

Adsorption of Nitrite Ions from Wastewater Using Bio-sorbents Derived from *Azadirachta indica* Plant

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Abstract: Economically cheap adsorbents have been investigated for nitrite removal from aqueous solution through batch adsorption studies. The adsorbents prepared from different parts of the plant, *Azadirachta indica*, are used in study. Effect of various physicochemical parameters, namely pH, adsorbent dose, contact time, initial concentration of the nitrite ion and effect of interfering ions present in water are studied. The experimental data is analyzed using Freundlich and Langmuir isotherms to determine the mechanism of adsorption process. It is found that the Langmuir model fitted well. The kinetics of adsorption process is studied using pseudo first-order and pseudo second-order models. It is observed that the kinetics of the process followed the pseudo second-order model. The methodology developed is successfully used to real groundwater samples polluted with nitrite ions.

Key words: Nitrite removal, batch adsorption, *Azadirachta indica*, application.

Introduction

Nitrates (NO_3^-), nitrites (NO_2^-) and Ammonium (NO_4^+) are the three important forms of nitrogen and they are present in agricultural, domestic and industrial wastewaters and in drinking water causing toxicity to human health (Katta and Jianping, 2000). Out of these, nitrite ion is one of the most significant pollutants of aqueous environment. In surface or ground waters, the increasing level of nitrite ion is due to the use of fertilizers in agricultural fields, many industrial processes and animal seepage (Panduru-Balint et al., 2012). The U.S. Environmental Protection Agency (USEPA, 1983) has established maximum contaminant levels of 10 mg/lit for nitrate and 1.0 mg/lit for nitrite.

The compounds of nitrogen are turning to be potential hazards for aquatic as well as for human beings because in the metabolic processes, the nitrate is reduced to nitrite which in turn depletes the oxygen from blood

cell causing a dreaded disease, methaemoglobinaemia (WHO, 1985). Further, the eutrophication in water bodies is one of the consequences of nitrogen contamination and this process endangers the aquatic life.

The N-nitroso compounds (NOC) in the human stomach are formed due to the reaction between nitrite and secondary or tertiary amine in acidic media. These NOC compounds are known to be carcinogenic, teratogenic and mutagenic (Mikuska and Vecera, 2003) and cause stomach and bladder cancer (Shams, 2000). Therefore, from the health and environmental approach, it is necessary to remove the nitrate and nitrite from water samples (Foglar et al., 2005).

Various conventional methods such as reverse osmosis, ion exchange, electrodialysis, chemical coagulation, membrane technique and adsorption process are available for removing nitrite ions from wastewater (Aslan and Turkman, 2004). The selection of the method depends on conditions like area, concentration,

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availability of resources etc. But most of these methods suffer from either one or other drawbacks, such as the high maintenance or operational cost, the disposal of the resulting sludge and tedious procedures involved in the treatment (Aslan and Turkman, 2004) and a universally acceptable method is still eluding the researchers.

The bio-adsorbents derived from natural sources in controlling the pollutive ions in waste waters are proving to be an effective solution. Our research groups is working on these lines of pollution control and some successful procedures have been developed for the removal of chromium (Naga Babu et al., 2016; Sree Devi et al., 2012), zinc (Papodu et al., 2014), aluminium (Aruna Kumari et al., 2012), fluoride (Suneetha et al., 2014; Suneetha et al., 2014-2015; Suneetha et al., 2015 a, b, c), phosphate (Hanumantha Rao et al., 2015), nitrite (Suneetha and Ravindhranath, 2014), ammonia (Suneetha and Ravindhranath, 2012) and some cationic and anionic dyes (Karimulla and Ravindhranath, 2014; Srinivasa Reddy and Ravindhranath, 2014).

In present work, the adsorbents prepared from different parts of the plant, *Azadirachta indica*, are used to study their adsorption nature towards nitrite ions in waste waters. The maximum extractability conditions of the adsorbents are optimized at various physicochemical parameters such as pH, contact time, adsorbent dose, initial concentration of the nitrite ion and effect of interfering ions present in water. Further, the nature of the adsorption process has been analyzed by Freundlich and Langmuir, isotherms and kinetics of adsorption have been studied using pseudo first-order and pseudo second-order models. The procedures developed are applied to nitrite contaminated polluted waters.

Materials and Methods

Reagents and Chemicals

All the chemicals used were of analytical reagent grade purchased from Merck, India and Sd. Fine Chemicals. A stock solution of sodium nitrite, sulphanilic acid reagent, α -naphthylamine reagent and sodium acetate (2M) were prepared as described in Standard methods for the Examination of Water and Wastewater (APHA, 1998).

Preparation of Adsorbents

Adsorbents used in the present study are: leaf and bark powder and leaf ash of *Azadirachta indica* plant. *Azadirachta indica* is an evergreen tree native to Southeast Asia and it belongs to **Meliaceae family**.

Traditional medicines can be prepared from all parts of this plant.

The raw materials (leaves and bark) were washed with double distilled water and the pieces were sun dried. The dried materials were powdered and sieved to get particles of size: <75 microns and activated at 105 °C in an oven and then employed in this study. Further, the leaves of the said plant were burnt to ashes, meshed and used in this work.

Experimental Procedure

For the removal of nitrite ions from the waste waters, batch adsorption studies were adopted (Metcalf and Eddy, 2003). Predetermined concentration of 250 ml of nitrite solution was taken into previously washed 500 ml stopper bottle and to it carefully weighed quantity of adsorbent was added. The pH value of the suspension was adjusted with dil. HCl or dil. NaOH solution using pH meter. The sample was shaken in mechanical shakers for a desired period and after the equilibration time the bottle was allowed to stand for 2 min for settling the adsorbent and it was filtered using Whatman No. 42 filter paper. The filtrate was analyzed for residual nitrite concentration by spectrophotometrically (Vogel, 1961).

Estimation of Nitrites

A measured quantity of the nitrite sample was taken in to a 50 ml volumetric flask. To it 1 ml of Sulphanilic acid reagent solution was added, mixed well and was allowed to stand at least 3 min and not more than 10 min at room temp in diffused light. Then 1 ml of the α -naphthylamine solution and 1 ml of 2M-sodium acetate solution to act as buffer (pH: 2.0 – 2.5) were added. The solution was then diluted to 50 ml and mixed well. After 10 min, but not later than 30 min, the O.D. of the reddish-purple colour developed was measured at 520 nm against blank using U.V and visible spectrophotometer (Systronics make). Previously, a standard graph was constructed between O.D. and known concentrations of nitrite, in conformation of Beer's law adopting Least Square Method for linear graphs. The measured O.D. values for the unknown solutions were referred to standard graph to determine the concentrations of nitrite in un-known samples.

The same procedure has been adopted for the experiments carried out by varying physicochemical parameters such that adsorbent dosage, pH, agitation time, initial concentration of nitrite solution and in presence of co-ions.

Figure 1: *Azadirachta indica* plant.

Results and Discussions

Effect of Various Physicochemical Parameters on Nitrite Removal

Effect of pH

The adsorption process was found to be influenced by the pH of the medium. The effect of pH change on the extraction of nitrite ions from waste waters was carried out within a range of pH 2 to 10 at optimum conditions of parameters. The obtained results were plotted and shown in Figure 2.

With the increase of pH, % of extraction of nitrite is found to be decreased and the optimum pH is found to be 2. The maximum removal at pH 2 is found to be 93.4% for leaves powder, 96.5% for leaves ash and 98.5% for bark powders of *Azadirachta indica*.

These observations may be accounted from the fact that the bio-materials have functional groups such as -OH/COOH and whose dissociation depends on pH. At high pH values, these groups dissociate to yield negative charge to the surface of the adsorbent and this negative charge caused the repulsion of negatively charged nitrite ions and, hence, % removal is low. But as the pH decreases, dissociation is less favoured and even protonation occurs at low pH values reusing positive charge to the surface. This positive charge holds on the negatively charged nitrite ions on the surface resulting in greater % of removal of nitrites from polluted waters.

Effect of Time of Agitation

The effect of agitation time on the extraction of nitrite ions from waste waters was carried out varying the agitation time from 1 to 10 hrs at optimum conditions of parameters. The obtained results were plotted and shown in Figure 3.

With increasing the agitation time, the extraction of nitrite ions was found to be increasing up to a certain period of time after which the rate of adsorption was found to be constant. The optimum time is found to be 6 hrs for leaves powder and 5 hrs for leaves ash and barks powder. This is attributed to the greater number of active sites presented initially on the surface of the

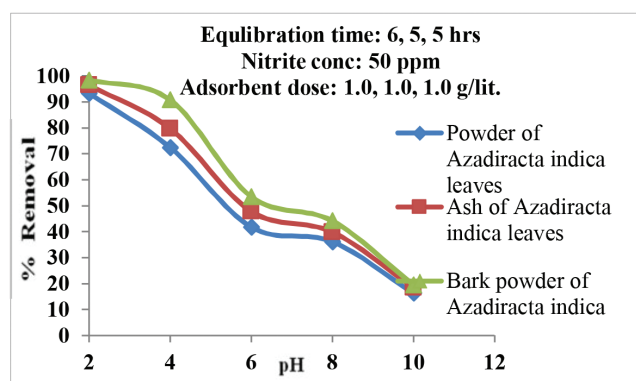


Figure 2: Effect of pH on nitrite removal.

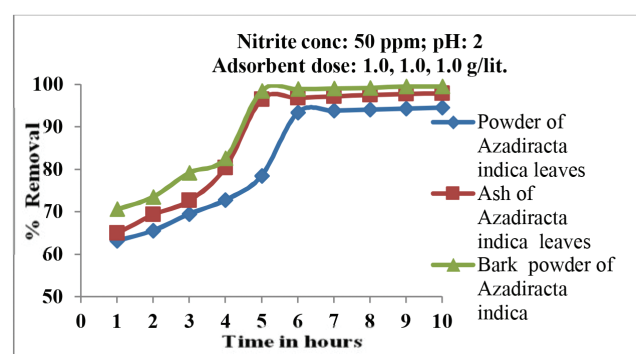


Figure 3: Effect of agitation time on nitrite removal.

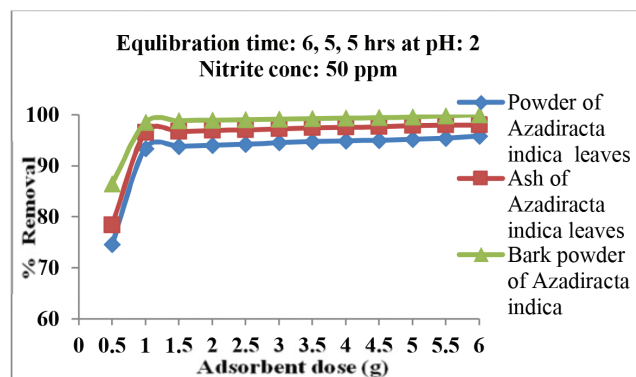


Figure 4: Effect of adsorbent dosage on nitrite removal.

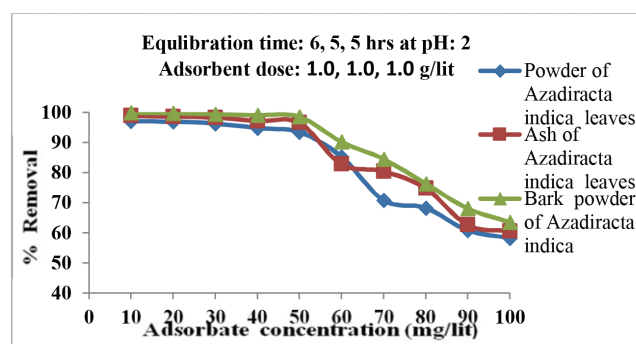


Figure 5: Effect of initial concentration of adsorbate on nitrite removal.

sorbent and are progressively used up with the time and a state of equilibration reached after a definite interval (Nurzulaifa et al., 2016).

Effect of Adsorbent Dosage

The effect of adsorbent dosage on the extraction of nitrite ions from waste waters was carried out varying the adsorbent dosage from 0.5 to 6.0 g/lit at optimum conditions of parameters. The obtained results were plotted and shown in Figure 4.

The nitrite uptake increases initially with increase in the adsorbent dose and remains constant after a certain sorbent concentration. The optimum sorbent dosage needed is 1 gm/lit. With the increase of sorbent concentration, the number of active sites available also increases and results in the greater removal of the nitrite ions. But after a certain dosage, the sites are blocked due to the deposition of the newly added adsorbent on the surface of the previously formed layers of adsorbent (Nurzulaifa et al., 2016).

Effect of Initial Concentration of Adsorbate

The effect of initial concentration of adsorbate on the extraction of nitrite ions from waste waters was carried out varying the adsorbate concentration from 10 to 100 mg/lit at optimum conditions of parameters. The obtained results were plotted and shown in Figure 5.

From Figure 5, it was observed that the extent of adsorption (%) gradually decreased with increasing initial nitrite ion concentration. With the increase of initial concentration from 10 to 100 mg/lit, the percentage removal (% *R*) is decreased from 97.0 to 58.3% with leaves powder as adsorbent; 98.8 to 60.5% with leaves ash; and 99.8 to 63.5% with barks powder. This is due to the non-availability of sufficient number of active site on the fixed adsorbent concentration to remove all the nitrite ions especially at higher concentrations of the adsorbate (Kagne et al., 2008).

Effect of Interfering Ions

The synthetic solutions of nitrite with ten-fold excess of one of the co-ions were prepared. Aliquot samples were agitated with the requisite quantities of adsorbent at the conditions of extraction as cited in Table 1. The results obtained are presented in Table 1.

It can be inferred from Table 1 that cations and anions (except sulphate) have marginal effect on the % removal of nitrite ions at the optimum conditions of extractions as cited in Table 1.

Adsorption Isotherms

An adsorption isotherm is a curve which depicts the movement of a substance from the aquatic environment to a solid-phase at a constant temperature and pH (Foo and Hameed, 2010). Mainly two important isotherms, Freundlich and Langmuir, are frequently used to describe the adsorption data.

The *Freundlich* (1906) adsorption isotherm is $\log(q_e) = \log k_F + \frac{1}{n} \log C_e$ and

Langmuir (1916) adsorption isotherm is

$$(C_e/q_e) = (a_L/k_L) C_e + 1/k_L$$

where k_F and $1/n$ are the Freundlich constants, C_i is the initial nitrite ion concentration, q_e (mg/g) is the amount of nitrite ions adsorbed per unit weight of the adsorbent (mg/g), k_L and a_L are the Langmuir constants related to capacity and energy of adsorption respectively and k_L/a_L is the theoretical monolayer saturation capacity, q_m . Further, in Langmuir isotherm, the separation factor $R_L = 1/(1+a_L C_i)$ is used. As per Hall et al. (1966), R_L indicates the isotherms shape and the adsorption nature as unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$) and irreversible ($R_L = 0$). The data are presented in Figures 6 and 7 and Table 2.

It is inferred from Table 2 that the correlation coefficient (R^2) values for Langmuir model are higher

Table 1: Effect of interfering ions on the extractability of nitrite with different bio-sorbents derived from *Azadirachta indica*

Adsorbents	Maximum extractability at optimum conditions	% of Extraction of Nitrite in the presence of ten-fold excess of interfering ions at optimum extraction conditions											
		SO_4^{2-}	NO_3^-	Cl^-	PO_4^{2-}	F^-	CO_3^{2-}	Ca^{2+}	Mg^{2+}	Fe^{2+}	Cu^{2+}	Zn^{2+}	Ni^{2+}
Leaves powder	93.4%, pH: 2, 6 hrs, 1.0 g/lit	64.2	85.5	97.8	97.8	96.8	98.1	92.6	92.4	92.8	92.9	92.7	92.5
Ash of leaves	96.5%, pH: 2, 5 hrs, 1.0 g/lit	64.7	87.4	97.8	97.1	93.8	98.9	95.6	95.7	95.8	95.9	96.1	95.4
Bark powder	98.5%, pH: 2, 5 hrs, 1.0 g/lit	62.4	89.3	96.8	98.7	82.5	97.1	97.4	97.6	98.1	97.9	98.0	97.5

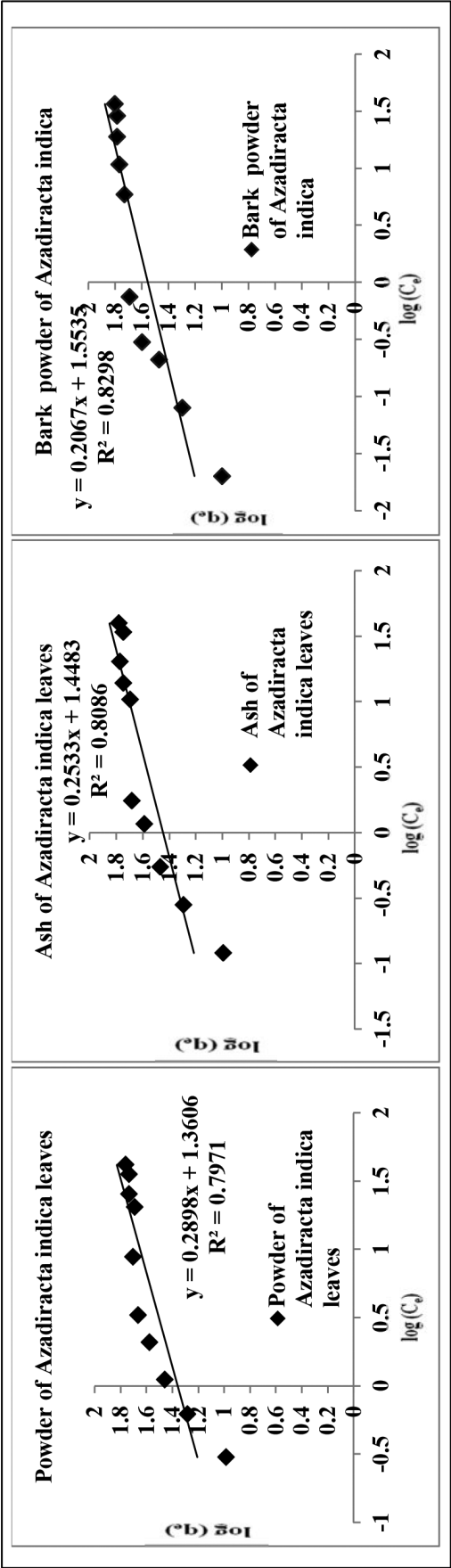


Figure 6: Freundlich adsorption isotherms.

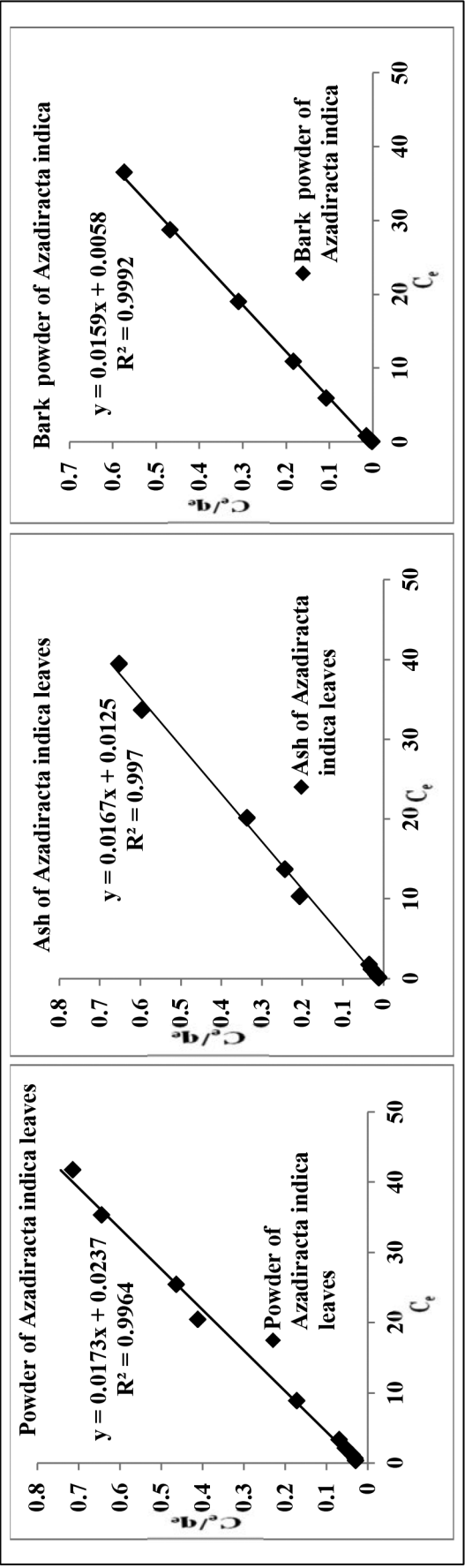


Figure 7: Langmuir adsorption isotherms.

Table 2: Freundlich and Langmuir constants with the correlation coefficients

<i>Adsorbents</i>	<i>Freundlich isotherm</i>			<i>Langmuir isotherm</i>			
	<i>Slope</i>	<i>Intercept</i>	<i>R²</i>	<i>Slope</i>	<i>Intercept</i>	<i>R_L</i>	<i>R²</i>
Leaves powder	0.2898	1.3606	0.7971	0.0173	0.0237	0.0267	0.9964
Leaves ash	0.2533	1.4483	0.8086	0.0167	0.0125	0.0147	0.9970
Barks powder	0.2067	1.5535	0.8298	0.0159	0.0058	0.0072	0.9992

than for Freundlich model and further, R_L values ranges between 0 and 1. This indicates that adsorption is more adequately described by the Langmuir adsorption isotherm and moreover, the adsorption is monolayer and homogeneous (Nurzulaifa et al., 2016).

Adsorption Kinetics

The correlation between the adsorbate uptake rate and bulk concentration of the adsorbate is explained by kinetic models. Two most common adsorption kinetic models, pseudo first-order and pseudo second-order, are used to describe the kinetic data.

The pseudo first-order rate (Lagergren, 1898) equation is $\log(q_e - q_t) = \log(q_e) - \frac{k_1 t}{2.303}$ and

The pseudo second-order rate (Yong et al., 2015) equation is $\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t)$

where q_e and q_t are the amount adsorbed (mg/g) at equilibrium and at time t , respectively and k_1 (min^{-1}) is the rate constant of the pseudo first-order and k_2 (g/mg.min) is the rate constant of the pseudo second-order adsorption process. The obtained data is presented in Figures 8 and 9 and Table 3.

It can be seen from Table 3 that R^2 values for the pseudo second-order adsorption model are close to one than the pseudo first-order model and so, the former is better-fit model to describe the kinetics of adsorption.

Applications of the Developed Bio-sorbents

The workability of the developed bio-sorbents for removing nitrite ion concentrations in naturally occurring polluted waters were tried. For this purpose, five samples were collected from five lakes polluted with nitrite at different places in Bapatla Mandal of Guntur Dist of Andhra Pradesh and the actual concentration of nitrite present in them was analyzed. Then these samples were subjected to the extraction of nitrites using the bio-sorbents developed in this work at optimum conditions

of pH, equilibration time and sorbent concentration. The results obtained were presented in Table 4.

From Table 5 it is observed that the concentration of nitrite in all the polluted water samples collected from different areas of Bapatla Mandal, Guntur Dist. varied from 5.0 to 11.5 mg/lit which is beyond the permissible limit by USEPA: 1.0 mg/lit (USEPA, 1983).

After removal of nitrites from polluted water samples using the above said adsorbents, the concentration of nitrite is below the permissible limit under optimum experimental conditions. Thus the methodology developed in this work using the new adsorbents is remarkably successful.

Conclusions

The present study shows that the adsorbents prepared from various parts of *Azadirachta indica* (leaf powder, leaf ash and bark powder) are effective adsorbents for the removal of nitrite ions from polluted waters. The adsorption process is studied at various physicochemical parameters such as pH, sorbent dosage, time of agitation and initial concentration of nitrite ions and the conditions are optimized for the maximum extraction of nitrite ions.

Maximum adsorption is found to be at pH 2.0 for all the three adsorbents and 93.4%, at equilibration time 6 hrs and adsorbent dosage 1.0 g/lit with the powder of *Azadirachta indica* leaves; 96.5%, at equilibration time 5 hrs and adsorbent dosage 1.0 g/lit with the ash of *Azadirachta indica* leaves; and 98.5% at equilibration time 5 hrs and adsorbent dosage 1.0 g/lit with the bark powder of *Azadirachta indica*. Even ten-fold excess of common cations and anions except sulphate, normally present in waters have shown only marginal interference with the extraction of nitrites from simulated synthetic waters at the optimum conditions of extraction. Equilibrium adsorption data is fitted better into the Langmuir than the Freundlich and the mechanism of the adsorption process is conformed to monolayer

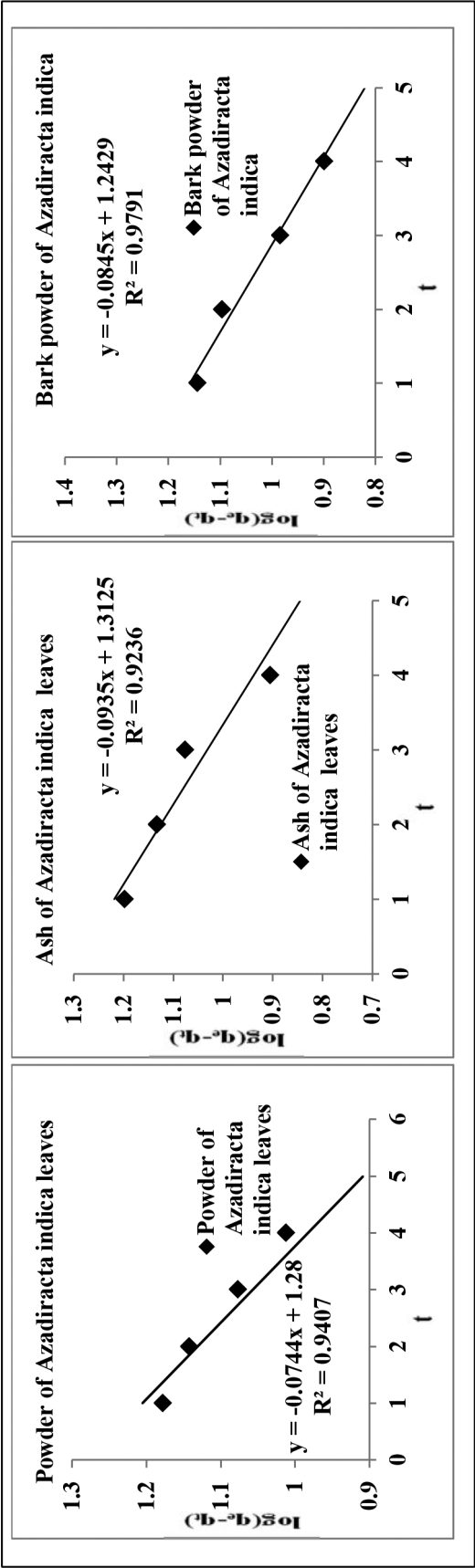


Figure 8: Pseudo first-order kinetics.

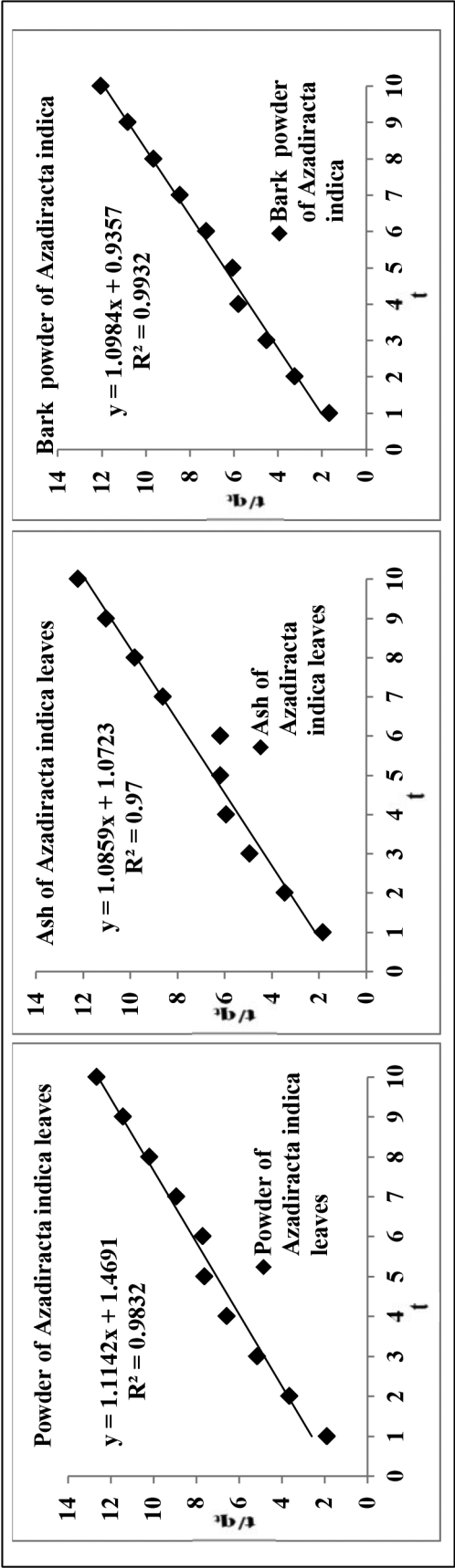


Figure 9: Pseudo second-order kinetics.

Table 3: Pseudo first- and second-order rate constants with the correlation coefficients

Adsorbents	Pseudo first-order			Pseudo second-order		
	Slope	Intercept	R^2	Slope	Intercept	R^2
Leaves powder	-0.0744	1.2800	0.9407	1.1142	1.4691	0.9832
Leaves ash	-0.0935	1.3125	0.9236	1.0859	1.0723	0.9700
Barks powder	-0.0845	1.2429	0.9791	1.0984	0.9357	0.9932

Table 4: Percentage of extractability of nitrites in polluted water samples

S. No.	Water samples	C_i (mg/lit) (initial concentration of nitrite)	C_f (mg/lit) (concentration of nitrite after removal)			% removal		
			A	B	C	A	B	C
1	Sample 1	5.0	0.4300	0.2900	0.1800	91.4	94.2	96.4
2	Sample 2	7.5	0.7050	0.4725	0.3300	90.6	93.7	95.6
3	Sample 3	9.0	0.5670	0.3870	0.2340	93.7	95.7	97.4
4	Sample 4	10.0	0.9500	0.8800	0.7200	90.5	91.2	92.8
5	Sample 5	11.5	0.9430	0.8280	0.5865	91.8	92.8	94.9

A: Powder of *Azadirachta indica* leaves; **B:** Ash of *Azadirachta indica* leaves and **C:** Bark powder of *Azadirachta indica*.

formation. The kinetic data is fitted to pseudo second-order than pseudo first-order model. The procedures developed in this work are applied to polluted waters in water bodies and found to be successful.

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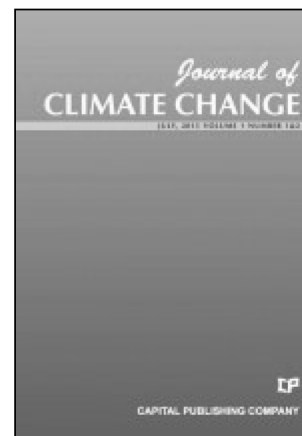
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Aims and Scope

Climate change is reality which deals with the problem of climate variability and change and it deals with descriptions, causes, implications, interactions, impact and responses among other causes. The purpose of the journal is to provide a platform to exchange ideas among those working in different disciplines related to climate variations. The journal also plants to create an interdisciplinary forum for discussion of evidence of climate change, its causes, its natural resource impacts and its human impacts. The journal will also explore technological, policy, economy, strategic and social responses to climate change. It will be peer-reviewed, supported by rigorous processes of criterion-referenced article ranking and qualitative commentary, ensuring that only standard accepted quality work of the greatest substance and highest significance is published.

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