



Removal of hinosan from underground water using NH₄Cl-modified activated carbon from rice husk

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Abstract

In the present study, NH₄Cl-modified activated carbon was synthesized from rice husk and used as an adsorbent for removal of hinosan from underground waters. The effect of some effective parameters on the adsorption of hinosan on the rice husk NH₄Cl-modified activated carbon (RHNAC) like pH, adsorbent dose, contact time, and temperature was evaluated in batch mode and the optimum conditions were determined. Kinetic of adsorption was studied by Langmuir and Freundlich's models. The equilibrium data were well fitted to the Langmuir isotherm model, and the maximum adsorption capacity of hinosan on RHNAC based on the Langmuir isotherm model was 81.366 mg g⁻¹. The experimental adsorption data had the best fitness with the pseudo-second-order kinetic model. The applicability of the prepared adsorbent (RHNAC) was compared with other activated carbons (ZnCl₂-modified activated carbon was prepared from rice husk and industrial activated carbon). The obtained results which were calculated from the selected adsorbents showed more desirability for RHNAC as an adsorbent. So, RHNAC could be introduced as an effective and cost-effective adsorbent for removal of hinosan from underground waters.

Keywords Hinosan · NH₄Cl-modified activated carbon · Rice husk · Adsorption · Underground waters

Introduction

Hinosan (edifenphos; O-ethyl S, sdiphenylphosphodithiate) is one of the most usable organophosphate pesticides for pest control in agriculture (Fattahi et al. 2010). Hinosan was sold by Bayer AG in 1968 to control rice blast disease (pyriculariaoryzae) (Scheinflug and Jung 1968). Fungal

diseases cause serious damages to farm products; hinosan and related phosphorous compounds such as p-chitosan are extensively utilized as fungicides. The residues of these toxic chemicals enter agricultural wastewater and slowly contaminate underground waters through penetration (Takase et al. 1973; Rijtema and Elias 2012).

Agricultural sewage is one of the worldwide environmental problems. They usually discharge to water sources and pollute them. Phosphorous compounds enter water sources through this way. Studies have proved that hinosan and related phosphorous compounds can have a deleterious effect on aqueous systems and human health (Rotich et al. 2003). According to the World Health Organization (WHO), hinosan is categorized at level II of toxicity (BALLANTYNE and SALEM 2006). This compound is used as rice blast in northern parts of Iran (Guilan Province). In Guilan province, it is used in liquid form, and it is classified as the most dangerous pesticides.

Organophosphorus insecticides can adversely affect the nervous system of insects and mammals (de Oliveira et al. 2018). Also, phosphorous compounds can cause the bad taste, odor, and color of water (Rastogi et al. 2008). The maximum allowed concentrations for individual or total pesticides in drinking water are 0.10 and 0.5 µg L⁻¹, respectively. Hence, a low-cost adsorption process with high performance for the

Highlights • Herein, activated carbons have been prepared from rice husks.

- Removal of hinosan from water was performed using activated carbons.
- NH₄Cl-modified activated carbon exhibited good adsorption capacity for hinosan.
- The method is simple, fast, and effective for removal of organophosphorus pesticides from water sources.

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decontamination of water is needed. Some fundamental methods for elimination of pollutants from water are namely photo-catalytic decomposition (Shawaqfeh and Al Momani 2010), oxidation (Lafi and Al-Qodah 2006), electrochemical decomposition (Samet et al. 2010), membrane filtration (Ehrampoush et al. 2017), and adsorption (de Souza et al. 2018; Teli and Nadathur 2018). Among the various methods, adsorption process gained much attention because it is an effective technique and has prominent advantages like cost-effectiveness, good applicability, and convenience. Besides, the range of materials that were applied as adsorbents is wide. Among the introduced adsorbents to adsorption, activated carbon is a suitable choice for this purpose and is frequently applied for water treatment because of a high surface area and complex pore structures. Moreover, the adsorption process by activated carbon has some advantages like low price, simple operation, and lack of sludge residue (Nourmoradi et al. 2018; Ravulapalli and Kunta 2018).

The pore size, surface area, and mineral matter content of carbon are the effective feature that determine the adsorption performance of carbon. The size of carbon is an effective characteristic that determines the adsorption capacity. By decreasing the size of carbon, the accessibility of the organic molecules to the inner surface of the adsorbent increases. For example, at experimental conditions, small molecules can access micropores, natural organic matter (NOM) can access mesoporous, and bacteria can access macropores (Moreno-Castilla 2004).

Almost any carbonaceous material such as coconut shell (Xu et al. 2013), wood and coal dust (Tahermansouri et al. 2013), rice husk (Muniandy et al. 2014), hazelnut husk (Aydemir et al. 2012), nut shell (Lataye et al. 2008), and tea industry waste (Kan et al. 2017) can be used for the readiness of activated carbon. Rice husk has recently gained much attention as a cheap source for this purpose (Liu et al. 2011). As the main byproduct of the rice milling industry, rice husk accounts for about 20% of all rice products. Thus, a large quantity of husk is available as a waste product so that proper utilization of husks helps the disposal problem be solved.

In order to improve the performance of activated carbon samples, they have been modified through different methods such as chemical treatments by potassium hydroxide, zinc chloride, sulfuric acid, and sodium hydroxide; in these methods, irregular-shaped pores and closed structure may reduce adsorption capacity and slow down adsorption kinetics. In recent published articles (Yaghmaeian et al.; Moussavi et al. 2013b), using ammonium chloride as chemical treatment was introduced as an effective method for chemical modification of activated carbon. In this procedure, the parallel and long channels in the activated carbon structure were formed because of the explosive properties of ammonium, which improve the availability of active sites on the surface of carbon for adsorption process (Alahabadi et al. 2016). Formation of

regular-shaped pores in this method besides high carbon content can make large graphitic sheets, which can cause strong interactions such as π - π electron donor-acceptor and make them an effective adsorbent for highly aromatic pollutants (Li et al. 2002; Jing et al. 2014).

In the present study, modified activated carbons were prepared from rice husk by two different methods (NH_4Cl and ZnCl_2) and their applicability was compared with each other and industrial activated carbon. The results show that NH_4Cl -modified activated carbon is an efficient adsorbent for the elimination of organophosphorus pesticide from underground waters. In this investigation, hinosan was selected as a model compound from organophosphorus pesticides. The kinetics of adsorption was investigated, and Langmuir and Freundlich isotherm models were applied to survey the thermodynamic behavior of adsorption.

Experimental

Materials

Hinosan was prepared from Bayer Company, and its 50% w/w stock solution was made by dissolving the appropriate amount of edifenphos (CAS No. 17109-49-8) in HPLC-grade methanol. Extruded activated carbon (EAC) in size range (within the range) of 200 to + 1000 μm was purchased from Merck. To study the adsorption process, distilled water was redistilled in the presence of KMnO_4 (0.05 M) to remove all organic compounds. Real samples were collected from the wells in rural areas of Guilan province.

Methods

To determine pH values, an Aqualytic pH meter (model AL 15) with a combined double junction glass electrode, calibrated against two standard buffer solutions at pH 4.0 and 7.0, was used. A GC (Agilent 6890 gas chromatograph equipped with a DP-5 column (30-m length, 0.32-mm internal diameter, 0.25-mm film thickness from J&W Scientific, Inc., Folsom, CA) with electron capture detector (GC-ECD) was used for determination of hinosan (U.S. EPA method (8141-B 2000)). During the analysis of hinosan, the column temperature was held at 100 °C for 1 min and then raised to 265 °C at a rate of 5 °C min^{-1} , and Eld for 5 min at 265 °C. N_2 (99.99%) was used as carrier gas with the flow rate of 5 mL min^{-1} . The temperature of the detector was 300 °C, and makeup gas was N_2 with a flow rate of 40 mL min^{-1} . A BET surface analyzer (SA-1100 manufactured by Sibata Scientific Technology Ltd., Japan) was used for measuring the surface area of the adsorbent. Fourier transform infrared spectra (4000–400 cm^{-1}) in KBr pellet were recorded on a Bruker IFS-66 FT-IR spectrophotometer (Bruker optics, Karlsruhe, Germany)

Preparation of modified activated carbon from rice husk

Rice husks were obtained from local rice-milling factories and washed with double-distilled water several times to remove contaminants and then dried in the oven at 105 °C for 12 h. The general procedure of the activation process in this study is described by Taker et al (Teker et al. 2009). Briefly, 25 g of dried rice husk was activated and mixed with 250 mL of 10% (w/w) ZnCl₂, and the resulting precipitates were dried in the oven at 110 °C.

The NH₄Cl-modified activated carbon (RHAC) was synthesized according to the method that was introduced by Moussavi et al. (2013a, Yaghmaeian et al., 2014). Briefly, the samples were then soaked in the 2.0 wt% NH₄Cl solution and shaken for 24 h at room temperature. The mixture was then sieved to separate the char, and the separated char was then oven-dried at 105 °C for 24 h. Pre-treated char was finally activated by oven-heating at 800 °C and activation for 2.5 h under N₂ atmosphere. The produced adsorbent is a mesoporous material with hydroxyl, aliphatic C–H, C=O, and carboxylic and carbonyl functional groups on its surface (Moussavi et al. 2013b).

The surface area of the RHAC (which is activated by ZnCl₂), industrial activated carbon (EAC, which purchased from Merck), and NH₄Cl-modified rice husk activated carbon (RHNAC) was measured by BET and compared with each other. Also, the removal efficiencies of them at optimum condition were calculated and compared. The results summarized in Table 1, and the removal efficiency of RHNAC is about 20% higher than that of RHAC and EAC that it can be attributed to strong interactions such as π - π electron donor-acceptor between adsorbent and analyte.

Batch adsorption experiments

All experiments were carried out in an adsorption batch under stirring at the constant speed (700 rpm). To study the adsorption performance, 0.1 g of RHNAC was inserted into a 1.000 L of the hinosan at optimized pH (8.5), and the solution was stirred at 700 rpm for 120 min. After the completion of adsorption, cellulose acetate filter (pore size 0.2 μ m) was used for filtration the suspension. pH (2–12), contact time (30–300 min), solution temperature (30–80 °C), and adsorbent dose were the variable parameters that were studied.

Table 1 Physical characteristic of RHNAC, RHAC, and EAC and comparison between their applicability

Sample	Surface area (m ² g ⁻¹)	Mean pore diameter (nm)	Removal efficiency (%)
RHNAC	985	2.33	91.2
RHAC	830	2.84	73.5
EAC	750	2.91	70.8

Analysis

For investigating the residual hinosan, the filtrate was analyzed by GC-ECD. The same experiments were performed for EAC and RHAC for comparison. After the required time in batch experiments, the outlet solution after filtration has been extracted with 5 mL of n-hexane and then was injected to GC-ECD (1 μ L). Also, the outlet solution was analyzed for unabsorbed hinosan by GC-ECD (all determinations were carried out by the standard addition method). The capacity of the adsorbent was calculated according to the following Eq. (1):

$$Q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

where C_0 and C_e are the initial and equilibrium concentrations (mg L⁻¹), V is the volume of solution (L), and m is the mass of adsorbent (g).

Results and discussions

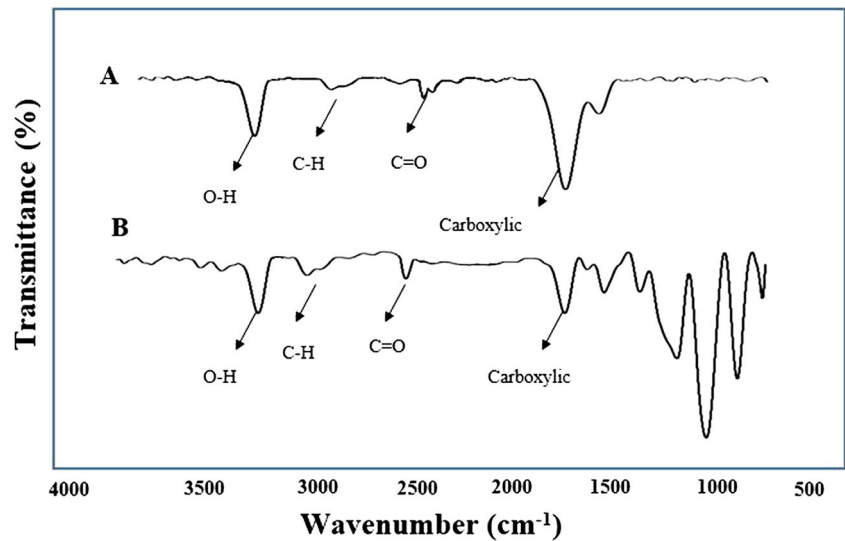
Activated carbon characterization

Figure 1 shows the FT-IR spectra of RHNAC (A) before and (B) after adsorption of hinosan. RHNAC shows an absorption band at wavenumber between 3200 and 3600 cm⁻¹ for the hydroxyl functional group. The absorption band at 2929 is attribute to aliphatic C–H, and adsorption band at wavenumber 2929 is attributed to C=O asymmetric stretching vibration of CO₂. After the adsorption, the hinosan characteristic peaks were added to RHNAC spectra. NH₄Cl-modified activated carbon can be an efficient adsorbent for pesticides containing aromatic ring and hydrogen bonding acceptor groups due to interaction through the physical π - π electron donor-acceptor and hydrogen bonding. So, it can be introduced as an effective adsorbent for organophosphorus pesticides and, in this study, hinosan was selected as a model compound from this group of pollutants.

Effect of pH

In this section, 0.1 g of RHNAC was added to 1.000 L of various samples with equal initial concentration of hinosan (180 μ g mL⁻¹) and different pH (2 to 12) for 2 h. Then the concentration of adsorbed hinosan was

Fig. 1 FT-IR spectra of RHNAC (A) before and (B) after adsorption of hinosan



measured. To the comparison, the same experiments were performed for RHAC and EAC. The obtained results are shown in Fig. 2a. According to the results, the adsorption of hinosan on RHAC and EAC is independent of pH because the adsorbent and hinosan cannot be protonated. But the removal efficiency increases in alkali media, when RHNAC was used as an adsorbent. It can be attributed to the deprotonation of adsorbent in alkali media and formation of the electron donor-acceptor interaction and hydrogen bonding between the analyte and adsorbent. So, the subsequent experiments

were performed in a weakly alkaline medium (pH = 8.5).

Amount of adsorbent

For this investigation, different dosages of the adsorbent (RHNAC) (0.02, 0.04, 0.06, 0.08, 0.1, and 0.12 g) were added to 1.000 L of hinosan sample with the concentration of 180 $\mu\text{g mL}^{-1}$ for 2 h, and the amounts of adsorbed hinosan were measured. To the comparison, the same experiments were performed for RHAC and EAC. Figure 2b shows that 0.1 g

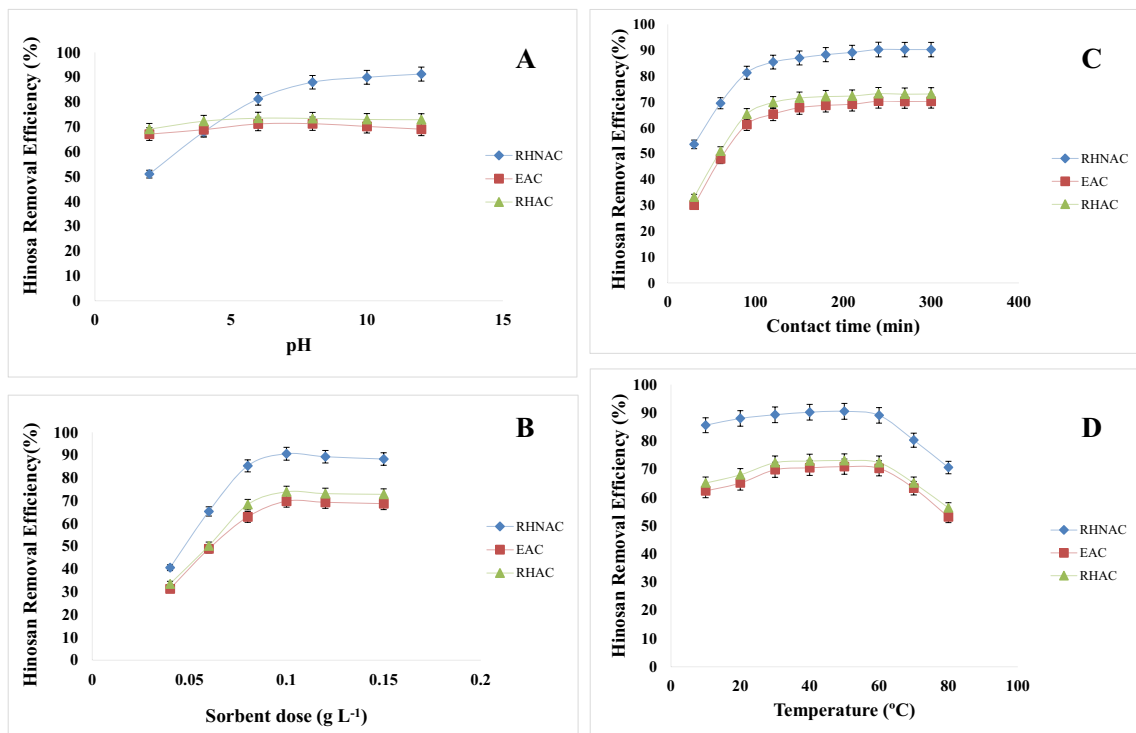


Fig. 2 Effect of a pH, b adsorbent dose (g L^{-1}), c contact time (min), and d temperature on adsorption of hinosan by RHNAC, RHAC, and EAC

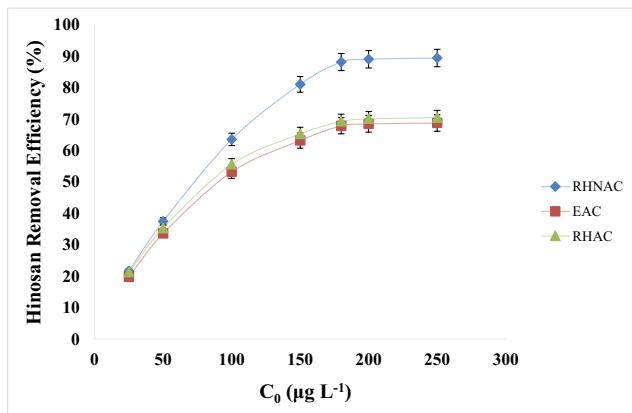


Fig. 3 Effect of initial concentration on adsorption of hinosan by RHNAC, RHAC, and EAC

of adsorbent is optimal (at working conditions) for removal of hinosan and after that, it will be almost constant.

Effect of contact time

In these experiments, 1,000-L sample of hinosan in a weakly alkaline medium with the concentration of $180 \mu\text{g mL}^{-1}$ was treated with 0.1 g of RHNAC for the different periods. For comparison, the same experiments were performed for RHAC and EAC. The obtained results (Fig. 2c) shown, the major part of the hinosan was removed after 2 h. Hence, 2 h was selected as optimum contact time in this research.

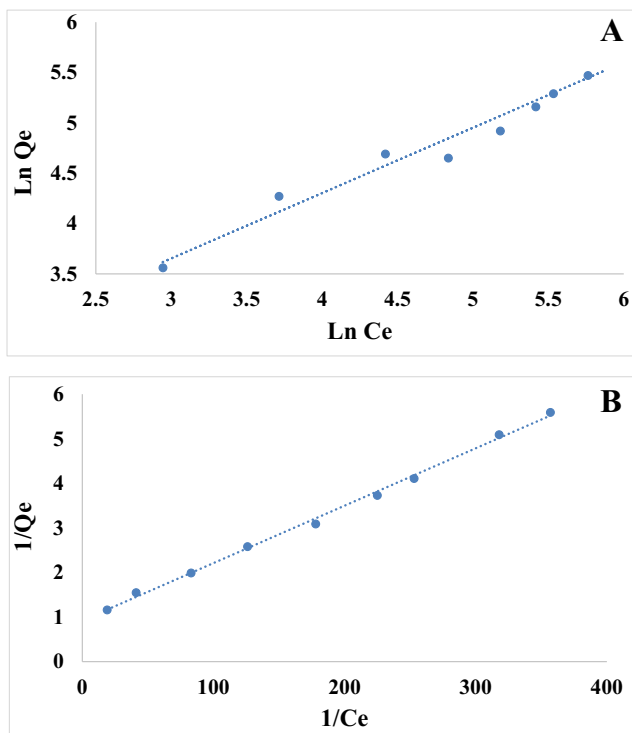


Fig. 4 **a** Modeling of adsorption Freundlich isotherm. **b** Modeling of adsorption Langmuir isotherm for removal of hinosan by RHNAC

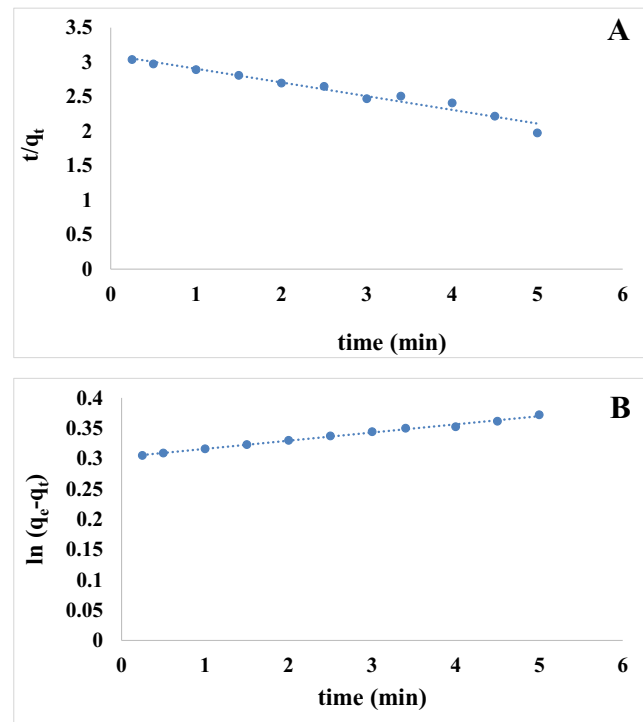


Fig. 5 **a** Lagergren's pseudo-first-order plot. **b** Pseudo-second-order plot of hinosan by RHNAC at the concentration level of $180 \mu\text{g mL}^{-1}$

Effect of temperature

In this investigation, 0.1 g of RHNAC was exposed to 1,000-L sample of hinosan in a weakly alkaline medium with the concentration of $180 \mu\text{g mL}^{-1}$ at different temperatures. For comparison, the same experiments were performed for RHAC and EAC. The obtained results (Fig. 2d) reveal, when the temperature increases from 10 to $50 \text{ }^\circ\text{C}$, the amount of adsorbed hinosan was increased and after that decreased which can be attributed to desorption at the higher temperature.

Effect of initial concentration and adsorption isotherm study

The equilibrium removal of hinosan was mathematically expressed regarding adsorption isotherms. The Langmuir and Freundlich models are the most common models and both models were studied in the present work. The theory of Langmuir adsorption isotherm suggests that the adsorption on adsorbent is monolayer and homogeneous. In this adsorption, the energy of adsorption is constant. The following equation was used (Azizian 2004) (Eq. (2)):

$$\frac{1}{Q_e} = \frac{1}{Q_{\max}} + \left(\frac{1}{Q_{\max} K_L} \right) \frac{1}{C_e} \quad (2)$$

where Q_e is the capacity of adsorbent (mg g^{-1}), C_e is the equilibrium hinosan concentration in solution (mg L^{-1}), Q_{\max} is the maximum adsorption capacity (mg g^{-1}), and K_L

Table 2 Adsorption equation parameters for RHNAC for removal of hinosan from water

Adsorbent	Langmuir			Freundlich		
	Q_{max} (mg g ⁻¹)	K_L (1 mg ⁻¹)	R^2	K_f	n	R^2
RHAC	81.366	0.0745	0.9974	3.5582	3.525	0.9605

is Langmuir adsorption constant, related to the free energy constant.

In the Freundlich isotherm, the adsorption is not restricted to a monolayer adsorption, and a heterogeneous system is suggested (Sotelo et al. 2014). Freundlich equation is represented below (3):

$$\ln Q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{3}$$

where Q_e is the equilibrium adsorption capacity of hinosan (mg g⁻¹), K_f is the constant that indicates adsorption capacity related to bond strength, C_e is the equilibrium concentration of hinosan (mg L⁻¹), and $1/n$ is the parameter, related to the intensity of adsorption or surface heterogeneity.

The initial concentration of hinosan, which is exposed to the adsorbent with the mass of 0.1 g for studying adsorption isotherms, varied from 25 to 250 μg mL⁻¹. The experiments were carried out under optimal conditions with RHNAC, RHAC, and EAC (Fig. 3). As the results show, RHNAC is a better adsorbent than EAC and RHAC. Therefore, the Langmuir and Freundlich adsorption isotherm models were used to describe the results of hinosan adsorption by RHNAC (Fig. 4a, b).

Comparison of the obtained results shows that RHNAC has the best performance, and according to the correlation coefficient value (R^2) that is shown in Table 2, the adsorption equation isotherm is well fitted with Langmuir isotherm suggesting that the hinosan adsorption on adsorbent is possibly a monolayer and homogeneous adsorption.

The value of Q_{max} obtained for RHNAC under the used experimental conditions, based on Langmuir isotherm plot, was 81.366 mg g⁻¹.

Kinetic study

Investigation of hinosan adsorption kinetic can be useful for evaluating its adsorption dynamics. Kinetic experiments were performed to determine the adsorption rate of hinosan for reaching an equilibrium concentration. The effect of

concentration on adsorption kinetics for hinosan was investigated at the concentration of 180 μg mL⁻¹. The volume of water samples and the mass of adsorbent were 1.000 L and 0.1 g respectively. Ten milliliters of water samples was taken periodically (30–300 min) and analyzed by GC-ECD after extraction using n-hexane. The obtained data was examined using Lagergren pseudo-first-order and pseudo-second-order kinetic models. The linear form of the kinetic models is shown below:

$$\log(Q_e - Q_t) = \log Q_e - k_1 t \tag{4}$$

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{t}{Q_e} \tag{5}$$

where Q_t and Q_e are the adsorbed amount of hinosan at time t and equilibrium time (mg g⁻¹), respectively. k_1 (min⁻¹) and k_2 (g mg⁻¹ min⁻¹) are the equilibrium rate constants of pseudo-first-order and the pseudo-second-order adsorption, respectively (Ho and McKay 1999).

The kinetics of adsorption was investigated by performing a set of experiments with a constant concentration of 180 μg mL⁻¹ of hinosan under optimum conditions carried out. The obtained results were applied to different kinetic models and it showed that the pseudo-second-order model described the adsorption kinetic of hinosan onto RHNAC better than the pseudo-first-order (Fig. 5a, b). The kinetics data derived from the plot of pseudo-first and pseudo-second-order model and kinetic are summarized in Table 3. It is also observed that correlation coefficients (R^2) for the pseudo-first order were lower than those for the pseudo-second-order that indicates the applicability of second-order nature of the adsorption process of hinosan by RHNAC.

Application in underground water

The applicability of the current method was evaluated by analyzing the real samples which were collected from the wells of Guilan Province which were polluted by hinosan. 1.000 L of real sample was added to

Table 3 Values of Hinosan in real samples before and after removal process by RHNAC

Pseudo-first-order			Pseudo-second-order			
q_{e1} (mg g ⁻¹)	k_1 (1 min ⁻¹)	R^2	q_{e2} (mg g ⁻¹)	k_2 (g mg ⁻¹ min ⁻¹)	q_e (exp) (mg g ⁻¹)	R^2
22.313	0.1989	0.9628	70.834	6.6×10^{-4}	70.834	0.9936

Table 4 Values of Hinosan in real samples before and after removal process by RHNAC

Sample	Before adsorption* (ppb)	After adsorption* (ppb)	% Removal
Roudsar	0.65 ± 0.04	0.07 ± 0.01	89.2
Langroud	1.1 ± 0.06	0.09 ± 0.01	91.8
Rasht	0.87 ± 0.05	0.06 ± 0.01	93.01
Fouman	1.36 ± 0.08	0.11 ± 0.02	91.9
Anzali	0.93 ± 0.05	0.079 ± 0.01	91.5

*Concentrations calculated after correction by preconcentration factor

RHNAC at optimum conditions, then desorbed by an organic solvent, and the extracted analyte was analyzed by GC-ECD. 1.000 L of real sample was extracted by n-hexane and injected to GC-ECD to compare with the results obtained from the proposed method. The results showed that 91% of the hinosan was removed by RHNAC. Table 4 shows the values of hinosan in real samples before and after removal by RHNAC. All of the analyses were replicated 3 times.

Also, the effect of total organic compounds (TOCs) on the removal efficiency of hinosan was studied, and the result is shown in Fig. 6. According to the result, the removal efficiency was not affected by other organic compounds at low concentration but by increasing the TOC, the removal efficiency decreased.

Conclusions

Based on the obtained results, RHNAC can safely be applied as a potential adsorbent for the removal of hinosan from underground water. RHNACs are cheap and easily available materials that can act as a good replacement for other adsorbents. Also, it can be a suitable solution for disposal problem of rice waste product. The capability of RHNAC as an adsorbent for the elimination of hinosan was studied and compared with two other activated carbons. Adsorption kinetics and adsorption isotherms for the elimination of hinosan by

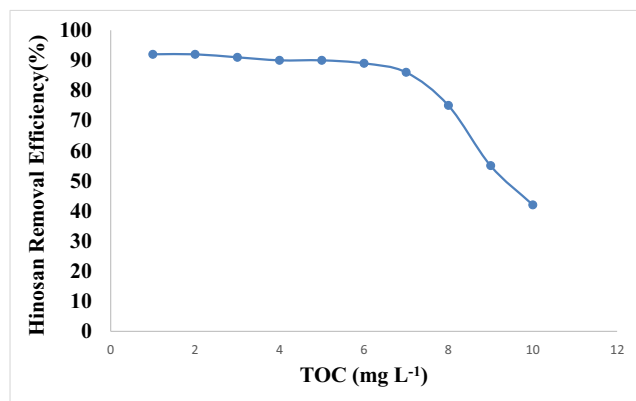


Fig. 6 The effect of total organic compounds (TOC) on the removal efficiency of hinosan

RHNAC were studied, and RHNAC was introduced as an effective adsorbent for the elimination of hinosan from the underground waters.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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