# REMOVAL OF PARACETAMOL FROM AQUEOUS SOLUTION BY ADSORPTION ONTO CARBON MATERIAL OBTAINED FROM NECTARINE KERNEL

**E. Tskhakaia*, N. Ananiashvili, G. Tatishvili, A. Chikhladze, S. Tskitishvili, N. Giorgadze, R. Jorbenadze**

*Iv. Javakhishvili TSU Institute of Inorganic Chemistry and Electrochemistry*
**e-mail: eizaveta.tskhakaia@tsu.ge*

*Received 28.03.2024*
*Accepted 29.06.2024*

***Abstract:*** *Removal of paracetamol from aqueous solution onto carbon material obtained from nectarine kernel by the original technology was studied in this work. Process performance was observed by varying the experimental features, such as retention time, particle size, adsorbent dose, solution pH, and concentration. Adsorption dynamics were studied employing Langmuir isotherms and Freundlich isotherms. The results showed that the adsorption of paracetamol on the sorbent developed by us was explained by isothermal dynamics. The maximum adsorption capacity of the adsorbent was found to be 24.65 mg/g (S% - 98.6). The study results proved that the obtained carbon material is an efficient, cheap sorbent that can be used for efficient removal of paracetamol from aqueous systems.*
***Keywords:*** *adsorption, paracetamol, carbon material, treatment, raw material.*
***DOI: 10.32737/2221-8688-2025-1-59-67***

## Introduction

During the last three decades, special attention was brought to studying newly discovered chemical pollutants. One of the most diverse and noteworthy groups of pollutants is pharmaceutical substances, which are produced in large amounts with their consumption increasing each year [1, 2].

Nowadays, one of the main problems is a pollution of water with pharmaceutical remains as they can keep their chemical activity in water and despite their small concentrations cause serious negative effects on animal and human health causing worldwide concern [3-8].

Studies show that among the pollutants found in water medications delivered to the surface [9], waste, and drinking water from public sectors, hospitals, veterinary clinics, pharmaceutical and biotechnological industries (as a result of incomplete utilization or partial neutralization of expired medications) are the main contributors. They are also accumulated from poultry and animal husbandry causing serious problems for the environment [10, 11].

Pharmaceuticals are considered to be a new type of stable organic pollutants [12]. They support the development of antibiotic-resistant bacteria in surface and underground waters [13]. The ways pharmaceuticals affect wildlife and ecosystem is largely unknown [14].

Studies concerning the existence of modi-cations in different types of water have determined a list of drugs most commonly discovered in water, NSAID (nonsteroidal anti-inflammatory drugs): paracetamol, aspirin, diclofenac, ibuprofen, etc. [3, 4, 15,16].

From the above-mentioned medications paracetamol (acetaminophen) is the most widely used one. It is considered to be a harmless drug (only being harmful if ingested in high dosages). Moreover, many commonly used medications contain paracetamol in different formulations as their main ingredient [17, 18].

Paracetamol is one of the pharmaceutical products most often detected in sewage treatment plant effluents, surface, and drinking waters ( 58-68% of pure paracetamol is excreted from the human body during therapeutic use) [19, 20]. It is one of the most frequently detected pharmaceutical products found in approximately 75% of natural waters such as rivers and lakes. Paracetamol is detected in wastewater at different concentrations throughout the world, with the recorded range between 0.025 mkg and 43.233 mkg [21-23].

Conventional wastewater treatment (chlorination, UV radiation, electrolysis, membrane bioreactor, etc.) no longer provides a sufficient level of purification from pollutants [24-26]. Therefore, residual quantities remain in treated water and have been accumulating in drinking water [27]. Due to this reason, many studies address this issue in 2 directions: improving the effectiveness of already existing purifying mechanisms or developing completely new methods [28].

Among the physical and chemical methods of detoxification of both gas and liquid media, adsorption stands out, as a universal process that allows almost complete removal of toxic impurities. The successful solution to many practical problems is determined by the choice of sorbents with optimal physical and chemical properties and cost for these purposes [29-34]. Currently, the most versatile sorbents are materials made from chemically activated carbon [35-37].

Studies have shown that carbon materials due to good sorption properties, high porosity, developed surface, mechanical strength, and antimicrobial properties can be considered as an alternative means of disinfecting water and neutralizing toxic substances. [38-40].

Wastewater treatment is mostly considered an added expense; hence, the cheapest process is always selected. Industries, as well as communities, are continuously struggling to adopt economical and robust treatment methodologies [41-43]. Therefore, the investigation of cheap and available adsorbent and economical routes for the subtraction of paracetamol is the objective of this research.

## Experimental part

The material used was nectarine kernel, from which the sorbent was prepared by a technology developed [44, 45] at the Institute of Inorganic Chemistry and Electrochemistry of the Ivane Javakhishvili Tbilisi State University. This technology was used for many types of raw materials (used tires, hazelnut and walnut shells, nectarine kernel, sawdust, plastics, etc.) [46, 47]. The process is single-stage and does not require preliminary treatment of raw materials. Carbon dioxide, water, and various gases generated in the process are used as reagents and energy sources [46, 47]. In this case, the reactor vessel (stainless steel) acts as a catalyst. Using this technology, carbon materials were obtained from recycled organic waste, which are cheap and efficient. The physical properties of the carbon material were characterized by Micrometrics GEMINI VII 2390T surface area analyzer. The chemical composition and morphology of obtained carbon materials were determined by scanning electron microscope (SEM) HITACHI TM 3030 Plus. The measurement results are shown in Tables 1- 2.

Adsorption investigations were carried out to obtain the optimal conditions with multiple swings of process variables. The experiments were performed employing contact time (5–120 min), adsorbent particle size (50–200 µm), adsorbent dose (0.02– 0.2 g), solution concentration (10-100 mg/L), and pH (2-10). The observation was carried out under static conditions at a constant temperature of 25$^o$C. The solution was separated from the sorbent by filtration. After filtration, the amount of paracetamol was determined by spectrophotometer Zuzi 4201/50 in 243 nm.

The amount of adsorbed paracetamol was calculated according to the mass balance [48]:

$$q = \frac{(C_o - C_e)V}{W}, \qquad (1)$$

where, q is the adsorption value, mg/g; V is the volume of solution, L; $C_0$, $C_e$, are initial and equilibrium concentrations, mg/L; W is the mass of adsorbent, g.

The rate of extraction (S, %) of paracetamol by the sorbent was calculated by the formula [48]:

$$S=\frac{(C_o-C_e)}{C_o}x100\% , \qquad (2)$$

Where, S is rate of extraction, %; $C_0$, $C_e$, are initial and equilibrium concentrations, mg/L;

## Results and discussion

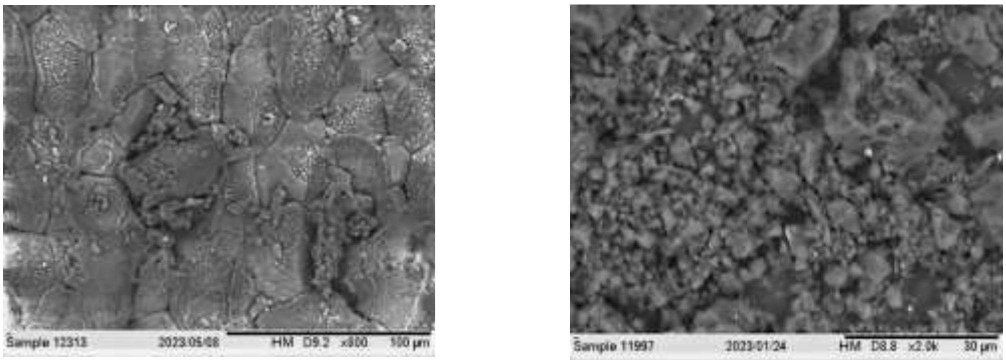
In the adsorption process by activated carbon a porous texture is required pore width of which is greater than the size of the drug molecule [8].

The adsorption capacity of carbonaceous materials is related to chemical and textural characteristics. Therefore, the presence of a well-developed pore distribution is very important. Also, the size of the micropores should be large enough to accommodate the adsorbate molecule [8]. The values of the textural parameters of carbon materials obtained from the nectarine kernel are shown in Table 1. These values were obtained from the adsorption isotherms of $N_2$ at –196◦C ($S_{BET}$, specific surface, micropore volume, micropore area) (Table 1).

**Table 1.** Textural parameters of the carbonaceous material

| Sample | $S_{BET}$ ($m^2 g^{-1}$) | Micropore area ($m^2 g^{-1}$) | Micropore volume ($cm^3 g^{-1}$) |
|---|---|---|---|
| Carbon material obtained from nectarine kernel | 651 | 537.25 | 0.26 |

The Scanning Electron Microscopy (SEM) images of the raw nectarine kernel and produced carbon material are shown in Fig. 1 A and B, respectively. Before treatment, the nectarine kernel shows a bulk structure with thick and large sheets as displayed in Fig. 1A. Powder produced after thermal treatment and grinding shows discrete sheets and carbon structures with enhanced surface area (Fig. 1B). The Chemical composition of the above-mentioned materials is given in Table 2. Therefore, the developed structure is favorable for adsorption.



A B

**Fig. 1.** Structure of (A) raw nectarine kernel and (B) carbonaceous material obtained from nectarine kernel

**Table 2.** Chemical composition of the raw nectarine kernel and carbonaceous material obtained from it

| Sample | Chemical composition of samples % (averaged) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | C | O | Ca | K | Si | S | Fe | Ni | Cu | Zn | Al | Pb | Cr |
| **Raw nectarine** | 50.60 | 47.01 | 0.49 | 0.90 | 0.07 | 0.14 | 0.44 | _ | _ | _ | 0.35 | _ | _ |

| | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **kernel** | | | | | | | | | | | | | |
| **Carbonaceous material obtained from nectarine kernel** | 93.09 | 5.63 | 0.16 | 0.55 | – | – | 0.30 | – | – | – | 0.27 | – | – |

### Effect of Particle Size

Adsorption is a surface phenomenon and adsorption efficiency is strongly affected by the particle size of the adsorbent, similar to other surface-induced reactions [49, 50]. Generally, the adsorption efficiency is inversely proportional to the particle size of the adsorbent material [51]. The smaller the particle size, the greater the efficiency of adsorption because of the particles with a small size have a larger capacity to adsorb the drug due to their larger total surface area [49]. The size of particles of the adsorbent was varied from 50 µm to 200 µm to check the effect of particle size on paracetamol removal efficiency. The results are shown in Fig. 2. The best result was reached by the particle size of 63 µm with 98% removal efficiency. Results elucidated that adsorption efficiency increases with a decrease in particle size up to a certain limit and then remains constant after achieving the peak value (Fig. 2). Hence, the particle size of 63 µm was selected as the optimum size and used in the rest of the experiments.

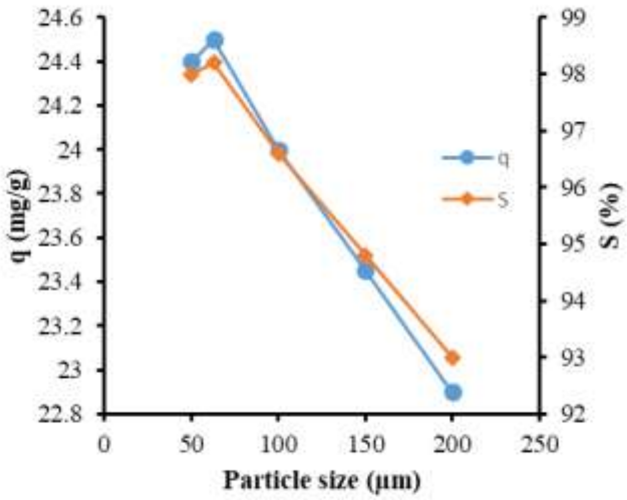



**Fig. 2.** Effect of particle size on adsorption of paracetamol on carbonaceous material obtained from nectarine kernel. Experimental conditions: Temperature – 25$^o$C, $C_0$ – 50 mg $L^{-1}$, $m_{ad}$ – 0.1g, time – 30 min, V – 50 mL, particle size –50-200 µm

### Effect of Retention Time

Adsorption is a time-based process and the impact of time can be positive or negative depending upon the process dynamics [50]. This experimental evidence provides us with the information about the effect of contact time on the adsorption of paracetamol. Contact time varied from 5 to 120 min. The obtained results are provided in Fig. 3.

In Fig. 3, it is observed that adsorption takes place at a very fast rate during the first 30 minutes, because in the first stage, the sorbate molecules are being adsorbed onto a surface where there are no other such molecules already attached, and consequently the sorbate-sorbate interactions are negligible [52]. Therefore, paracetamol molecules reach the adsorption sites easily. After some time, this is followed by a slower rate of adsorption (120 min) before equilibrium is attained, since the number of free sites on the carbon material decreases, and the non-adsorbed molecules are assembled at the surface, thus limiting the capacity of adsorption.

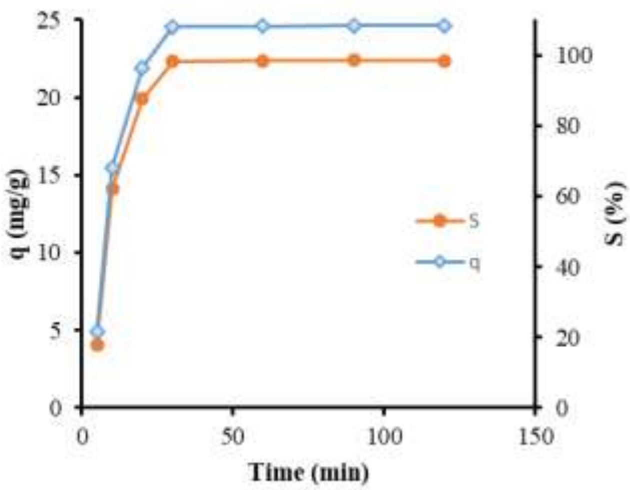



**Fig. 3.** Effect of retention time on adsorption of paracetamol on carbonaceous material obtained from nectarine kernel. Experimental conditions: Temperature – 25°C, $C_0$ – 50 mg $L^{-1}$, $m_{ad}$ – 0.1g, time – 5 - 120 min, V – 50 mL, particle size –63µm.

30 minutes was chosen as the optimal retention time instead of 90 minutes since the difference between the adsorption values in the two was very trivial (0.75%). Therefore, the rest of the experiments were carried out in 30 minutes to reach the greatest effect in the least amount of time.

**Effect of Amount of Adsorbent**

Provision of the adsorbent required proper selection because the availability of active adsorption sites and active surface area is a crucial parameter that significantly affects the removal efficiency [53]. The effect of adsorbent dose on the sorption of paracetamol was investigated by ranging the masses of the adsorbents between 0.02 g to 0.2 g, while keeping the rest of the parameters constant ($C_0$ – 50 mg $L^{-1}$; time – 20min; pH – 2.13, temperature - 25°C). The results show that sorption efficiency (S%) and adsorption capacity (q) varied inversely (Fig. 4). An increase in sorption efficiency with an increase in sorbent dose was recorded whereas adsorption capacity decreased from 75.0 mg $g^{-1}$ to 12.02 mg $g^{-1}$.

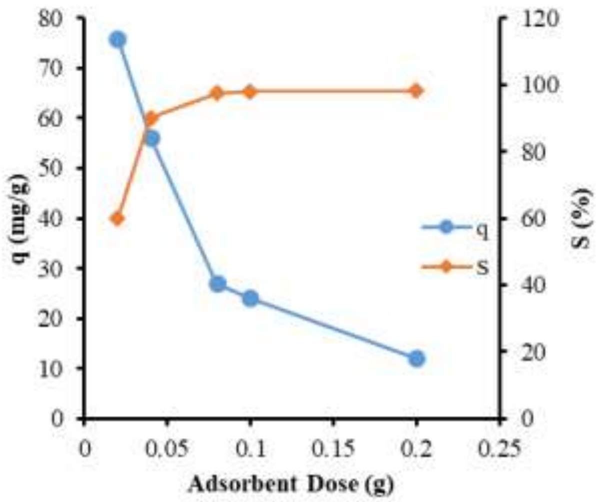



**Fig. 4.** Effect of adsorbent dose on adsorption of paracetamol on carbonaceous material obtained from nectarine kernel. Experimental conditions: Temperature – 25°C, $C_0$ – 50 mg $L^{-1}$, $m_{ad}$ – 0.02 – 0.2g, time – 30 min, V – 50 mL, particle size –63µm.

An increase in the amount of adsorbent increased the number of active sites which is reciprocated in the value of S% by increasing it. This is explained by the fact that, all the sites are not readily available for binding the drug molecules, thus overlapping in between the sites occurs. As a result, drug uptake capacity (q) decreased with increasing adsorbent doses.

**Effect of pH**

Generally, system pH is an imperative parameter that significantly influences the process efficiency. Therefore, the effect of pH was investigated by fluctuating it from 2 to 10 while keeping all the other factors constant. High adsorption potential was observed from low to neutral pH (Fig. 5). The results elucidated the pH value of 6.5 to be the optimum pH having the highest removal efficiency (98.66%) and adsorption capacity (24.7 mg $g^{-1}$).

Adsorption of paracetamol depends on the solution pH. At acidic pH, the adsorption was maximum because paracetamol consists of substituted phenol (pKa = 9.38), which is most stable at around pH of 5-6. In basic area, the adsorption is observed to be lower as phenols are dissociated into anions.

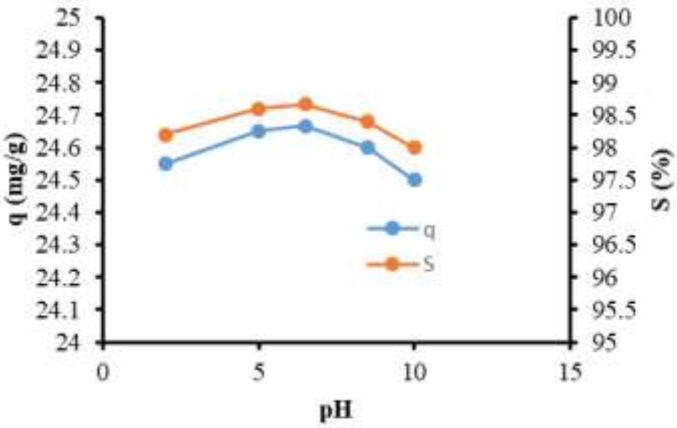



**Fig. 5.** Effect of pH on adsorption of paracetamol on carbonaceous material obtained from nectarine kernel. Experimental conditions: Temperature – 25°C, $C_0$ – 50 mg $L^{-1}$, $m_{ad}$ – 0.1g, time – 30 min, V – 50 mL, particle size –63µm.

**Effect of Concentration and Adsorption Isotherms**

In addition, the concentration effects were also studied. Fig. 6 shows the original adsorption isotherm, which reveals that the capacity at low concentration is larger than that at high concentration. Studies have shown that at low concentrations of paracetamol in the solution adsorption occurs almost completely in 30 minutes. Due to this phenomenon, in this case, the experiment was carried out for 5 minutes.

To investigate the adsorption equilibrium characteristics of carbonaceous material prepared from nectarine kernel at different concentrations (10-100mg $L^{-1}$) of paracetamol the two most common adsorption models namely Langmuir and Freundlich were built to explain the adsorption process.

The Langmuir and Freundlich models have been used in this study. The linear model of Langmuir isotherm and Freundlich isotherm are shown in Equations (3) and (4) respectively [54].

$$C_e/q_e = C_e/q_m + 1/K_L q_m \qquad (3)$$

$$\ln q = \ln(K_F) + \ln C_e/n. \qquad (4)$$

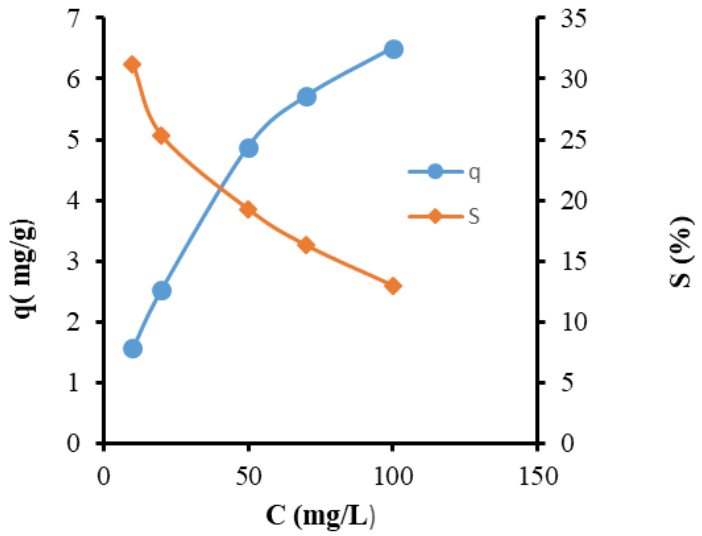
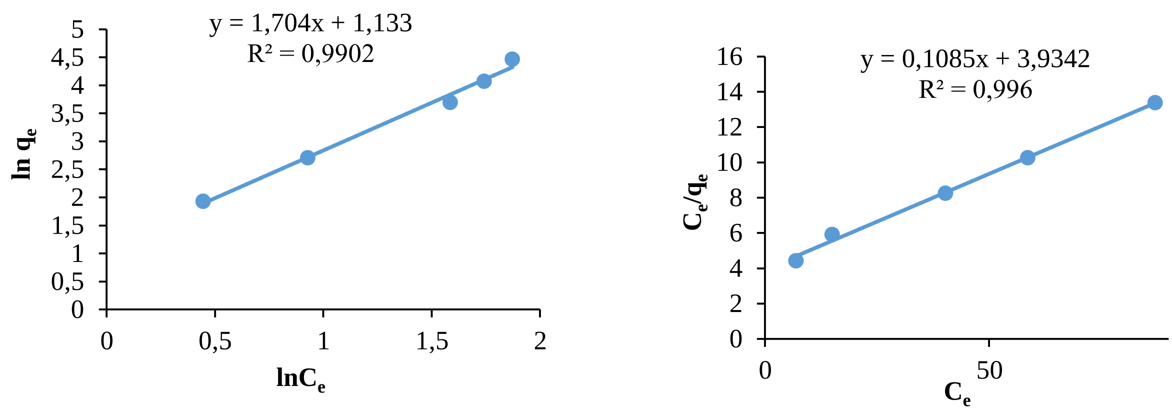



**Fig. 6.** Effect of concentration on adsorption of paracetamol on carbonaceous material obtained from nectarine kernel. Experimental conditions: Temperature – 25°C, $C_0$ – 10– 100 mg $L^{-1}$, $m_{ad}$ – 0.1g, time – 5 min, V – 50 mL, particle size –63µm.

where $q_e$ (mg/g) is the quantity adsorbed on the adsorbent at equilibrium, $C_e$ (mg $L^{-1}$) is the concentration at equilibrium, $q_m$ (mg $g^{-1}$) is the maximum sorption capacity relative to complete monolayer capacity, $K_L$(L $mg^{-1}$) is Langmuir constant, $K_F$ (mg $g^{-1}$) is distribution coefficient and tells the amount of drug adsorbed on sorbent for unit equilibrium concentration, and ‘‘n” is the adsorption intensity.

The adsorption isotherms relate the amount of paracetamol adsorbed at equilibrium $q_e$ (mg $g^{-1}$) to the paracetamol concentration at equilibrium, $C_e$ (mg $L^{-1}$), and the plots are given in Figure 7. The Langmuir isotherm for paracetamol adsorption is shown in Fig. 7A and Freundlich isotherm in Fig. 7B.

As it is shown on Figures 7A and B, the coefficients of correlation obtained are higher than 0.95 for the studied models. This suggests that the equilibrium of the adsorption of paracetamol on the carbonaceous material from nectarine kernel is adequately represented by the models of Langmuir and Freundlich.




**A** **B**

**Fig. 7.** Adsorbent equilibrium of paracetamol on carbonaceous material obtained from nectarine kernel (A) Langmuir isotherm, and (B) Freundlich isotherm. Experimental conditions: Temperature – 25°C, $C_0$ – 10–100 mg $L^{-1}$, $m_{ad}$ – 0.1g, time – 5 min, V – 50 mL, particle size –63µm.

## Conclusion

In this work naturally available nectarine kernel was used as raw material to obtain the carbonaceous material, which was used as an adsorbent for the removal of paracetamol from water. Results elucidated that the maximum amount of paracetamol that can be bound by 0.1 gram of sorbent from 50 ml solution is 98.6%. Isothermal studies showed that the adsorption of paracetamol on developed adsorbents can be explained by using Langmuir isotherm as well as Freundlich isothermal model. It is established, that low-cost, high-value carbonaceous material obtained from the nectarine kernel by the original technology exhibited good adsorption capacities without activation and modification and can be successfully used for the removal of pharmaceutical drugs from wastewater.

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