

# Thermodynamic Study of Amoxicillin and Naphthalene Adsorption on Activated Carbon Derived from *Salvadora persica*

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## ABSTRACT

**Objectives:** The pollution of aquatic bodies by Naphthalene and antibiotics effluent from activities of many industry is a significant environmental challenge affecting the ecosystem. Batch process was used to remove Naphthalene (NT) and Amoxicillin (AMO) with activated carbon prepared of *Salvadora persica* as adsorbent (ACSP). **Methods:** Batch studies were performed to evaluate and optimize the effects of various parameters such as contact time, temperature of the solution, NT and AMO concentrations and adsorbent dosage. The adsorbent characteristics were determined using SEM and BET technique. **Results:** The surface areas of ACSP and SP were 289.4 and 51.27 m<sup>2</sup>/g, for the adsorbents respectively. The equilibrium of the adsorption process was attained at 75 min adsorbent. The interaction of NT and AMO ions onto ACSP was spontaneously endothermic and it

agreed with pseudo-second-order equation. **Conclusion:** This attempt has shown the potential of modified clay as a suitable eco-friendly sorbent for removing NT and AMO.

**Key words:** Naphthalene, Adsorption, Amoxicillin, ACSP, Thermodynamics.

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DOI: 10.5530/ijpi.2021.1.8

## INTRODUCTION

In recent century the supply of water for domestic, agricultural and industrial usage as well as for production of green fuels is highly essential for the survival of our world. Industrial effluents carry a large variety of high-concentration hydrocarbons into the environmental water.<sup>1,2</sup> Polycyclic aromatic hydrocarbons (PAHs) are kind of industrial effluents with two or more aromatic rings that produce during the incomplete burning of coal, oil, gas, or other organic compounds.<sup>3,4</sup> These types of pollutants are highly toxic and can cause carcinogenic and mutagenic effects.<sup>5</sup> Naphthalene is the simplest form of PAHs that may release (up to 2 mg/L) into ambient water from industrial sectors.<sup>6</sup> The allowable concentration of naphthalene reported by the US Environmental Agency for aquatic life protection is 0.6 mg/L.<sup>7</sup> The higher concentrations of naphthalene in water may lead to serious health problems including hemolytic anemia in children and anemia.<sup>8</sup>

Pharmaceuticals especially antibiotics are among the numerous emerging pollutants that have been detected in different water systems.<sup>9</sup> Studies have shown that most antibiotics cannot be absorbed and digested well by living organisms. For this reason, approximately 25–75% of the antibiotics ingested by humans and animals form part of the excrement and urine.<sup>10,11</sup> In general, antibiotics reach water systems through the discharge of poorly treated wastewaters and effluents discharges.<sup>12</sup> The presence of the antibiotics in aquatic systems is of concern because of their toxicity to aquatic life and other organisms.<sup>13</sup> Their toxicity may cause a long-term effect on ecological sustainability even if they are present in trace levels.<sup>14</sup> In addition, antibiotics are known to have adverse effects such as emergence of multi-resistant bacteria, acute and chronic toxicity in humans and animals.<sup>15,16</sup>

Among other antibiotics, amoxicillin (AMO) is the most widely used drug for the treatment of bacterial infections such as ear, nose, throat, skin and lower respiratory tract.<sup>17</sup> It has been reported that over 80% of

oral administration of AMO in humans is excreted through urine after 2 h of consumption.<sup>18</sup> Recently, the presence of AMO in concentration ranges of ng/L to mg/L in surface water, domestic and industrial wastewater and hospital waste has been reported.<sup>19,20</sup>

Membrane separation, advanced oxidation processes, biodegradation and adsorption are commonly used techniques for the removal of pollutant from wastewater.<sup>21,22</sup> Among these techniques, adsorption processes by using solid adsorbents have the advantages of high removal efficiency, low operational cost, easy handling and facile regeneration.<sup>23</sup> Therefore, adsorption processes have become one of the best treatment technologies to reduce the level of organic pollutants in wastewater.<sup>24,25</sup>

The choice of activated carbon precursor largely depends on its availability, cost and purity, but the manufacturing process and intended applications of the product are also important considerations.<sup>26,27</sup> Therefore, evaluation of biomass is getting increased attention over the world as it is renewable, widely available, cheap and environmental friendly.<sup>28</sup> *Salvadora persica* or Arak is an evergreen perennial halophyte belonging to the family Salvadoraceae and it is considered an important crop plant for marginal coastal areas of arid and semiarid regions, where the salt concentration of the soil would inhibit the growth of most other crops.<sup>29</sup> *Salvadora persica* has wide geographic distribution ranging from Rajasthan (India), Nepal and Malaysia in the east through Pakistan, Iran, Iraq, Saudi Arabia and Egypt to Mauritania in the west and from North Africa through Sudan, Ethiopia and Central Africa to southwestern Africa.<sup>29</sup>

The objective of this work is to investigate the potential and adsorption efficiency of active carbon from *Salvadora persica* (ACSP) for the removal of AMO and NT from aqueous solutions. The effects of operational parameters such as temperature, amount of adsorbent, initial AMO and

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NT concentration and contact time on the adsorption of AMO and NT onto ACSP were analyzed to determine the factors controlling the rate of adsorption. Kinetic studies were also evaluated using Pseudo-first order, Pseudo-second order and Intraparticle diffusion, as well as the thermodynamic data were studied.

## MATERIALS AND METHODS

All the chemicals used in this study were of analytical grade purchased from Sigma Aldrich Co. *Salvadora persica* (SP) were collected from southern Iran. They were then transported to the laboratory and washed with deionized water. Thus SP were oven dried at 105°C for 24 hr. Dried SP were grounded to a fine powder and then sieved using a standard sieve of 90 µm to 150 µm. Part of the raw SP powder was soaked in 50% phosphoric acid for 24 hr at 105°C and then oven dried for 48 hr at 105°C. Dried sample was charred using a heating mantle at 300°C for 24 hr followed by ignition in an electric muffle furnace at 550°C for 24 hr. The sample was left to cool and washed with 0.1 M HCl and then washed with 0.1M NaOH to raise the pH of the activated carbon to 7. The sample was then oven dried at 105°C for 24 hr and kept in a stoppered bottle ready for use.

**AMO and NT removal experiments:** AMO and NT removal experiments with the SP stem ash were carried out as batch tests in 200 mL flasks under magnetic stirring. Each test consisted of preparing a 100 mL of AMO and NT solution with a desired initial concentration and pH by diluting the stock dye solution with distilled water and transferring it into the beaker on the magnetic stirrer. The pH of the solution was adjusted using 0.1N HCl or NaOH solutions. A known mass of SP stem ash (adsorbent dosage) was then added to the solution and the obtained suspension was immediately stirred for a predefined time. After the desired contact time, the samples were withdrawn from the mixture by using a micropipette and centrifuged for 10 min at 3600 rpm. All tests were performed in duplicate to insure the reproducibility of the results; the mean of the measurements is reported. All experiments were performed at room temperature. The investigated ranges of the experimental variables were as follows: AMO and NT concentration (10 to 100 mg/L), SP Mass (0.1-2 g/L) and mixing time (10 to 180 min), at a temperature between 10 -50°C and at a constant pH of 7. After centrifugation, supernatants were analyzed for the determination of the final concentration of AMO and NT by using an UV-VIS spectrophotometer set at maximum absorbance. Then, the amount of AMO and NT adsorbed,  $q_e$  (mg/g), was obtained as follows:<sup>30</sup>

$$q_e = \frac{V(C_0 - C_e)}{W}$$

Where,  $C_0$  and  $C_e$  are the initial and equilibrium liquid phase concentration of AMO and NT (mg/L), respectively; V is the volume

of the solution (L) and W is the amount of SP used (g). To express the percent of AMO and NT removal, the following equation was used:<sup>31</sup>

$$\% R = \frac{(C_0 - C_e)}{C_0} \times 100$$

## RESULTS

SP and ACSP have surface area of 51.27 and 289.4 m<sup>2</sup>/g respectively. The effect of acid activation caused the creation of more surface which resulted to about nine-fold increment compared to SP. SEM analysis are shown in Figure 1.

The contact time is one of the most important parameters for practical application. The effects of contact time on adsorption were studied at 30°C, initial AMO and NT concentration of 20 mg/L and 1 g/L adsorbent dosage for 10 to 180 min. Figure 2 shows the effect of contact time on the adsorption capacity and percent removal of AMO and NT by ACSP at a constant initial concentration. The adsorption capacity and percent removal of AMO and NT by ACSP considerably increased during the initial adsorption stage and then continued to increase at a relatively slow speed with contact time until a state of equilibrium was attained after 60 min.

Different dosages of the ACSP were transferred into separate flasks after which 50 ml of 60 mg/L AMO and NT was added. Accumulation of AMO and NT ions in the ACSP as a function of the sorbent mass is shown in Figure 3.

Several kinetic models have been applied to examine the mechanism of the adsorption of dye from aqueous solution. Pseudo first order, pseudo second order and Intra-particle diffusion kinetic models were studied.

The linearized Pseudo-first order rate equation is expressed as:

$$\text{Log}(q_e - q_t) = \text{log } q_e - \frac{K_1}{2.303} t$$

Where  $q_e$  and  $q_t$  are the amounts of AMO and NT adsorbed (mg/g) at equilibrium and at time t (min) respectively,  $K_1$  (1/min) is the adsorption rate constant, t is the contact time (min).

The linear form of pseudo-second order is given as:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e}$$

$K_2$  is pseudo-second order constant (g/mg min). As shown in Table 1, indicating that the pseudo-second order equation fitted the experimental data well with a correlation close to unity.

The intra-particle diffusion model was applied to investigate the adsorption rate limiting step. The intra-particle diffusion model (IPDM) formula as given by Weber and Morris is.

**Table 1: The results of kinetic model studies related to the AMO and NT adsorption onto ACSP.**

Parameters		Intraparticle diffusion			Pseudo-first order			Pseudo-second order		
$C_0$ (mg/L)	$(q_e)_{exp}$	K	I	R <sup>2</sup>	$(q_e)_{cal}$	$K_1$	R <sup>2</sup>	$(q_e)_{cal}$	$K_2$	R <sup>2</sup>
25	24.25	0.541	4.72	0.851	10.25	0.011	0.811	23.11	0.0037	0.997
50	45.14	0.472	6.27	0.864	24.38	0.025	0.795	40.75	0.0032	0.993
75	71.57	0.328	7.59	0.886	39.14	0.021	0.746	64.39	0.0024	0.994
100	92.37	0.169	8.76	0.894	51.76	0.027	0.872	90.82	0.0023	0.995
25	19.44	0.325	5.11	0.804	7.25	0.009	0.785	17.85	0.0057	0.993
50	35.06	0.289	7.89	0.818	14.78	0.008	0.852	36.54	0.0044	0.992
75	59.72	0.248	9.25	0.801	29.35	0.011	0.789	61.72	0.0036	0.998
100	74.38	0.214	10.24	0.838	44.86	0.013	0.741	84.75	0.0031	0.997

$$q_t = Kt^{1/2} + I$$

$K$  (mg/(gmin<sup>1/2</sup>)) is the rate constant for intra-particle diffusion (IPD).

The value of  $I$  shows that the line did not pass through the origin,  $C \neq 0$ , implying that IPDM is not the sole rate limiting step, hence, there are some other processes affecting the adsorption. The value of  $I$  may be due to differences in mass movement of adsorbate in the beginning and ending processes of the sorption, implicating boundary layer control in the adsorption.

The thermodynamic parameters depend on the reaction process and they can help in the detail study of adsorption process mechanism. In this experimental study, different temperature values were used. The enthalpy ( $\Delta H^0$ ) in KJ/mol, entropy ( $\Delta S^0$ ) in KJ/mol and standard free energy ( $\Delta G^0$ ) in kJ/mol, for sorption of AMO and NT onto ACSP was determined with following equations:

$$\Delta G^0 = \Delta H^0 - T\Delta S^0$$

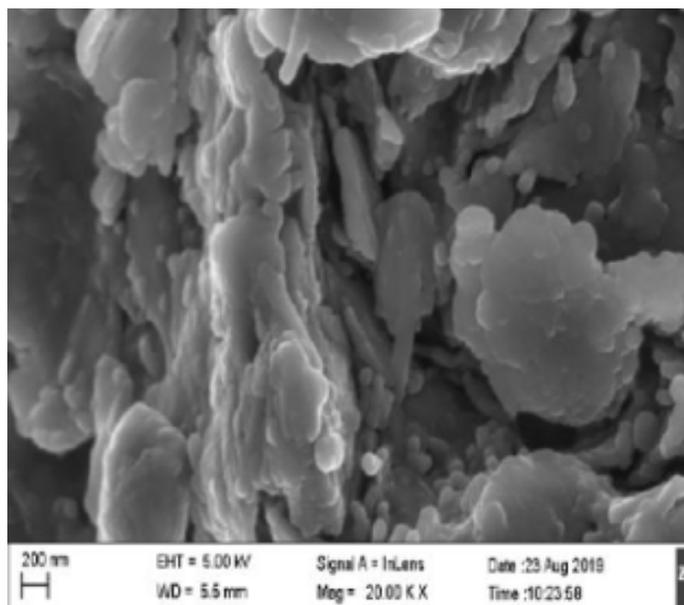
$$\ln(KL) = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}$$

$$\Delta G^0 = -RT \ln KL$$

Where, the universal gas constant ( $R$ ) in J/mol. K, Temperature ( $T$ ) in kelvin (K) and  $K_L$  is the equilibrium constant. Thermodynamic conditions will give more information on the reaction mechanism and sorption process (Table 2).

**Table 2: Thermodynamic parameters for the adsorption of AMO and NT onto ACSP.**

Temperature (K)	G <sup>0</sup> (KJ/mol)	H <sup>0</sup> (KJ/mol)	S <sup>0</sup> (KJ/mol K)
293	-4.719		
303	-6.051	37.49	0.276
313	-7.274		
323	-8.649		
293	-2.945		
303	-4.358	30.78	0.118
313	-5.572		
323	-6.085		



**Figure 1:** SEM images of ACSP.

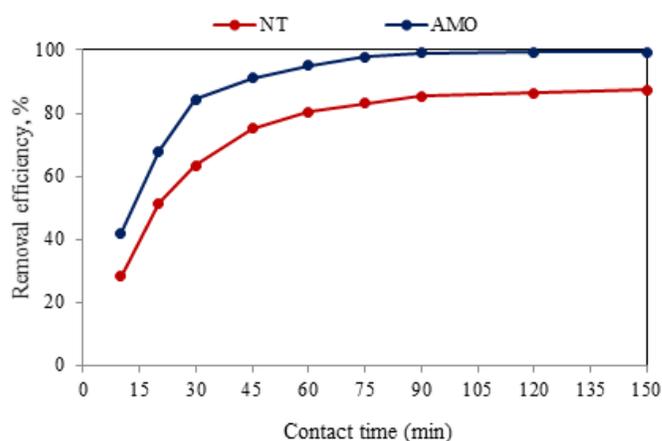
## DISCUSSION

The primary aim of acid activation of AC is to increase the adsorption area and void capacity because they affect adsorbent behaviour greatly. The adsorption tendency as exhibited by ACSP materials is attributed to their corresponding large available surface area.<sup>28</sup> Furthermore, studies by many researchers has shown that the relationship existing between ACSP available area and its adsorption ability is directly proportional.<sup>29,30</sup>

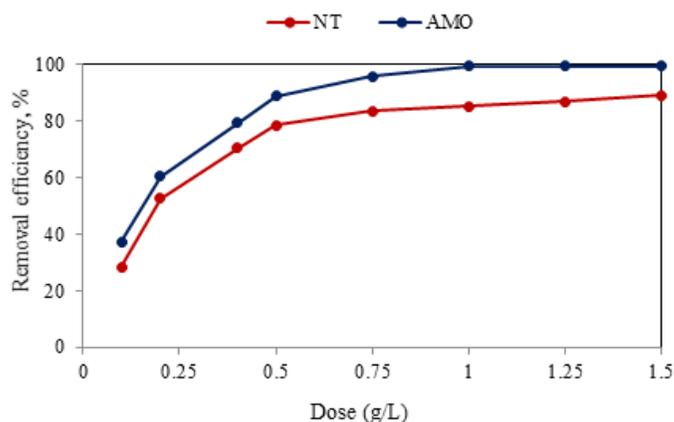
Figure 1 show the images of ACSP. Their surfaces are irregular, rough, porous and heterogeneous structure which is a suitable property of an adsorbent,<sup>31</sup> and this can aid molecular transport during the adsorption process. This porous structure suggests that adsorption in wastewater is chemisorption or physisorption process.

Rapid increase at the onset of adsorption process may be attributed to high solute concentration gradient as well as vacant pore voids. However, the contact time increased and AMO and NT uptake slows down, this is because the available adsorption sites is been occupied. The movement of AMO and NT into the pore surface will be slow due to few sites available; this will continue until equilibrium is achieved.<sup>32</sup>

From Figure 3, percentage adsorption increased between 0.1 to 1 g/L; the dose increased the availability of adsorption sites. However, beyond 1 g/L, AMO and NT were few in the reaction medium to interaction with available surface area, since almost all ions are adsorbed. Hence,



**Figure 2:** Effect of contact time on AMO and NT removal efficiency (pH= 7, dose mass= 1 g/L, Tem= 30°C).



**Figure 3:** Effect of ACSP mass on AMO and NT removal efficiency ( $C_0 = 20$  mg/L, pH= 7, time = 60 min, Tem= 30°C).

the adsorption gradually became constant and additional adsorbent will make no significant a contribution.<sup>33</sup>

In industrial design for operation of adsorption chambers, a simple yet efficient mathematical correlation is required and followed. The interaction of the adsorbate depends primarily on the type of adsorbate but more so on the physico-chemical characteristics of the sorption system in entirety.<sup>34</sup> The sorption occurs in various stages: a) the adsorbent when comes in contact with the comparatively high voluminous aqueous adsorbate, a simple osmotic interaction takes place at the adsorbent's outer surface throughout, b) once a breach is created at the surface of the adsorbent, aqueous adsorbate moves or diffuses inside the adsorbent in a pathway created due to vanderwaal forces of the pore structure and c) sorption till pore saturation. Moreover, combinations of these operational parameters are optimized under various conditions to mimic the fluctuating influx loading in the sorption system.<sup>32</sup> Hence various assumptions pertaining to the adsorption system under specific operating conditions needs to be hypothesized with respect to the physico-chemical characteristics, surface adsorbing potential and thermo mechanical stability of the adsorbent.<sup>29</sup>

Some reaction models predict that the sorption is the rate limiting step in the adsorption process; while some models support the theory that the diffusion is the rate limiting step. So, it is imperative that fitting the models that best describes the system helps in understanding the other variables which will otherwise be overlooked.<sup>34</sup> Diffusion of liquid adsorbate into the adsorbent is a predominant case in adsorption where the solute molecules enter into the adsorbent through the surface, occupy the pores and finally reach the saturation.<sup>35</sup> The intra-particle diffusion model utilizes the assumption that the vanderwaals surface forces are predominant in the surface of the adsorbent and these forces have the ability to pull the solute molecule towards the vacant porous sites. At lower concentrations, the model bears striking resemblance with Henry's isotherm, only that the Weber Morris model is time dependent.<sup>36</sup>

Thermodynamic conditions will give more information on the reaction mechanism and sorption process (Table 2). They were examined at different temperature of 25°C, 30°C, 35°C, 40°C and 45°C. The value of enthalpy change ( $\Delta H^\circ$ ) is positive, which indicates an endothermic process.<sup>36</sup> The enthalpy of AMO and NT onto ACSP are 19.4 and 21.7 KJ/mol respectively. This suggests that the interaction of AMO and NT ions with ACSP is endothermic.<sup>37</sup> The entropy change ( $\Delta S^\circ$ ) is positive and signifies a strong affinity of ions for adsorbent and a high degree of randomness.<sup>38</sup> The value of Gibbs free energy ( $\Delta G^\circ$ ) is negative and it measures the spontaneity of a reaction process. From Table 2, it can be seen that as temperature increased from 293-323 K, the free energy increased (become more negative). This indicates a favorable and energetically spontaneous reaction.<sup>39-41</sup> The analysis shows the process was characterized by high randomness and spontaneous endothermic.

## CONCLUSION

The applicability of ACSP prepared from SP was studied through sorption of AMO and NT from aqueous solution. The sorption experiments were designed so that to predict the behavior pattern of the adsorbent. The adsorbent dosage was optimized at 1 g/L while the experiments were conducted at temperature (20°C to 50°C) to mimic the treatment plant operability. The diffusion kinetics was determined to predict the rate determining step and to substantiate the intra-particle diffusion at lower concentrations. After consideration of the thermodynamics and kinetics and diffusion studies, it could be suggested that ACSP could be suggested for large scale operations due to the raw material availability.

## ACKNOWLEDGEMENT

The authors are grateful from Student research committee of Zahedan University of Medical Sciences because of supporting of this research.

## CONFLICT OF INTEREST

The authors declare no conflict of interest.

## ABBREVIATIONS

ACSP: Activated carbon-*Salvadora persica*; NT: Naphthalene; AMO: Amoxicillin.

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**Article History:** Submission Date : 01-11-2020; Revised Date : 05-12-2020; Acceptance Date : 01-01-2021

**Cite this article:** Mostafapour FK, Haseeb S, Balarak D, Moein H, Sajadi AA. Thermodynamic Study of Amoxicillin and Naphthalene Adsorption on Activated Carbon Derived from *Salvadora persica*. *Int. J. Pharm. Investigation*. 2021;11(1):41-5.