

Synthesis and characterisation of some new non-conventional materials as low-cost adsorbents for removal of nitrates from groundwater in Al-Qurayyat city northern Saudi Arabia

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Abstract: Groundwater in Saudi Arabia contains high concentrations of nitrates. Nitrates are respected as the main groundwater pollutant causing dangerous health and environmental influences. The removal of nitrate from groundwater in Al-Qurayyat, Saudi Arabia, was investigated in this work using palm wastes. Palm fibers (PFI) and palm fronds (PFR) were initially employed as feedstock for the manufacturing of adsorbents, which were activated by heating to 300 °C. The activation of PFI and PFR resulted in an increase in surface area. Batch tests were used to determine the best environment for nitrate adsorption on PFI and PFR. The best factors for nitrate removal were as follows: the contact time was 20 min, the initial concentration of NO_3^- was 20 mg/L, the dose was 2 g/L and the pH was 6. The adsorption capacity of the PFI and PFR was 72% to 77%. In terms of nitrate removal effectiveness under local optimal circumstances, comparisons with commercial activated carbon (CAC) and anion exchange resin (Purolite A 520E) revealed that PFI and PFR are inferior to CAC and Purolite A 520E. However, the PFI and PFR were able to remove nitrate economically even under the most basic operating parameters.

Keywords: water pollution; agricultural wastes; fronds; palm fibers

In Saudi Arabia, in general, and Al-Qurayyat city, in specific, the main water source is the traditional source, which is groundwater, and non-traditional source, which is purification. Groundwater is used in many parts of the country, and other places, it is the only supply of water. Because of the absolute number of people, the yearly water supplies were expanded in 1980 from 2 000 million cubic meters to 27 000 million cubic meters in 1990 approximately. In 1992 the prerequisites increased to 30 000 million cubic meters (Dabbagh and Abderrahman 1997, Al-Omran et al. 2018). The degradation of groundwater is owing to many incorrect procedures in industrialisation and agriculture caused by humans in Saudi Arabia (Alabdula'aly 1997). Currently, great parts of superficial and groundwater are exposed to the danger of nitrate pollution in many countries and areas. This is mostly associated with the incessant growth of manufacturing wastewater, mineral fertiliser, domestic dirt and waste compost and other water pollution causes (Jin et al. 2012). The great concentration of nitrate in water can disturb

human health and create environmental dangers, which may cause cancer to humans and destroy biodiversity in the region (Sandor et al. 2001, Hajhamad and Almasri 2009, Alabdula'aly et al. 2010, Keränen et al. 2015, Yang et al. 2017, Lv et al. 2021, Wang et al. 2021). Amongst the various ways of nitrate removal, adsorption methods have the benefit of simple process, little energy depletion and a small treatment series. Consequently, it is extremely significant to choose the suitable nitrate adsorption material (Oztürk and Bekta 2004, Samatya et al. 2006, Zhang et al. 2014). Numerous adsorbents, such as chitosan (Hu et al. 2016, Karthikeyan et al. 2019), ion exchange resin (Nur et al. 2015) and activated carbon (Oztürk and Bekta 2004) can remove nitrate in water. Conversely, extremely of these materials have large production charges, complex production techniques or are difficult to regenerate. Hence, they are not apposite for the behaviour of large areas of nitrate polluted water. The use of low-cost manufacturing and agricultural waste as an adsorbent for wastewater treatment has gotten a lot more attention in recent years

(Pollard et al. 1992). Several agricultural wastes have been investigated as adsorbents in wastewater treatment in recent years (Marshall and Johns 1996, Khan et al. 2004, Hegazi 2013, Uzma 2013). Low cost, the convenience of handling, environmental eco-friendly, high affinity, and high capacity are all desirable qualities in an adsorbent for the removal of organic and inorganic chemicals in wastewater. A batch adsorption study of nitrate ions from aqueous solution by modification bentonite, fly ash and wheat straw has also been reported (Suryadi et al. 2016, Liu et al. 2018, Behrouz et al. 2019, Cheng et al. 2019).

The palm tree is an evergreen plant that requires pruning after 3 to 7 years when the old leaves have dried up. As a result, each year a significant volume of residue is produced as a result of the removal of dried leaves, necessitating optimal management and effective usage (Sanati et al. 2013). Palm leaf has been shown to be a low-cost adsorbent for heavy metal removal in some studies (Zare and Ghasemi 2017). As a result, the current study looked at the ability of the palm fibers and fronds to remove nitrate ions from aqueous solutions. The palm fibers and fronds are the fiber cellulosic containing frequently polysaccharides, have a common formula of $C_x(H_2O)_y$. These signify a varied change of polymers, originally named carbohydrates (Al-Khanbashi et al. 2005). Palm fibers and fronds have drawn special attention as potential adsorbents due to their low charge compared to activated carbon and high content of hydroxyl functional groups, as well as their carbon content and surface area, all of which contribute to their extreme adsorption ability for contaminant removal in wastewater (Riahi et al. 2009, Mahmoud and El-Halwany 2013). Several previous studies showed the effectiveness of palm waste, whether modified or not, in removing nitrates from wastewater (Bashir et al. 2018, Manikam et al. 2019). The aim of the current study was to investigate the probability of using the palm fibres and fronds for the removal of NO_3^- . The results of pH, adsorbents dose, initial NO_3^- concentration and contact time on the adsorption capability were examined. Adsorption isotherm models were also investigated. This research was performed in Al-Qurayyat city northern Saudi Arabia.

MATERIAL AND METHODS

Collection of groundwater samples. Groundwater samples were collected by the Ministry of Environment, Water and Agriculture from the wells of the study area in the city of Al-Qurayyat in the north of the Kingdom of Saudi Arabia. Groundwater samples were

collected in 1-L glass bottles that had been carefully rinsed with distilled water after being completely cleansed with tap water. At the sampling locations, samples were kept on ice and then refrigerated until they were tested in the laboratory. Determination of nitrate concentration was performed spectrophotometrically at λ_{max} 220 nm utilising the Double Beam UV spectrophotometer (ST-UV-1901PC-US, Hunan, China). To determine the mean value, each experiment was repeated three times. All samples were determined as dilutions sequences and directly inserted into the cell of the spectrophotometer. In the groundwater collected from the various wells, the average nitrate concentration was 89 mg/L. The maximum pollutant limits for drinking water (45 mg/L as NO_3^-) were exceeded at this concentration (Alabdula'aly et al. 2010).

Preparation and synthesis of adsorbents. The palm fibers (PFI) and palm fronds (PFR) (Figure 1) were collected from the field in Al-Qurayyat city in the Kingdom of Saudi Arabia. The accumulated biomaterial was cleaned several periods with distilled water to create the PFI and PFR surface of all the dirt fragments held to it. The moist matter was then dried at 100 °C for 4 h. To chemically activate PFI and PFR, they were soaked in H_3PO_4 (2% v/v) instantly under room temperature for 24 h, then eliminated and heated to 300 °C for 3 h. The mixture was then allowed to cool before being sieved to the desired particle size for adsorption testing using a 60-mesh sieve (0.25 mm). Finally, chemical activation was used to prepare PFI and PFR, which matched the performance of PFI and PFR (Lim et al. 2010).

Characterisation, test methods and equipment. The samples' external morphology was studied using a scanning electron microscope (SEM) (S-4800, HITACHI, Tokyo, Japan). Energy Dispersive X-ray (EDX, Eindhoven, Holland) analysis was carried out by the Philips XL30 ESME model, Eindhoven, Holland

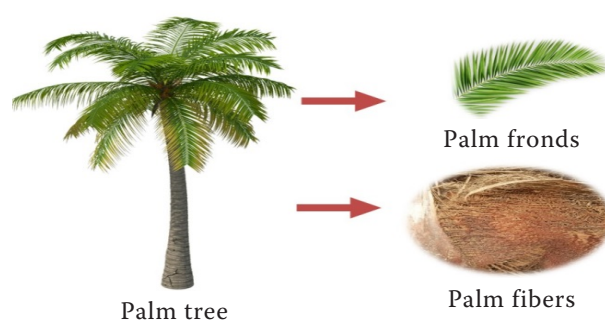


Figure 1. Photograph of palm fibres and palm fronds

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(Resolution of 2.4 nm and 3.0 nm under operating conditions were vacuum 20–30 kV and 0.8 torrs with BSD (Back Scattered Detector) and employed to certain the elemental composition of adsorbents.

Batch adsorption experiments. The batch adsorption studies were carried out to assess the impacts of several operational parameters on NO_3^- adsorption by PFI and PFR, such as the initial solution pH, contact time, initial NO_3^- concentration, and adsorbents dose. In each batch experiment, one of the components changed, while the others remained the same. At pH values ranging from 2 to 10, the effect of the starting solution pH on NO_3^- removal by PFI and PFR was investigated. 0.1 mol/L HCl and 0.1 mol/L NaOH were used to adjust the pH. With a contact time ranging from 5 to 60 min, the amount of nitrate removed by PFI and PFR was estimated. Different doses in the range of 0.5–5.0 g/L were used to evaluate the effect of PFI and PFR quantity on advancing the removal amount of nitrate. The batch investigations were shown by adjusting the concentration of NO_3^- the solution from 10 mg/L to 50 mg/L to evaluate the influence of the initial nitrate concentration on the adsorption ability of nitrate by PFI and PFR. Each batch adsorption experiment was carried out in a volumetric flask with a NO_3^- solution of 50 mL. After adjusting the variable factor and correcting the others, the mixture was constantly stirred with a magnetic mixer at 120 rpm for 2 h. The sample was then passed through filter paper, and the residual nitrate concentration in the filtrate solution was determined using a spectrophotometer. Experiments were repeated three times to assure repeatability, and average results were provided. Data were stated as mean $\pm$ standard deviation ($n = 3$). In batch experiments, the amount of NO_3^- adsorbed per unit mass of P-fibres and P-fronds (nitrate adsorption capacity onto the adsorbents) at time t , Q_t (mg/g), and at equilibrium, Q_e (mg/g), as well as the nitrate removal percentage at equilibrium were calculated using the equations below (Mehdinejadani et al. 2019):

$$Q_t = \frac{(C_i - C_t)}{m} \times V \quad (1)$$

$$Q_e = \frac{(C_i - C_e)}{m} \times V \quad (2)$$

$$\text{Removal efficiency (\%)} = \frac{C_i - C_e}{C_i} \times 100 \quad (3)$$

Where: C_i – initial NO_3^- concentration (mg/L); C_t and C_e – residual NO_3^- concentrations at time t and equilibrium (mg/L); V – volume (L); m – adsorbent dose (g/L); V – solution volume (L). Isotherms designate the capability of an

adsorbent and its operative dependence on the amount of adsorbate present (Weber 1974). The adsorption capacity at equilibrium, q_e , was calculated at various beginning NO_3^- concentrations in order to determine the best adsorption isotherm. After that, the q_e and equilibrium concentrations were fitted to the Langmuir and Freundlich isotherms, which are extensively used in water treatment (Weber 1974). The isotherm models of Langmuir Eq. (4) (Langmuir 1918) and Freundlich Eq. (5) (Freundlich and Heller 1939) were used to analyse the experimental data, which were stated as follows:

$$\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m} \quad (4)$$

$$\log q_e = \frac{1}{n} \log C_e + \log k_F \quad (5)$$

The equilibrium and highest adsorption capabilities, respectively, are q_e and q_m . C_e is the solution's equilibrium concentration; K_L is the equilibrium constant for adsorption heat; and K_F and n are the equilibrium constants for adsorption capacity and strength, respectively.

RESULTS AND DISCUSSION

Characteristics of adsorbents. The micrographs obtained using SEM illustrating the porous structure of PFI and PFR produced in this study are shown in Figure 2A, B. On the surface and inside PFI and PFR, the apparent mesoporous structure of the two adsorbents could be seen. NO_3^- was easily caught by the active sites of PFI and PFR due to the porous, irregular, and uneven structure of the material, allowing the adsorption system to reach equilibrium with a relatively substantial adsorption capacity when aqueous solution diffused into the interior of PFI and PFR. Figure 2C, D shows a difference in the surface of PFI and PFR, with uneven protrusions and a heterogeneous morphology appearing on the solid surface following adsorption, which was attributed to the fibres and fronds expanding during the process (Manikam et al. 2019). The surface atomic composition of the examined adsorbents, as fixed by EDX experiments, is listed in Table 1. EDX analysis signified that the PFI was contained of 28.65, 4.34 and 67.30% (by weight) of carbon, nitrogen and oxygen, respectively and showed that the PFR was contained of 30.28, 3.64 and 65.89% (by weight) of carbon, nitrogen and oxygen, respectively. The weight of nitrogen is increased to 24.76% and 22.53% in NO_3^- loaded PFI and nitrate-loaded PFR, respectively. The increased weight of nitrogen in

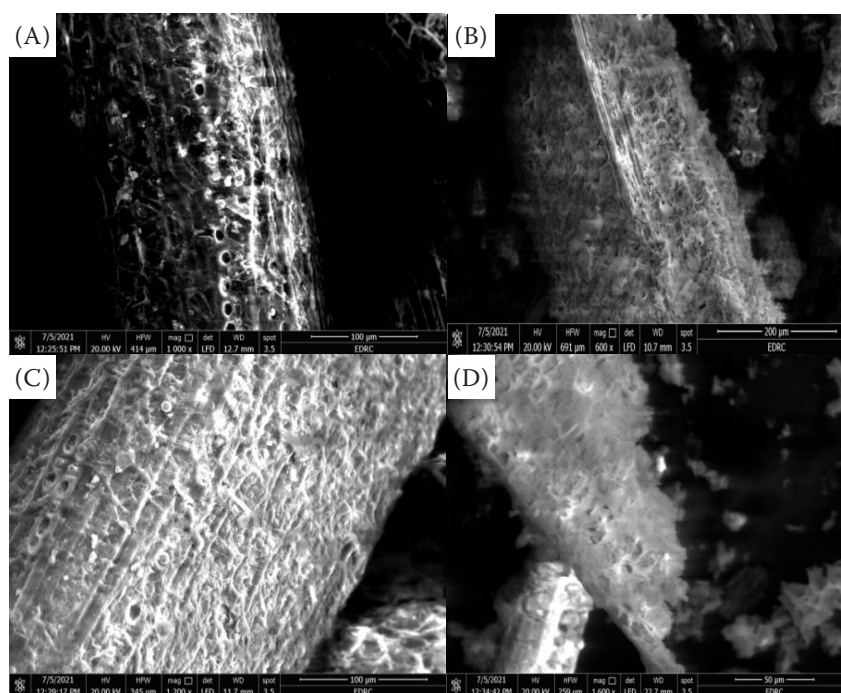


Figure 2. Scanning electron microscope (SEM) of palm fibres (PFI) and palm fronds (PFR) before and after adsorption, before adsorption (A) for PFI and (B) for PFR and after adsorption (C) for PFI and (D) for PFR

NO_3^- loaded PFI and nitrate-loaded PFR revealed that the adsorbent removed a significant quantity of NO_3^- from the solute (Srivastava and Singh 2017, Bashir et al. 2018, Tejada-Tovar et al. 2021).

Influence of initial solution pH. Figure 3 shows that when pH increased from 2 to 6 the NO_3^- removal effectiveness increased. However, as the pH rises from 8 to 10, the removal efficacy decreased. As a result, the PFI and PFR can effectively remove NO_3^- from water in a wide pH range, with a maximum removal efficiency of 77.0% for PFI and 72.0% for PFR at pH 6. The competition for both adsorbent active sites between chloride and nitrate anions may explain the slight decrease in elimination efficacy at acidic pHs (Milmile et al. 2011, Mazarji et al. 2017). Furthermore, the addition of 0.1 mol/L HCl to the nitrate solution increases the concentration of the Cl^- in the solution at acidic pHs. The nitrate removal efficacy is reduced in this case due to the

inhibitory effect of the Cl^- , which will be demonstrated in the paper comparing anions results. The decrease in removal effectiveness at alkaline pHs could be due to an increase in negatively charged sets, the contest between OH^- and NO_3^- for the PFI and PFR active sites, and the interface *via* a passive passage in the pores (Xu et al. 2013, Mazarji et al. 2017). The pH of 6 was selected to accord the other batch examinations in this study, based on the data achieved in this section.

Influence of contact time. Figure 4 shows that during the first 20 min of the experiment, the NO_3^- removal per cent increases fast from 30.0% to 77.0% for PFI and 22.0% to 72.0% for PFR. A large amount of early adsorption may be due to the limited number of accessible adsorption seats and the rapid concentration drop between the NO_3^- in the solution and the adsorbent surface during the initial adsorption phase. During the experimental interval, these active

Table 1. Chemical composition of palm fibres (PFI) and palm fronds (PFR) before and after adsorption

Element	Before adsorption		After adsorption	
	PFI	PFR	PFI and NO_3^-	PFR and NO_3^-
(mass % $\pm$ error)				
Carbon	28.65 $\pm$ 0.85	30.28 $\pm$ 0.60	26.20 $\pm$ 0.42	27.64 $\pm$ 0.51
Nitrogen	4.34 $\pm$ 0.47	3.64 $\pm$ 0.18	24.76 $\pm$ 1.16	22.53 $\pm$ 1.02
Oxygen	67.30 $\pm$ 0.25	65.89 $\pm$ 0.15	49.05 $\pm$ 1.26	49.74 $\pm$ 0.76

Mass % errors signify standard deviation ($n = 3$)

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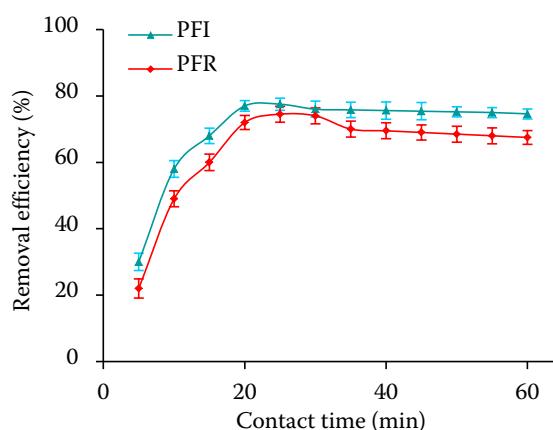


Figure 3. Effect of pH on nitrate removal efficiency by using palm fibres (PFI) and palm fronds (PFR). Error bars signify standard deviation ($n = 3$)

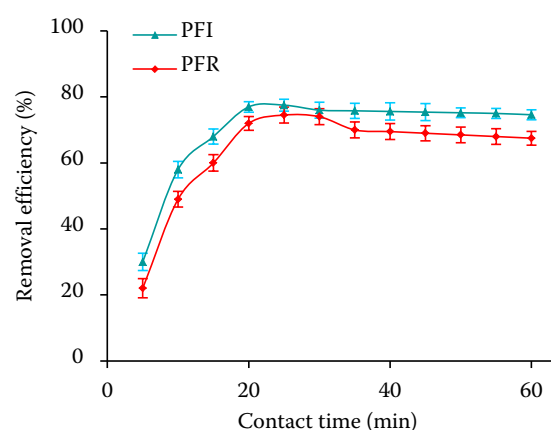


Figure 4. Effect of contact time on nitrate removal efficiency by using palm fibres (PFI) and palm fronds (PFR). Error bars signify standard deviation ($n = 3$)

sets and concentration gradients decrease regularly, lowering the adsorption rate until equilibrium is reached. Figure 4 indicates that after 20 min, the removal % is fairly steady and does not increase as contact duration increases. As a result, the equilibrium contact duration of 20 min was chosen and completed in the other batch studies. These results are in line with those of other reports that used another adsorbent for nitrate removal (Hafshejani et al. 2016, Mazarji et al. 2017).

Influence of adsorbent dosage. As shown in Figure 5, increasing the adsorbents dosage from 0.5 g/L to 2.0 g/L significantly increases the NO_3^- removal capability from 44.0% to 77.0% for PFI and 35.0% to 72.0% for PFR, and then the amounts begin to fall as the dosage of adsorbents is increased beyond this limit. The significant improvement in NO_3^- adsorption onto PFI and

PFR in the range of 0.5 g/L to 2.0 g/L can be qualified to a greater number of variable parts and accessible adsorbent sets (Milmile et al. 2011). Similarly, the lessening of NO_3^- adsorption on the scale of 2.0 g/L to 5.0 g/L can be assigned to the accumulation of exchanger fragments at large amounts of adsorbent, which leads to a reduction in the adsorbent's useable surface area (Katal et al. 2012). Later on, as the PFI and PFR dosages are raised, the efficacy remains practically constant and does not rise. As a result, the 2.0 g/L PFI and PFR dosage was chosen as the optimal dose, and the following batch trials were carried out at this dose.

Influence of initial nitrate concentration. A specified examination of Figure 6 shows that the removal per cent enlarges from 70.0% to 77.0% for PFI and from 68.0% to 72.0% for PFR when the initial NO_3^- concentration grows from 10 mg/L to

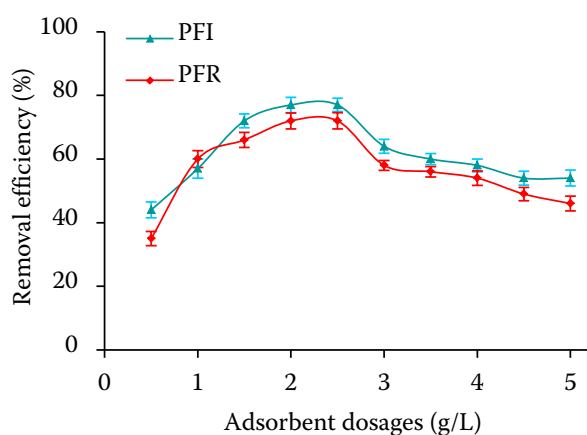


Figure 5. Effect of adsorbents dosage on nitrate removal efficiency by using palm fibres (PFI) and palm fronds (PFR). Error bars signify standard deviation ($n = 3$)

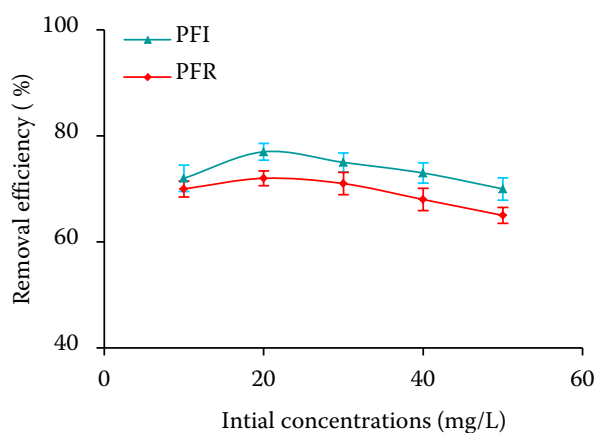


Figure 6. Effect of initial nitrate ion concentration on nitrate removal efficiency by using palm fibres (PFI) and palm fronds (PFR). Error bars signify standard deviation ($n = 3$)

Table 2. Isotherm parameters of nitrates adsorption onto palm fibres (PFI) and palm fronds (PFR)

Adsorbent	Langmuir constant			Freundlich constant		
	q_m (mg/g)	K_L (L/mg)	R^2	K_F ((mg/g) (L/mg) $^{1/n}$)	$1/n$	R^2
PFI	204.08	0.0221	0.8098	4.2159	0.9394	0.9776
PFR	243.90	0.0150	0.7920	3.8990	0.9098	0.9901

q_m – highest adsorption capability; K_L – equilibrium constant for adsorption heat; K_F – equilibrium constants for adsorption capacity

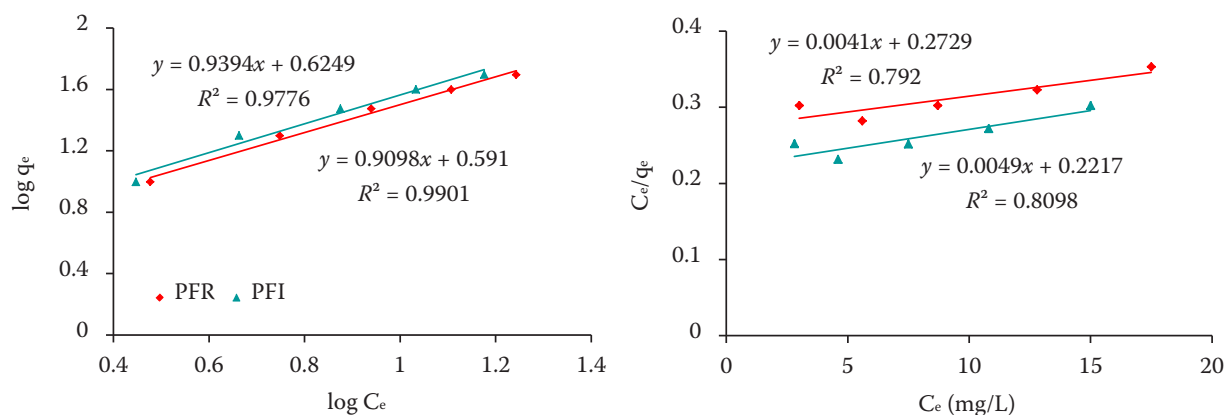


Figure 7. Adsorption isotherms of nitrate. (A) Freundlich model; (B) Langmuir model. q_e – equilibrium adsorption capabilities; C_e – residual NO_3^- concentrations at equilibrium

20 mg/L. However, the percentage removal of the initial NO_3^- ions concentration decreases as the NO_3^- concentration increases, indicating that the adsorbent was gradually saturated with NO_3^- . As the driving force increased with the initial NO_3^- concentration, adsorption had to overcome the increasing mass transfer barrier between the solute phase and the adsorbent phase. The initial period of adsorption was easy and rapid, with nitrate ions adsorbed on the

exterior surface area of PFI and PFR. The exterior surface was then soaked, avoiding the adsorptive sets; NO_3^- then entered the pores and adsorbed on the interior surface of PFI and PFR over a long period (Derylo-Marczewska et al. 2019).

Adsorption isotherm for nitrate adsorption onto PFI and PFR. The factors for both adsorption isotherms are summarised in Table 2 and Figure 7. When the R^2 values for the Langmuir and Freundlich isotherms are compared, the Freundlich isotherm performs exceptionally well in terms of adsorption. When $1/n$ is between 0.1 and 1.0, adsorption is more likely to occur, however, when $1/n$ is more than two, adsorption becomes more difficult (Carter et al. 1995). PFI and PFR were found to be beneficial for nitrate adsorption in this experiment ($1/n = 0.9394$ for PFI and $1/n = 0.9098$ for PFR). The Freundlich model suggests that adsorption happens by monolayer adsorption on irregular surfaces of the adsorbent, with the interaction between adsorbed ions (Derylo-Marczewska et al. 1984).

Comparison between PFI, PFR, anion exchange resin, and commercial activated carbon. The performances of PFI and PFR were compared with anion exchange resin (Purolite A 520E) and commercial activated carbon (CAC). Thus, the adsorbent dosage of 2 mg/L, initial nitrate concentration of 55 mg/L, and

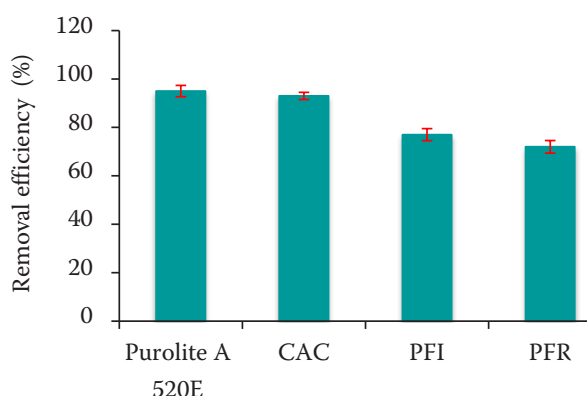


Figure 8. Adsorption nitrate efficiency with different adsorbents: Purolite A 520E; CAC – commercial activated carbon; PFI – palm fibres; PFR – palm fronds. Error bars signify standard deviation ($n = 3$)

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pH of 6.0 was used as initial experimental conditions. After running a *t*-test on the per cent nitrate removal of the four adsorbents, it was observed that there was a significant difference between them (Figure 8). Purolite A 520E and CAC had more excellent means, with 95.0% and 93.0% nitrate removal rates, respectively. In contrast, PFI and PFR had removal rates of 77.0% and 72.0%, respectively. The ascendancy of CAC's surface area above that of PFI and PFR (Haycarb 2019) can be used to describe this. These findings are in line with those of other studies that employed commercial activated carbon (Oztürk and Bektas 2004) and ion exchange resin (Kalaruban et al. 2017) to remove substantial amounts of nitrate from water. Furthermore, as the fundamental issue with CAC is its high cost, the use of PFI and PFR can be respected. In conclusion, the results of this investigation showed that for nitrate removal, the PFI and PFR required the simplest operating conditions. As a result, it is proposed that PFI and PFR can be used for their potential to remove nitrate from natural water resources.

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