

Wang LI¹, Weiming MO^{2*}, Caiyan KANG^{1,3*}, Minjun ZHANG¹, Mianwu MENG¹
and Menglin CHEN¹

ADSORPTION OF NITRATE FROM AQUEOUS SOLUTION ONTO MODIFIED CASSAVA (*Manihot esculenta*) STRAW

ADSORPCJA AZOTANÓW Z ROZTWORU WODNEGO NA ZMODYFIKOWANEJ SŁOMIE MANIOKU *Manihot esculenta*

Abstract: The performance of a new anion exchanger prepared from *raw cassava straw* (RCS), for the removal of nitrate from aqueous solutions was evaluated in this study. The cassava straw was modified by epichlorohydrin in the presence of pyridine. The influencing factors, adsorption kinetics, and thermodynamics model of nitrate adsorption onto the *modified cassava straw* (MCS) were studied. The results showed that the zeta potentials of RCS and MCS were -20.5 mV and $+37.3$ mV, nitrogen contents (N %) of RCS and MCS were 0.43 and 4.96%, respectively. The best nitrate removal results was reached at 0.2 g of adsorbent dosage and pH range of 6.0–12.0. The modified cassava straw adsorbed nitrate(V) quickly, reaching equilibrium within 30 minutes. The kinetics of nitrate adsorption at different initial concentrations (25, 50 and 75 mg/dm³) all fit a second order reaction. The adsorption rates were controlled by both membrane diffusion and intra-particle diffusion. The adsorption data fit the Freundlich adsorption isotherm and the Langmuir adsorption isotherm. The maximum adsorption capacity was 2.14, 2.00 and 1.81 mmol/dm³ at 293, 303, and 313 K, respectively.

Keywords: adsorption kinetics, nitrate, modified cassava straw, isotherm

Eutrophication is a severely environmental problem in all over the world [1, 2]. Nitrogen-containing compounds such as nitrate(V), nitrate(III) and ammonia are the common pollutants, and especially nitrate(V) is the mainly pollutant for eutrophication. In recent decades, several researches for nitrate(V) removal, such as biological de-nitrification, reverse osmosis, electro-dialysis, breakpoint chlorination and ion exchange are being developed [3-6]. Compared with these methods, adsorption method seems to be a simple, effective, and relatively low-cost method, and nitrate(V) can be recovered [7]. Recently, lignocellulosic agricultural residues such as wheat straw, rice straw, giant reed, coconut coir pith used as raw material to prepare anion exchangers have been intensively reported, which seems a potential way to obtain novel and low cost anion adsorbent [8-12].

¹ School of Environmental Science and Resource, Guangxi Normal University, Guilin 541004, China

² College of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin 541004, China

³ Guangxi Key Laboratory of Environmental pollution control theory and technique

*Corresponding author: kangcy_ok@126.com

In China, Guangxi Zhuang autonomous region is the largest base of cassava (*Manihot esculenta*) production [13]. Annual output of cassava is about 6 million Mg (ton), which is an idea feedstock for fuel ethanol and starch production [14]. Thus, large quantities of cassava residue are generated annually as a major by-product of the starch and fuel ethanol industry. The total annual production of cassava straw estimated in 2006 was 3.6 million Mg (ton) [15]. Much of the cassava residues were burned and discarded without proper utilization. Recent years, researches on preparing functional polymers from agriculture residues have been investigated [16]. Cassava residues could be a raw material for the synthesis of certain kinds of functional polymers [17]. In this paper, we prepare an adsorbent based on *raw cassava straw* (RCS) to remove NO_3^- from aqueous solutions.

Our experiments indicate that the *modified cassava straw* (MCS) is an effective nitrate(V) adsorbent. MCS adsorbent could reduce nitrate(V) pollution from aqueous solutions and effectively utilize cassava residue, which would otherwise go to waste.

Experimental

Preparation of MCS

The modified cassava anion exchanger was prepared from raw cassava straw obtained from Nanning, Guangxi, China. The raw material was washed with water to remove dirt, dried at 80°C and sieved into particles in the range 150–200 μm . The details of the preparation are as follows: 2 grams of the product was dispersed in 100 cm^3 N,N-dimethylformamide (DMF) in a 1000 cm^3 3-neck round bottom flask. A 100 cm^3 aliquot of epichlorohydrin was added and stirred for 1 h at 100°C. Next, a 25 cm^3 pyridine catalyst was added to the solution to accelerate the rate of wetting and extension of the cellulose polymer. The mixture was stirred for 1 h at 100°C. The reaction product was washed with diluted ethanol at 40°C (ethanol:water = 1:1) in order to remove pyridine salts and epichlorohydrin excess. Finally amino groups were introduced into epoxypropyl - by-product after reaction with 33% (w/w) dimethylamine solution (35 cm^3) for 3 h at 100°C. The reaction product was again washed with ethanol (50%) and pure water until the eluant was neutral, and then the *modified cassava residue* (MCR) was dried at 60°C in a drier.

Characterization of MCS

ZETASIZER 3000HSA (Malvern apparatus Ltd, Britain) was used to determine the zeta potential of RCS and MCS.

Element analyzer (PE 2400 Series II, Perkin-Elmer, USA) was used to determine total nitrogen contents (N%) of RCS and MCS, respectively. The *total exchange capacity* (TEC) is calculated using the following equation [18]

$$TEC = \frac{N\%}{1.4} \quad (1)$$

where TEC is the total exchange capacity [mEq g^{-1}] of MCS; N% the total nitrogen of MCR, and 1.4 is the correction coefficient.

Adsorption experiments

Adsorption experiments were carried out in 50 ml nitrate solution placed in a 250 ml conical flask. The flask was agitated in an orbital shaker at 120 rpm and room temperature.

The liquid samples were taken out in a given time interval (60 min). Equilibrium nitrate(V) concentration was determined by means of the rucine-sulfanil colorimetric method [19] with an UV-visible spectrophotometer. The equilibrium concentration in the solid phase, q_e [mg/g] was given as:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (2)$$

where, q_e is the amount of anion adsorption per gram MCS at equilibrium, C_0 and C_e [mg/dm³] are the concentrations of anions at the initial and equilibrium, respectively. V is the volume of the solution [dm³] and m is the amount of MCS [g].

Results and discussion

Characterization of MCS

Table 1 shows the change of element and Zeta potential in RCS in comparison with MCS. The zeta potential of RCS and MCS were -20.5 mV and 37.3 mV, respectively. The chemical groups, such as $-\text{OH}$, $-\text{COOH}$ and $-\text{OCH}_3$, in lignin and cellulose contained in RCS have the negative Zeta potential [20]. According to chemical modification, amine groups from dimethylamine had been introduced to make the Zeta potential of MCS positive. Total nitrogen contents (N %) of RCS and MCS were theoretically calculated as 0.43 and 4.96%, respectively. This shows that the reactions proceed efficiently and lots of amine groups from dimethylamine had been introduced into MCS. According to Eq. (1), total exchange capacity (TEC) of RCS and MCS were 0.31 and 3.54 mEq g⁻¹, respectively. The comparison of total exchange capacity and Zeta potential suggested MCS has a much higher potential in adsorption ability.

Table 1
Changes of element and Zeta potential between RCS and MCS

	N [%]	C [%]	H [%]	Zeta [mV]
RCS	0.43	41.95	6.07	-20.5
MCS	4.96	50.09	7.45	37.3

Effect of adsorbent dosage

The effect of MCS dosage (0.01, 0.05, 0.1, 0.2, 0.4, 0.6, 0.8, 1.0 g) on the adsorption of NO_3^- is shown in Figure 1. When the MCS dosage was 0.01 g, the removal efficiency of NO_3^- was 45.78%. With the increasing of dosage, the total surface and positive charge of adsorbent also increased; it was useful to the removal of NO_3^- through electrostatic interaction. As the dosage increased to 0.2 g, the trend of removal efficiency tended to be stabilized, and the adsorption reached equilibrium at dosage of 0.2 g, with the NO_3^- removal of 97%. So, a dosage of 0.2 g seemed to be optimal and was therefore maintained for the following experiments.

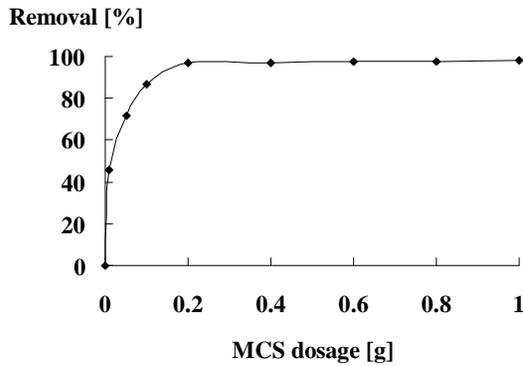


Fig. 1. Effect of sorbent dosage on adsorption (Contact time 60 min, Initial NO_3^- concentration 50 mg/dm^3 , $T = 293 \text{ K}$, $\text{pH} = 6.8$)

Effect of initial pH

An important parameter which controls the adsorption of NO_3^- to MCS is the pH of the solution. The effect of pH was studied over the range 2.0–10.0 by adjusting the pH of $50 \text{ mg/dm}^3 \text{ NO}_3^-$ solutions with dilute HCl and NaOH solutions and then agitated with 0.2 g of adsorbent for 60 min. The removal efficiency of NO_3^- at different pH values (2.0, 4.0, 6.0, 7.0, 8.0, 10.0, and 12.0) is shown in Figure 2. As shown in Figure 2, an increase in the removal of NO_3^- occurred in a range of pH from 2.0 to 4.0. The removal efficiency was maximal and almost constant, when the equilibrium solution pH was between 4.0 and 10.0. The lower removal at pH less than 4.0 is due to competition of chloride ions from the HCl medium, even though the adsorbent surface is positively charged. The NO_3^- removal decrease observed with increasing pH from 10.0 to 12.0 was attributable to increasing the number of negatively charged adsorbent sites, resulting in electrostatic repulsion, and the significant increase of OH^- , which will compete with NO_3^- for adsorption [21, 22].

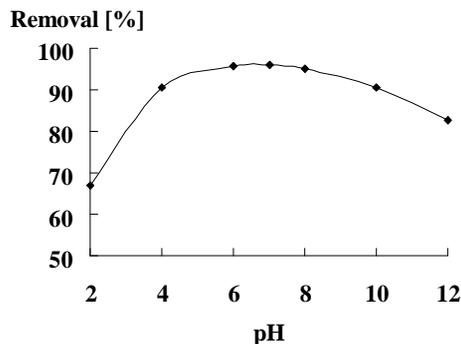


Fig. 2. Effect of pH on adsorption (Contact time 60 min, Initial NO_3^- concentration 50 mg/dm^3 , adsorbent dose: 0.2 g, $T = 293 \text{ K}$)

Adsorption kinetics

The kinetics of nitrate(V) adsorption by MCS were showed in Figure 3. The curves of different initial concentration (25, 50 and 75 mg/dm³) have similar trends. From their shape, the curves can be divided into three distinct as time passes. In the first period ($t < 5$ min), curves increased sharply and the removal of NO₃⁻ is nearly 80%. In the second period ($5 < t < 30$ min), q_t increases slowly. In the third period ($t > 30$ min), q_t was steady and the adsorption reached equilibrium.

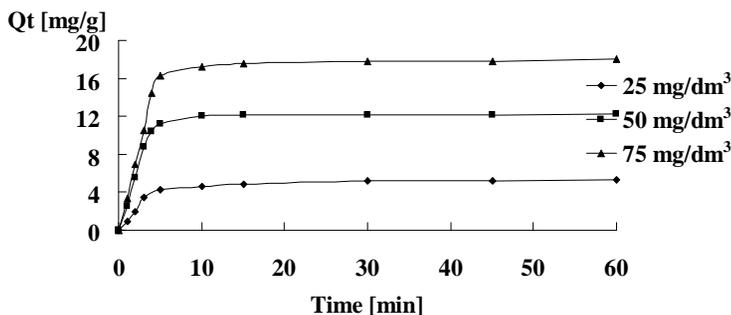


Fig. 3. Adsorption curve of NO₃⁻ under different initial concentrations (T = 293 K, pH = 6.8)

The experimental data was fitted with the pseudo-first-order equation, pseudo-second-order equation and intra-particle equation [23]. The results are depicted in Figures 4, 5 and 6. The fit parameters were listed in Table 2. The pseudo-first-order model is:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{3}$$

Pseudo-second-order equation is:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

Intra-particle equation is:

$$q_t = k_p t^{0.5} \tag{5}$$

where q_e [mg g⁻¹] is the equilibrium concentration of nitrate(V) in MCS; q_t [mg g⁻¹] is the average concentration of nitrate(V) at time t [min] in MCS. K_1 is the pseudo-first-order rate constant [min⁻¹]. K_2 is the pseudo-second-order rate constant [g mg⁻¹ min⁻¹]. k_p is the intra-particle rate constant [g mg⁻¹ min^{-0.5}].

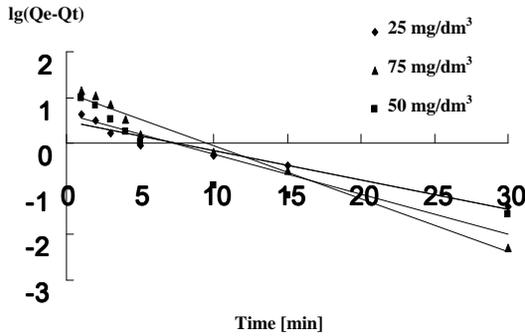


Fig. 4. Lagergren plots (first-order-kinetics) for adsorption (T = 293K, pH = 6.8)

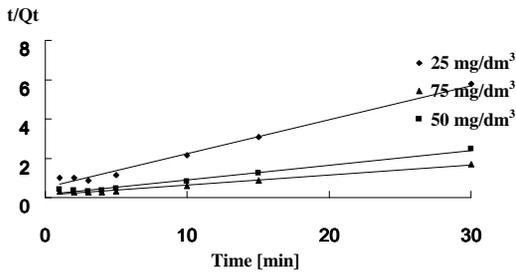


Fig. 5. Lagergren plots (second-order-kinetics) for adsorption (T = 293 K, pH = 6.8)

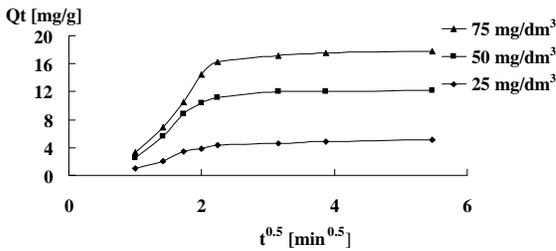


Fig. 6. The Kannan-Sundaram plots for adsorption (T = 293 K, pH = 6.8)

Table 2

Kinetics parameters for adsorption

C_0 [mg dm ⁻³]	Pseudo-first-order		Pseudo-second-order		Intra-particle diffusion equation	
	K_1 [min ⁻¹]	R^2	K_2 [g mg ⁻¹ min ⁻¹]	R^2	K_p [g mg ⁻¹ min ^{-0.5}]	R^2
25	0.155	0.9496	0.0585	0.9881	2.8	0.9682
50	0.213	0.8177	0.038	0.9854	7.299	0.9786
75	0.269	0.9775	0.0187	0.9813	10.824	0.9921

From Table 2, the kinetic data well fitted the pseudo-first-order and the pseudo-second-order model. But the correlation coefficients (R^2) of pseudo-second-order are greater than pseudo-first-order. So, the adsorption process was controlled by pseudo-second-order equation. The Figure 6 is of general type, *ie* intra-particle equation initial curved and final linear portion. The initial curved portions may be attributed to the boundary layer diffusion effect, while the final linear portions may be due to intra-particle diffusion effects. The slope of the linear portion has been defined as a rate parameter (K_p) indicating characteristic of the rate of adsorption in this region where intra-particle diffusion is rate limiting. As shown in the plot, rate parameters (K_p) were obtained as 2.80, 7.299 and 10.824 ($\text{g mg}^{-1}\text{min}^{-0.5}$) for 25, 50 and 75 mg/dm^3 of initial nitrate(V) concentration, respectively. From the figure, it may be observed that the straight line did not pass through the “zero” point and this further indicates that the intra-particle diffusion is not only the main rate-controlling step. So the adsorption process may be of a complex nature consisting of both surface adsorption and intra-particle diffusion [24].

Adsorption isotherms

In the adsorption process, temperature is an important influential factor. Solutions of different initial concentrations - 50, 100, 150, 200, 250, 300, 350, 400 mg dm^{-3} KNO_3 (as NO_3^-) - were used to investigate the effect of concentration on nitrate(V) removal by 0.2 g of adsorbent in 50 cm^3 of solution and shaken in a thermostat at 120 rpm for 60 min at different temperature (293, 303 and 313 K). Figure 7 shows the effect of different temperature (293, 303 and 313 K) on the adsorption of NO_3^- by MCS. The experimental data were fitted with the Langmuir and Freundlich isotherm model equations [25].

The Langmuir adsorption equation in the linear form is:

$$\frac{1}{q_e} = \frac{1}{Q_{\max}} + \frac{1}{bQ_{\max}} \frac{1}{C_e} \tag{6}$$

and the Freundlich equation in the linear form is:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{7}$$

where Q_{\max} is the monolayer capacity of the sorbent [mg g^{-1} or m mol g^{-1}], b is the Langmuir constant [$\text{dm}^3 \text{mg}^{-1}$] or [$\text{dm}^3 \text{mol}^{-1}$] and related to the free energy of adsorption, K_F is the Freundlich constant, n (dimensionless) is the heterogeneity factor. The estimated parameter values for the data gathered in the present study are presented in Table 3.

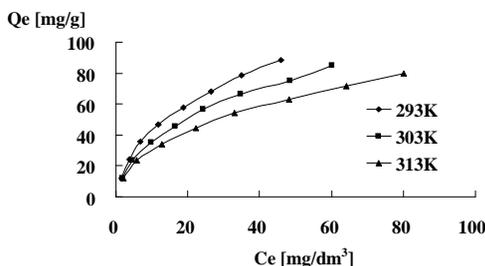


Fig. 7. Equilibrium adsorption isotherms onto adsorbent (293 K/303 K/313 K)

Table 3

Values of Langmuir and Freundlich parameters obtained for the studied systems

<i>T</i> [K]	Langmuir			Freundlich		
	Q_m [m mol dm ⁻³]	b [dm ³ mg ⁻¹]	R^2	K_f	n	R^2
293	2.14	0.0689	0.9829	11.0215	1.78	0.9883
303	2.00	0.0571	0.9724	9.9748	1.8744	0.9939
313	1.81	0.0496	0.9753	9.2329	2.0012	0.9945

From the correlation coefficients listed in Table 3, we found that Freundlich isotherm fit the experimental data better than Langmuir isotherm. The Q_m and K_f of MCS for NO₃⁻ adsorption decreased with the temperature increasing, suggesting that the adsorption reaction was exothermic, $n > 1$ showed that the adsorption of nitrate(V) by MCS is preferable. The adsorption capacity of MCS is compared with some modified adsorbents prepared from agricultural by-product and activated carbons as shown in Table 4. Experimental data shown in Table 4 indicates that MCS is excellent in the adsorption of NO₃⁻. This provides strong evidence for the potential of MCS in applications for the removal of nitrate(V) from aqueous solutions.

Table 4

 Q_{max} in different adsorbents for NO₃⁻ adsorption

Adsorbent	Q_{max} [mmol/dm ³]
MCS	2.14
Modified wheat straw	2.08[8]
Activate carbon	0.11[26]
Amberlite IRA-900	1.15[26]
Rice hull	1.2[16]
Bagasse	1.02[16]

Conclusions

Cassava straw was converted to an anion exchanger by reaction with epichlorohydrin and dimethylamine and the adsorption equilibrium and kinetics were investigated in batch experiment. The results showed the zeta potentials of RCR and MCR were -20.5 mV and +37.3 mV, nitrogen contents (N %) of RCS and MCS were 0.43 and 4.96%, respectively. Compared with other adsorbents, the Q_m of MCS showed that MCS is a potential adsorbent in applications for the removal of nitrate(V) from aqueous solutions.

The adsorption of NO₃⁻ onto MCR was influenced by the dosages, agitation time, pH and temperature. The adsorption was a rapid process, and reached equilibrium at 30 min. The best removal results were achieved at the dosage of 0.2 g and pH of 6.0÷12.0. The Q_m of MCR for NO₃⁻ adsorption decreases with the temperature increasing.

The research results of the equilibrium and kinetics of adsorption of nitrate(V) onto MCS showed that the equilibrium data were described by Langmuir and Freundlich isotherm models respectively, and were found to agree very well with the latter, and the kinetics of nitrate(V) adsorption at different initial concentrations (20, 50, and 75 mg/dm³) all fit a pseudo-second order equation. The adsorption rates were controlled by both membrane diffusion and inter-particle diffusion.

Acknowledgements

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ADSORPCJA AZOTANÓW Z ROZTWORU WODNEGO NA ZMODYFIKOWANEJ SŁOMIE MANIOKU *Manihot esculenta*

Abstrakt: Dokonano oceny wydajności nowego wymiennicza anionowego, wytworzonego ze słomy surowego manioku (RCS), w usuwaniu azotanów z roztworów wodnych. Słomę manioku zmodyfikowano za pomocą epichlorohydryny w obecności pirydyny. Zbadano kinetykę adsorpcji, czynniki wpływające oraz model termodynamiczny adsorpcji azotanów na zmodyfikowanej słomie manioku (MCS). Wyniki pokazały, że potencjały zeta RCS i MCS wynosiły $-20,5$ mV i $+37,3$ mV, zawartości azotu (% N) w RCS i MCS wynosiły odpowiednio 0,43 i 4,96%. Najlepsze wyniki usuwania azotanów uzyskano, wykorzystując 0,2 g adsorbentu, w zakresie pH $6,0 \pm 12,0$. Zmodyfikowana słoma manioku szybko adsorbowała azotany(V), osiągając stan równowagi po 30 minutach. Kinetyka adsorpcji azotu z roztworów o różnych stężeniach początkowych ($25, 50$ i 75 mg/dm³) wskazuje na reakcję II rzędu. Szybkość procesu adsorpcji kontrolowała zarówno dyfuzja membranowa, jak i dyfuzja cząsteczkowa. Dane doświadczalne opisano za pomocą modeli izoterm Freundlicha i izoterm Langmuira. Maksymalne pojemności sorpcyjne wynosiły 2,14, 2,00 i 1,81 mmol/dm³ w temperaturach odpowiednio 293, 303 i 313 K.

Słowa kluczowe: kinetyka adsorpcji, azotany, modyfikowana słoma manioku, izoterma