = -bq_e + bq_L \tag{7}$$

The estimated model parameters and regression coefficient values of the equilibrium isotherm models for adsorption of SA, CA and GA on the activated carbon are given in Table **2**.

The Toth equation is related to multilayer adsorption. This model is most useful in describing heterogeneous adsorption systems which satisfy both low and high-end boundary of adsorbate concentration. The Toth model equation given as follows:

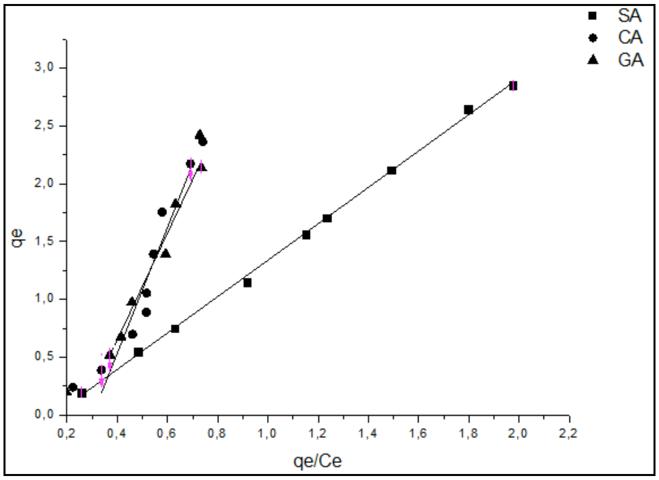


Figure 8: Adsorption isotherms from the Langmuir model. Conditions: T = 24±2°C, adsorbent amount = 0.050 g, pH = 3.

$$q_e = \frac{q_{T.}C_e}{\left(\frac{1}{k_T} + C_e^{\,m_T}\right)^{\frac{1}{m_T}}} \tag{8}$$

The equation can be linearized as (see Figure **9**):

$$\ln\left(\frac{q_e}{q_T}\right) = \ln(C_e) + \frac{1}{m_T} \cdot \ln\left(\frac{1}{k_T} + C^{m_T}\right) \tag{9}$$

where *qe* is the adsorbed amount at equilibrium (mg/g), *Ce* is the equilibrium concentration of the adsorbate (mg/dm^3), q_T the Toth maximum adsorption capacity (mg/g), k_T is the Toth equilibrium constant, and m_T is

Table 2: Results from Fittings to the Langmuir, Freundlich and Temkin Models Corresponding to the Equilibrium Data of the Adsorption of SA, CA and GA on the Activated Carbon

		Salicylic Acid	Clofibric Acid	Gallic Acid
		(SA)	(CA)	(GA)
Freundlich	kF/(mg/g)	0.49	0.28	0.23
	1/n	3.99	1.80	2.02
	N	0.25	0.56	0.49
	R^2	0.93	0.96	0.98
Temkin	A/(mg/g)	1.22	1.01	0.96
	B	3.61	1.727	1.856
	R^2	0.63	0.92	0.91
Langmuir	B	-0.64	-0.22	-0.24
	q$_L$(mg/g)	0.37	4.83	3.73
	R^2	0.998	0.89	0.94

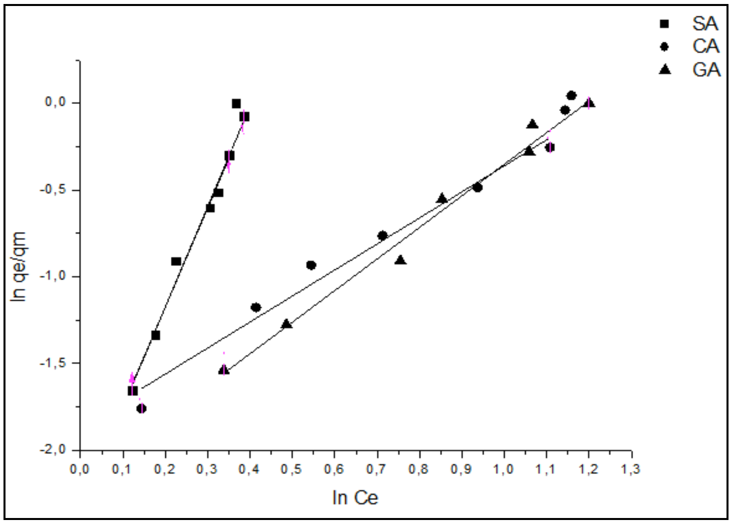


Figure 9: Adsorption isotherms from the Toth model. Conditions: T = 24±2°C, adsorbent amount = 0.050 g, pH = 3.

the Toth model exponent. If m_T = 1 this expression reduces to the Langmuir adsorption isotherm.

3.5. Kinetic Studies

Kinetics models are used to examine the rate of the adsorption process and the potential rate controlling step. The kinetic models pseudo-first order and the pseudo-second order [42] were employed to investigate the kinetics of adsorption of emergent molecules on the activated carbon.

It has been assumed that in the pseudo-first order model the adsorption rate at time *t* is proportional to the difference between the adsorbed amount at equilibrium, q_e, and the quantity q_t adsorbed at this time. The rate constant of adsorption of the first order is deduced from the model established by Lager green. It is expressed according to the following equation:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{10}$$

Integrating the equation, the kinetic rate expression becomes as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \tag{11}$$

where q_e is the equilibrium sorption capacity (mg/g), q_t is the sorption capacity at any time *t* (mg/g) and k_1 is the pseudo-first order rate constant (min^{-1}). The values of the parameters are included in Table **3**.

Experimental results were also analyzed in terms of pseudo-second order model. This model is often used successfully to describe the kinetics of the pollutant-binding reaction on the adsorbent. The model makes it possible to characterize the kinetics of adsorption taking into account both the case of a fast fixation of the solutes on the most reactive sites and that of a slow fixation on the sites of weak energy [41]. The pseudo-second order adsorption kinetic rate equation is expressed as:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{12}$$

After integration, the kinetic rate expression becomes as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e} + \frac{t}{q_e} \tag{13}$$

where k_2 (g.mg^{-1}min^{-1}) is the rate constant of pseudo-second order adsorption, q_e and q_t (mg/g) are the adsorption capacity at equilibrium and at time *t*. If the

Table 3: Kinetic Parameters of Adsorption of SA, CA and GA on the Activated Carbon (Pseudo-First-Order)

		CA	SA	GA
Pseudo first order	k_1/ (1/min)	0.071	0.098	0.15
	$qe_{,1}$/(mg/g)	1.26	1.50	1.58
	R^2	0.85	0.94	0.87

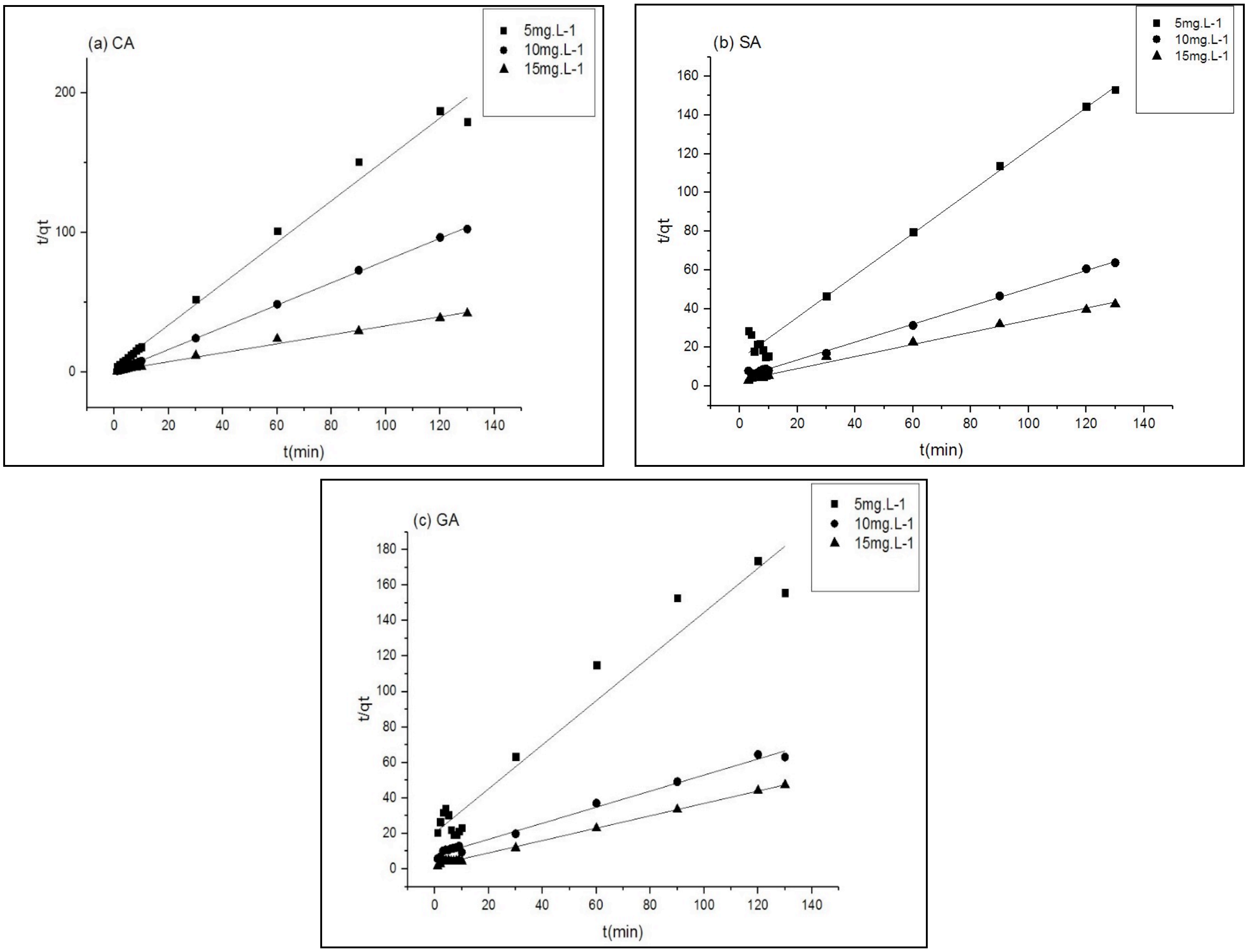


Figure 10: Pseudo-second order kinetics fits. Conditions: T = 24±2°C, adsorbent amount = 0.050 g, pH = 3.

Table 4: Kinetic parameters of adsorption of SA, CA and GA on the activated carbon (pseudo-second-order)

		CA	SA	GA
Pseudo second order	k_2/ (g.mg^{-1}.min^{-1})	1.383	0.105	0.058
	qe,$_2$/(mg/g)	1.265	2.169	2.212
	R^2	0.999	0.997	0.991

second order kinetic equation is applicable, the plot of *t/q* against *t* should give a linear relationship (see Figure **10**). The values of the parameters are included in Table **4**. These results indicated that the pseudo-second order kinetic model provide an excellent correlation of the adsorption of CA, SA and GA on the activated carbon. Thus, the reaction is more inclined towards chemisorption.

CONCLUSIONS

In the present study the potentiality of a commercial activated carbon for remove three emerging contaminants, clofibric acid, salicylic acid and gallic acid from aqueous solutions were evaluated and used to develop a better understanding of the mechanisms of adsorption of these compounds. Several conclusions can be summarized from the experimental results:

- The initial concentration of the pollutants and the pH of solution are important parameters that can affect the adsorption process.
- A very important effect of the pH on the removal of organic pollutants was observed. The adsorption capacity was found to be maximum in the acidic range for all the compounds.

- The percent removal of CA, SA and GA increases with increase of the adsorbate dose.
- The kinetic study shows that 10 minutes are sufficient for adsorbing a large amount of these compounds from aqueous solutions.
- The kinetics data followed closely to the pseudo-second order model.

Finally, the activated carbon can be considered as an efficient adsorbent for the removal of organic pollutants from wastewater.

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