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ORIGINAL PAPER

# Removal of 4-chloro-2-nitrophenol occurring in drug and pesticide waste by adsorption onto nano-titanium dioxide

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**Abstract** The present study deals with removal of 4-chloro-2-nitrophenol (4C2NP) as a model contaminant from pharmaceutical and pesticide industries using titanium dioxide nanoparticles as an adsorbent. 4C2NP is recalcitrant and persistent toward biodegradation and its generation in aqueous environment during formulation, distribution and field application of pesticides is often unavoidable. Batch experiments were carried out to investigate the effect of contact time, nano-titanium dioxide dosage, initial pH, initial 4C2NP concentration and temperature on adsorption efficiency. The results showed that the adsorption capacity was increased with increasing 4C2NP concentration and temperature. Optimum conditions for 4C2NP adsorption were found to be initial pH ≈ 2, nano-titanium dioxide dosage ≈ 0.01 g/250 mL and equilibrium time ≈ 1 h. Titanium dioxide nanoparticles recorded a maximum capacity of 86.3 mg/g at optimal conditions. The linear correlation coefficients of Langmuir, Freundlich and Temkin isotherms were obtained. The results revealed that the Freundlich isotherm fitted the experimental data better than the other isotherm models.

**Keywords** Nanoparticle · Phenolic compounds · Adsorption isotherm · Wastewater

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## Introduction

Environmental pollution due to technological developments is one of the most important problems of this century. Industrial use of phenol and its derivatives over the past decades has led to serve environmental pollution. These compounds are toxic and carcinogenic, and they can persist for many years in the environment because of their resistance to microbiological degradation (Khan and Anjaneyulu 2005; Chen et al. 2009). Owing to their toxicity, their polluting effects on our echo-system provide possible human health risk. Chronic toxic effects due to phenols reported in humans include vomiting, difficulty in swallowing, anorexia, liver and kidney damages, headache, fainting and other mental disturbances (Calace et al. 2002). While the World Health Organization (WHO) has recommended the permissible phenolic concentration of 0.001 mg/L in potable waters, the European Union (EU) has set a maximum concentration level of 0.5 μg/dm$^3$ of total phenols in drinking water (WHO 1963; Dursun and Tepe 2005). Moreover, the maximum permitted concentration level of phenols being 0.5–1 mg/L for industrial wastewaters (Sokol and Korpal 2004). Thus, the removal of phenolic compounds from wastewater before its discharge, is necessary to reduce their side effects on the environment and human health. In the last decade, various methods have been proposed to remove phenols, including advanced oxidation process (Samarghandi et al. 2007; Saritha et al. 2007), biological degradation (Agarry and Solomon, 2008), ultrafiltration (Acero et al. 2005), ozonation (Benitez et al. 2000; Gharbani et al. 2010) and adsorption (Hashizume 2004; Roostaei and Tezel 2004; Mahvi 2008; Dabhade et al. 2009; Subramanyam and Das 2009; Belarbi and Al-Malack 2010). Since the other methods need special process requirements or special catalysts, adsorption seems to be a good choice in terms of cost and operation for removal of phenol and its derivatives.

Activated carbon (Gonzalez-Serrano et al. 2004; Kumar et al. 2007; Tseng et al. 2010), microporous and mesoporous carbon (Derylo-Marczewska et al. 2010; Haque et al. 2010), Zeolit (Wang et al. 2010) and clay minerals (Arellano-Cardenas et al. 2005) are a few of the adsorptive materials which have been tested for adsorption of phenols from aqueous solutions. In most cases, adsorbents have diameters in the range of submicron to micron and have large internal porosities to ensure adequate surface area for adsorption. However, the diffusion limitation within the particles leads to decreases in the adsorption rate and available capacity. Therefore, it is important and interesting to develop a novel adsorbent with a large surface area, small diffusion resistance and high capacity for adsorption. The relative large specific surface area of nanoparticles enables them to become candidate for adsorption of phenol and substituted phenols. Nanoparticles have high-adsorption capacity. In addition, the operation is simple and the adsorption process is rapid (Lei et al. 2010). Some researches have reported that nanosized inorganic oxides such as nano-$TiO_2$ could successfully remove phenolic compounds from contaminated water (Robert et al. 2000; Bekkouche et al. 2004).

In this research, efficiency and performance of nano-$TiO_2$ in adsorption of USEPA listed 4-chloro-2-nitrophenol (4C2NP), widely available in bulk drug and pesticide wastes, was studied. The study was carried out aiming at optimization of conditions for removal of 4C2NP from aqueous solutions by nano-$TiO_2$ adsorbent. Moreover, experimental data fitted various isotherm equations to determine the best isotherm to correlate the experimental data. The experiments were conducted in School of Pharmacy, Tabriz University of Medical Sciences, during 2010–2011.

## Materials and methods

Nano-$TiO_2$ (Degussa P25) was purchased from Degussa, Germany. Particle size and surface area of nanoparticles were about 20 nm and 15–50 $m^2/g$, respectively. 4C2NP ($C_6H_4ClNO_3$, $M_w = 173.56$ g/mol) was supplied by Fluka, Germany.

Adsorption of 4C2NP was carried out by a batch method to obtain equilibrium data. The variation of the 4C2NP concentration versus time in the aqueous solution was monitored under various conditions such as nano-$TiO_2$ dosage (0.005, 0.01, 0.02, 0.03, 0.04, 0.05 and 0.1 g/250 mL), initial pH (2, 4, 6, 8, 10 and 12), initial 4C2NP concentration (2, 4, 6, 8 and 10 mg/L) and temperature (25, 35, 45 and 55°C). A stock solution was prepared by dissolving the required amount of 4C2NP in ethanol (from Merck) and then diluting it to the appropriate concentration by double-distilled water (pH of stock solution was 5.7). The initial pH was adjusted by adding either HCl or NaOH. Adsorption was achieved by adding a known amount of nano-$TiO_2$ into 250 mL of 4C2NP solution of known concentration, pH and temperature, and the mixture was shaken in a shaking water bath at a speed of 140 shakes/min. Samples were taken at predetermined time intervals, centrifuged (Hettich/UNIVERSAL 16-R) at 10,000 rpm for 10 min and the analysis of 4C2NP remaining in the solution was done using a UV-160 Shimadzu spectrophotometer at a wavelength of 219 nm (acidic pHs) or 234 nm (neutral and alkaline pHs). 4C2NP concentration was calculated from the calibration curve. The calibration graph was constructed using several points as absorbance versus 4C2NP concentration in the range of 2–10 mg/L and the results evaluated by liner regression. The adsorbed 4C2NP amounts onto the $TiO_2$ nanoparticles (mg/g) were calculated from the following relation:

$$q_t = \frac{(C_0 - C_t)}{M} V \tag{1}$$

$$q_e = \frac{(C_0 - C_e)}{M} V \tag{2}$$

where $C_0$, $C_t$ and $C_e$ are the initials, at any time $t$ and equilibrium 4C2NP concentration (mg/L), respectively; $V$ is solution volume (L); and $M$ is nano-$TiO_2$ mass (g).

## Results and discussion

### Effect of contact time

To find out the optimum contact time, 0.1 g of nano-$TiO_2$ was added to 250 mL of 4C2NP solution with concentration of 10 mg/L at 25°C. The pH of solution was 5.7 and was not corrected. The mixture was shaken for 180 min and samples were taken at 10 min intervals (Fig. 1).

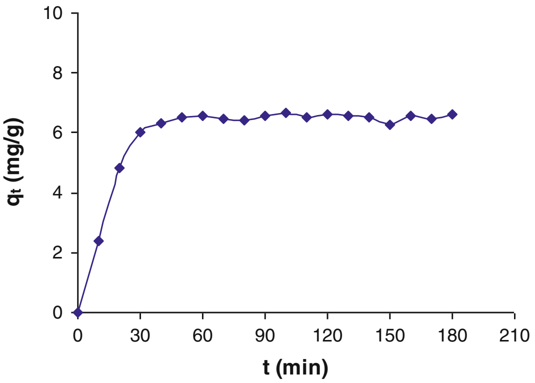

**Fig. 1** Effect of contact time on 4C2NP adsorption onto nano-$TiO_2$ (nano-$TiO_2$ dose: 0.1 g/250 mL, pH: 5.7, initial 4C2NP conc.: 10 mg/L, temp.: 25°C)

The removal of 4C2NP by adsorption onto nano-$TiO_2$ was found to be rapid at the initial period of contact time, and then slowed with the increase of contact time. This is obvious from the fact that a large number of vacant surface sites are available for adsorption during the initial stage and with passage of time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid phase and in the bulk liquid phase. Figure 1 also indicates that the time required for equilibrium is 1 h. Thus, for all equilibrium adsorption studies, the contact period was kept 1 h.

## Effect of nano-$TiO_2$ dosage

To study the effect of nano-$TiO_2$ dose on the adsorption of 4C2NP, the experiments were done under the conditions described at the previous stage with contact time of 1 h and variable nano-$TiO_2$ dose (0.005, 0.01, 0.02, 0.03, 0.04, 0.05 and 0.1 g/250 mL). Figure 2 shows the effect of nano-$TiO_2$ dosage on removal of 4C2NP. A trend of increment in adsorption capacity with increment in adsorbent dose was observed from 0.005 to 0.01 g/250 mL. Adsorbent recorded a maximum capacity of 77.78 mg/g at 0.01 g/250 mL dosage. The initial increment in adsorption capacity with an increase in adsorbent dosage was expected, since number of adsorbent particles increases, and thus, more surface areas were available. Further increment in adsorbent dosage beyond maximum adsorption capacity at 0.01 g/250 mL led to the decline in capacity, as shown in Fig. 2. This reduction in capacity had been explained as due to the aggregation of nano-$TiO_2$ particles, which reduced the interfacial area between the reaction solution and the adsorbent. However, similar observations can be found in literature. Saritha et al. (2007), who utilized $TiO_2$ powder to adsorb 4C2NP, suggested that the amount adsorbed (mg/g) decreased as the amount of the adsorbent increased. As a result, nano-$TiO_2$ dosage of 0.01 g/250 mL was used in the subsequent experiments of this work.

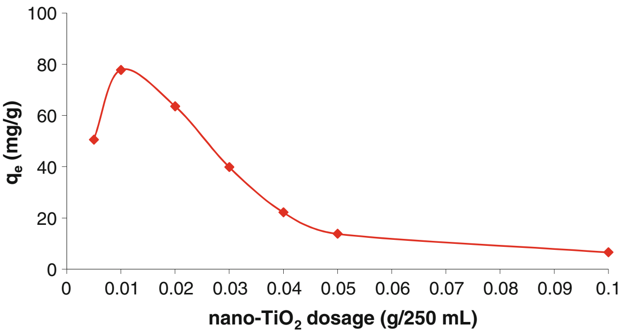

**Fig. 2** Effect of nano-$TiO_2$ dosage on 4C2NP adsorption (pH: 5.7, initial 4C2NP conc.: 10 mg/L, temp.: 25°C, contact time: 1 h)

## Effect of initial pH

In this study, understanding of pH was important, because, the pH of the solution influences the distribution of active sites on the surface of nano-$TiO_2$. To determination the effects of solution pH on adsorption capacity; 4C2NP adsorption was examined by adding different amounts of 0.1 M HCl or 0.1 M NaOH to obtain different pHs (i.e., 2, 4, 6, 8, 10 and 12). In Fig. 3, the effect of initial pH on the adsorption of 4C2NP onto nano-$TiO_2$ at 25°C and initial 4C2NP concentration of 10 mg/L is depicted. As shown, it can be deduced that the amount of 4C2NP adsorbed on nano-$TiO_2$ increased by decreasing pH and the highest amount of 4C2NP adsorption was at pH 2 (86.3 mg/g).

The pH solution controls the electrostatic interactions between the adsorbent and the adsorbate (Anbia and Ghaffari 2009). The adsorbent surface charge is neutral at isoelectric point (IEP), where the $pH_{IEP}$ for the Degussa P25 $TiO_2$ is ranged between 6.2 and 6.9 (Bourikas et al. 2005). While the surface of the adsorbent carries positive charge at pH values lower than IEP, it has negative charge at pH values higher than IEP (Lei et al. 2010). At low pH of 6, nano-$TiO_2$ surface is positively charged and since there is no electrostatic repulsion between the unionized 4C2NP species and the positively charged surface, the adsorption is higher. On the other hand, for $pH > pK_a$ (4C2NP, $pK_a = 6.46$), the phenols dissociate, forming phenolate anions, while the surface of nano-$TiO_2$ is negatively charged. The electrostatic repulsion between the identical charges lowers the adsorption capacities. Besides, the phenolate anions are mostly soluble in the aqueous solution, and stronger adsorbate-water bonds must be broken before adsorption can take place (Terzyk 2003). Similar result was reported for adsorption of reactive red 195 on $TiO_2$ nanoparticles (Belessi et al. 2009). Thus, the rest of the experiments were performed at pH 2.

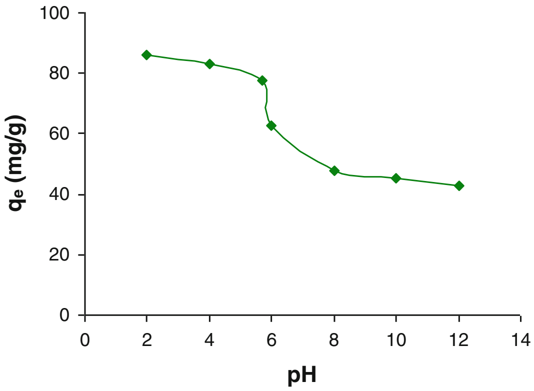

**Fig. 3** Effect of pH on 4C2NP adsorption onto nano-$TiO_2$ (nano-$TiO_2$ dose: 0.01 g/250 mL, initial 4C2NP conc.: 10 mg/L, temp.: 25°C, contact time: 1 h)

## Effect of initial concentration

The effect of initial 4C2NP concentration in the range of 2–10 mg/L on the adsorption rate of 4C2NP onto nano-$TiO_2$ is shown in Fig. 4. As shown, when the initial 4C2NP concentration is increased from 2 to 10 mg/L the amount of 4C2NP adsorbed per unit weight of the nano-$TiO_2$, at equilibrium conditions and the constant temperature as 25°C, increased from 6 to 86.3 mg/g. This is obvious from the fact that the initial 4C2NP concentration provides an important driving force to overcome the whole mass transfer resistance. In addition, the increase of the initial concentration of 4C2NP for the same mass of nano-$TiO_2$ (0.01 g/250 mL) creates a great increase of molecules of 4C2NP in the solution. Thus, higher interaction occurred between 4C2NP and $TiO_2$ nanoparticles (Bekkouche et al. 2004).

## Effect of temperature

To study the effect of temperature, 0.01 g of nano-$TiO_2$ was added to 250 mL of 4C2NP solution with concentration of 10 mg/L at pH 2 and the experiments were performed at temperatures of 25, 35, 45 and 55°C. Based on results (Fig. 5), the adsorbed amount of 4C2NP on nano-$TiO_2$ increases with the increasing temperature. In fact, the temperature has two important effects, which are known to increase the diffusion rate of the adsorbate molecules across the boundary layer and in the internal pores of the adsorbent particle, owning to the decrease in viscosity of the solution (Onal et al. 2007).

## Adsorption isotherm studies

Adsorption isotherms are important to describe how solutes interact with adsorbent. Adsorption isotherm studies were carried out with different initial 4C2NP concentrations ranging from 2 to 10 mg/L. Equilibrium adsorption isotherm data were analyzed according to the linear forms of Langmuir, Freundlich and Temkin adsorption isotherm equations (3–5), respectively:

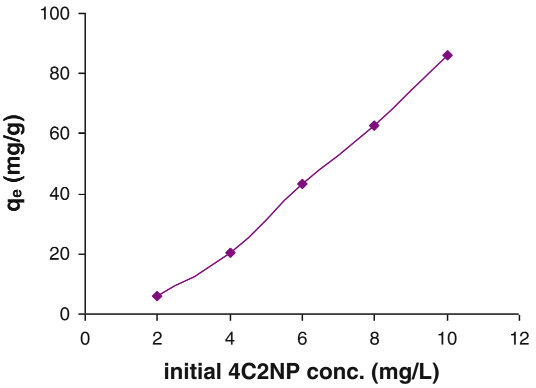

**Fig. 4** Effect of 4C2NP initial conc. on its adsorption onto nano-$TiO_2$ (nano-$TiO_2$ dose: 0.01 g/250 mL, pH: 2, temp.: 25°C, contact time: 1 h)

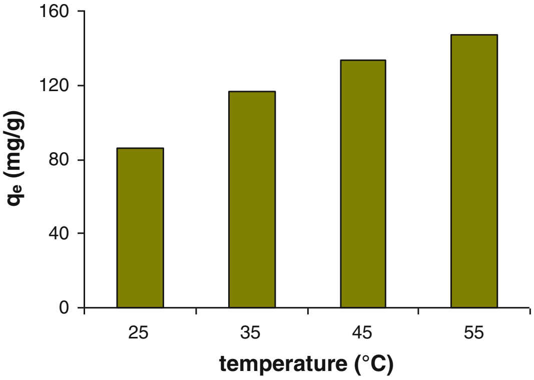

**Fig. 5** Effect of temperature on 4C2NP adsorption onto nano-$TiO_2$ (nano-$TiO_2$ dose: 0.01 g/250 mL, pH: 5.7, initial 4C2NP conc.: 10 mg/L, contact time: 1 h)

$$\frac{1}{q_e} = \left(\frac{1}{K_L q_m}\right)\frac{1}{C_e} + \frac{1}{q_m} \tag{3}$$

$$\mathrm{Ln}q_e = \left(\frac{1}{n}\right)\mathrm{Ln}C_e + \mathrm{Ln}K_F \tag{4}$$

$$q_e = B_1\mathrm{Ln}C_e + B_1\mathrm{Ln}K_T \tag{5}$$

where $C_e$ (mg/L) is the equilibrium concentration of 4C2NP, $q_e$ (mg/g) is the amount of 4C2NP adsorbed at equilibrium, $q_m$ (mg/g) is the maximum adsorption at monolayer and $K_L$ (L/mg) is the Langmuir constant including the affinity of binding sites. $K_F$ [(mg/g)(L/mg)$^{1/n}$] and $n$ are the Freundlich constants indicating adsorption capacity and intensity, respectively. $K_T$ (L/g) and $B_1$ are the Temkin constants ($K_T$ is the equilibrium binding constant and $B_1$ is related to the heat of adsorption). The values of Langmuir, Freundlich and Temkin parameters were calculated from the slope and intercept of linear plots of $1/q_e$ versus $1/C_e$, Ln$q_e$ versus Ln$C_e$ and $q_e$ versus Ln$C_e$, respectively. Figure 6a–c displays the adsorption isotherms plots. The isotherm constants along with the correlation coefficients are listed in Table 1.

It can be seen from the Table 1 that the adsorption process could be explained by all models from comparing the results of the correlation coefficient values. However, careful observation may describe Freundlich isotherm better than others. This means that heterogeneous occupation of the surface may be predominated and also physisorption occurs rather than chemisorption (Nandi et al. 2009). The Freundlich parameters, $K_F$ and $n$ were found to be 2.013 and 0.497, respectively. A relatively $n \ll 1$ indicates that adsorption intensity is favorable over

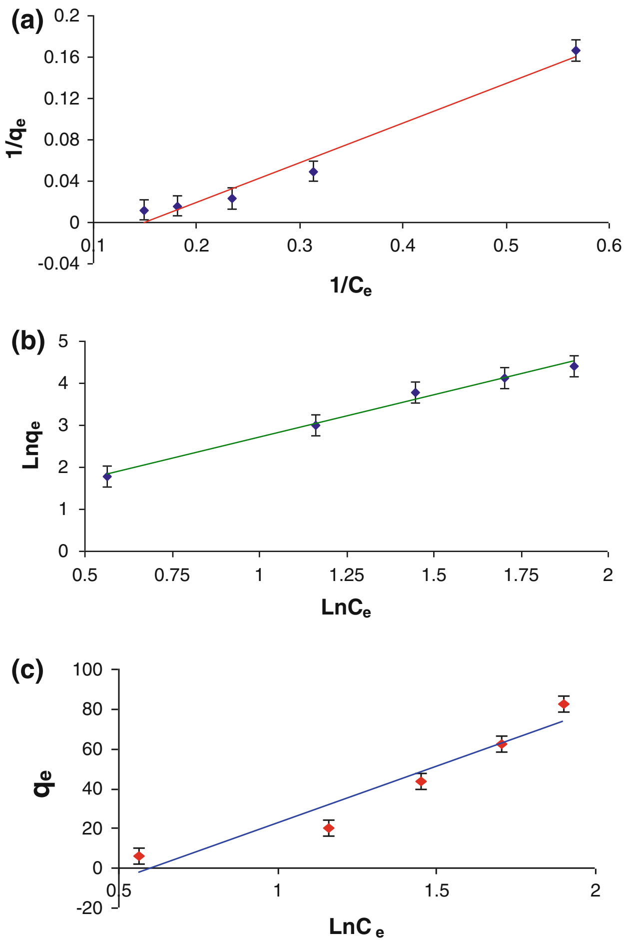

**Fig. 6** Plots of linearized Langmuir (**a**), Freundlich (**b**) and Temkin (**c**) adsorption isotherms

**Table 1** Isotherm parameters for 4C2NP adsorption onto nano-$TiO_2$ (nano-$TiO_2$ dose: 0.01 g/250 mL, pH: 2, temp.: 25°C, contact time: 1 h)

| Langmuir model | | | Freundlich model | | | Temkin model | | |
|---|---|---|---|---|---|---|---|---|
| $q_m$ | $K_L$ | $R^2$ | $n$ | $K_F$ | $R^2$ | $B_1$ | $K_T$ | $R^2$ |
| 17.543 | 0.149 | 0.972 | 0.497 | 2.013 | 0.991 | 56.979 | 0.547 | 0.922 |

the entire range of concentrations studied, while $n > 1$ means that adsorption intensity is favorable at high concentrations, but much less at lower concentrations (Hameed et al. 2007). Therefore in the present study, adsorption intensity is favorable over the entire range of concentrations studied.

## Conclusion

This investigation examined the adsorption of 4-chloro-2-nitrophenol onto titanium dioxide nanoparticles (Degussa P25) at various conditions. It was found that a trace amounts of nano-$TiO_2$ (0.01 g/250 mL) shows significant adsorption for 4C2NP. This may be due to the high specific surface area of nanoparticles and the absence of internal diffusion resistance. The results suggested that the adsorption capacity decreased as the pH rose, but increased with an increase of initial 4C2NP concentration and temperature. The Freundlich isotherm model was found to be the best fitting isotherm model and this means that heterogeneous occupation of the surface may be predominated and also physisorption occurs rather than chemisorption.

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