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Chitosan-coated Homoionic K-clinoptilolite: A solution for Sustainable Agriculture via Simultaneous Slow Potassium Release and Water Desodication

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Abstract: Increasing salinity in agricultural land poses a serious challenge to sustainable agriculture, emphasizing the urgent need for innovative soil and water management strategies. This study tested chitosan-coated homoionic K-clinoptilolite (0%, 10%, and 20% chitosan, referred to as K-C, $Ch_{10}K-C$, and $Ch_{20}K-C$, respectively) as an alternative solution for slow potassium release and water desalination. Clinoptilolite, a natural zeolite with ion exchange properties, was used as an effective potassium fertilizer after undergoing a K-homo-ionization process followed by chitosan coating.

The chitosan coating acts as a biodegradable layer that slows the release of potassium ions, ensuring sustained nutrient availability for plants. Moreover, this combination shows potential for reducing soil sodicity by selectively releasing potassium while simultaneously adsorbing sodium ions from saline water. This process improves soil structure and enhances crop yields. Laboratory tests demonstrated that it not only prevents excessive potassium release, which could exacerbate salinity, but also significantly reduces sodium content, contributing to environmental resilience. These findings indicate that $Ch_{10}K-C$ and $Ch_{20}K-C$ are sustainable solutions for managing saline irrigation water and saline soils, offering a pathway toward environmentally friendly agricultural practices.

The study utilized adsorption isotherm models for Langmuir and Freundlich, to evaluate the factors influencing Na⁺ adsorption by ChK-Cs in water.

The results showed that the Freundlich model, as long as the most highly suited for the experimental data, demonstrate a heterogeneous adsorption process. Additionally, the adsorption kinetics followed a pseudo-second-order model, demonstrating the efficiency of ChK-Cs in removing sodium ions.

In conclusion, homo-ionized clinoptilolite coated with chitosan serves as an excellent adsorbent to reduce sodicity, making it an ideal choice for sustainable agriculture. These materials offer a promising approach to mitigating the challenges posed by salinity in agricultural practices while promoting sustainable resource management.

Keywords: : Na+ Adsorption; Soil sodicity reduction; Slow-release fertilizer; Sustainable agriculture; Chitosan-coated Clinoptilolite.

1-Introduction:

In the pursuit for sustainable agricultural practices, managing of nutrient delivery and soil health has become increasingly critical, especially in regions challenged by soil salinity and nutrient deficiencies. Chitosan-coated homoionic K-Clinoptilolite emerges as a promising solution that addresses these intertwined challenges through its innovative

Received:30/11/2024 Accepted: 9/2/2025 application in controlled potassium release and saline water desodication [1].

Clinoptilolite, a natural zeolite, possesses unique ion-exchanging properties, making it an ideal medium for nutrient retention and release. When homoionic with potassium (K), can significantly enhance potassium availability to plants, a vital macronutrient essential for various physiological processes [2]. However, the efficacy of potassium release can be further optimized through a chitosan coating, a biopolymer derived from chitin. This biodegradable coating not only shields the zeolite from rapid leaching and environmental degradation but also facilitates the gradual and controlled release of potassium [3].

Moreover, using saline waters for irrigation is becoming necessity in arid and semi-arid regions, where freshwater scarcity is prevalent Chitosan-coated homoionic [4]. K-Clinoptilolite can aid in desalting saline water. thereby rendering it more suitable for agricultural use. The cation-exchange capacity of this biocomposite allows it to attract and sodium ions while simultaneously bind releasing potassium ions, contributing to improved soil structure, enhanced nutrient uptake, and reduced salinity levels in the soil [5] [6].

This innovative approach not only underscores the potential of utilizing natural resources for sustainable agricultural practices [7] but also aligns with the principles of circular economy by employing biodegradable materials that minimize environmental impact [8]. As agricultural demands continue to escalate in a changing climate, the integration of chitosan- coated homoionic K-Clinoptilolite represents a forward-looking strategy aimed at promoting resilient and productive farming systems while preserving the integrity of our ecosystem [9].

2. Materials and methods

2.1. Materials:

Clinoptilolite was obtained from Eurofins GfA lab service GmbH (Germany manufacturing), Chitosan from General Drug House (P) Ltd in India. At the same time Potassium chloride and other chemicals were procured from El-Gomhouria Company for Drugs in Egypt. To prepare the aqueous standard solutions of the required compounds, the salt was dissolved in distilled water.

2.2. Instrumentals:

instrumental	• Description		
Flame Atomic	Model 3300 from Perkin		
Absorption	Filmer USA was used for		
Spectrometer	elemental analysis of Na and		
(FAAS)	K ions		
(IAAS)	For pH massurements (WPA		
pH-meter	Linton Cambridge UK)		
	where Clinoptilolite samples		
	ware subjected to (XPD)		
	analysis using Cu K radiation		
X-ray diffraction	and a Ni filter at constant		
(XRD)	voltage 40 kV and 30 mA		
	using Empyrean Powder		
	Diffractometer		
	The Chemical analyses for the		
	major oxides (mass %) and		
	some minor elements of the		
	somplas have been performed		
X-ray fluorescence	using (XPE) on a modern		
spectrometry	using (ARI) on a modern		
(XRF)	spectrometer (Avios WD		
	VDE Spectromator		
	DA Nalytical 2005		
	r Alvalytical, 2005, Notherlands)		
	(ETID) and a true record and		
	(FTIR) spectra were recorded		
Fourier transform	Eichen Scientifie (Welthem		
infrared (FTIR)	Fisher Scientific, (Waltham,		
spectra	What was a set of the		
	ABr periet at approximately		
	The Remen shift of the		
	applaced and a sellected on an i		
	Raman Plus 532S portable		
portable laser	laser Raman spectrometer		
Portable faser	using a BAC151C Raman		
spectrometer	microscope optical sample		
spectrometer	inspection system (B&W		
	TEK USA) and 20		
	100X lenses		
	(Ver. 7.04 Serial Number:		
	MAL 107/157 Malvern		
	Instruments I td. I ondon		
	United Kingdom) was used to		
Zetasizer NANO- ZS	optimize the formulations of		
	suspensions, emulsions and		
	protein solutions predict		
	interactions with surfaces and		
	ontimize the formation of		
	films and coatings		
	model (TGA PT 1000 lineais		
	USA was used for measuring		
Thermogravimetric	weight change (loss or gain)		
analysis (TGA)	and the rate of weight change		
	as a function of temperature		
	time and atmosphere		
	unic, and aunosphere.		

2.3. Methods

2.3.1. Chitosan Refining.

A refining step in the chemical processing of chitosan was employed by the dissolution of 50 g in 200 ml 5% acetic acid. Then adding a KOH solution to ensure complete precipitation of the chitosan. The filtration process will be occurred for the solution, also will be followed by drying process for the precipitated chitosan, at 110°C [10].

2.3.2. Homo-ionization technique

The raw Clinoptilolite was ball-milled and sieved to obtain a -112-micron portion (50%) coded as UnC.

It underwent a pretreatment process to replace exchangeable ions with potassium as a beneficial ion for plant growth, which may help reduce the sodicity of irrigation water when released from the homo-ionized clinoptilolite to the aqueous phase.

A mass (m) of 10 g of UnC was mixed with a volume (V) of 100 mL of a 1M KCl solution, and the suspension was shaken for 48 hours, and then centrifuged. The supernatant solution was discarded and stored for metal analysis. Another fresh 100 mL of 1M KCl solution was added to the centrifuged UnC, and the saturation step was repeated four times. The final centrifuged UnC was washed with distilled water until a chloride-free filtrate was obtained using the AgNO₃ test. Then, the Khomo-ionized clinoptilolite was dried at 70°C up to a constant mass. The resulting product was coded as (K-C).

The second step involved forming the Chitosan-coated homoionic K-clinoptilolite product, which was coded as (ChxK-C), by adding x% of the precipitated water-soluble chitosan to the (K-C) product (x=0, 10 or 20%).

The flame atomic absorption spectrometry (FAAS) was used for Na^+ concentrations (C, in mg L⁻¹) measurement in the supernatant liquors for each batch were purposeful using [11] [12].

The released metal content (M, mg g⁻¹) was calculated by:

 $M = \frac{C(mg L^{-1}) . V(L)}{m(g)} mgg^{-1} \quad (1)$

the leached Na ions Cation exchange

capacity (CEC, meq per 100 g) was calculated using 1M KCl by Eq (2):

$$CEC = \sum \frac{M \text{ of the metal ion}}{atomic \text{ mass}} .100 \quad (2)$$

2.3.3. Adsorption studies

To analyze HCl-leachable elements, 0.1 g of the Clinoptilolite samples were shaken in100 ml of 1M HCl at 50°C for 24 h, then the suspensions were filtered. The metal content (M, mg g-1) was set and measured by Eq (1).

2.3.4. Effect of initial concentrations

The effect of initial concentrations (C_0) was examined at room temperature by adding 0.1 g of each adsorbent with 20 mL of 50-400 mg L⁻¹ of Na⁺ aqueous solutions as NaCl. Then, the suspensions were shaken for six hrs. and left for equilibration for two days. Suspension's proceeded, centrifuging was where the equilibrium for of Na (Ce) in concentrations the supernatant liquors were determined by FAAS.

2.3.5. Effect of contact time

Shaking time effect for each adsorbent, was studied by taking 0.1 g from the adsorbent and make it suspended in 20 mL of initial concentration 100 mg L⁻¹ of Na⁺ aqueous solutions at room temperature. Where The suspensions were shaken for the time intervals of 10-120 minutes. Then, the centrifugation process was occurred for the suspensions, and final concentrations of Na (C_t) in the separated liquors were determined by FAAS.

2.3.6. Mathematical calculations

2.3.6.1. Adsorption isotherm model fitting

The Langmuir and Freundlich adsorption models [13] were used to fit the experimental and that express by Eq. 3:

$$\frac{C_{e}}{q_{e}} = \frac{1}{K_{l} q_{max}} + \frac{1}{q_{max}} C_{e} (3)$$
Where:

- qe,Na⁺ quantity adsorbed per unit mass of adsorbent at equilibrium (mg g^{-1}).

- q_{max} is the maximum adsorption capacity at monolayer coverage (mg g $^{\text{-1}})$

- Ce, is the equilibrium concentration in solution (mg L^{-1})

- K_L , the Langmuir constant related to adsorption energy (Lmg⁻¹).

- q_e , the equilibrium adsorption capacity, was calculated by the equation:

$$q_e = \frac{V(C_0 - C_e)}{m}$$
 (4)

By graphing, we determine the relation and values of Langmuir constant and the maximum adsorption capacity, where the slope and intercept of the linear plots of (C_e/q_e) versus C_e can obtain the value of q_{max} and b, which gives a straight line of slope $1/q_{max}$, corresponding to a complete monolayer coverage (mg g⁻¹) and the intercept is $1/(K_Lq_{max})$.

the Langmuir isotherm essential characteristics and the feasibility can be shown in terms of a non-dimensional equilibrium factor R_L that is calculated by Eq. (5):

$$R_L = \frac{1}{1 + C_0 K_L} \qquad (5$$

Freundlich linear equation form is expressed by Eq. (6):

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{6}$$

Where, Freundlich isotherm adsorption constants, $K_F~(dm^3~g^{\text{-1}})$ and n respectively referee to:

- the adsorption capacity

- adsorption intensity

The intercept value of K_F can be obtained from the values of K_F and the slope 1/n which generated from the plot of ln q_e versus ln C_e

The Dubinin–Radushkevich-Kaganer isotherm is given by [14]:

$$\ln q_e = \ln q_m - K_{D-R} \varepsilon^2 (7)$$

Where,

- Polanyi potential (ε) was expressed by

$$\varepsilon = \operatorname{RT} \ln \left(1 + 1/C_{e} \right).$$

- theoretical adsorption capacity is q_m.

- the absolute temperature is T in Kelvin,

- universal gas constant is R, 8.314 (J mol $^{\text{-1}}$ K $^{\text{-1}}$).

- constant related to the adsorption energy is KD-R

- constant KD-R used for determination The mean free energy of adsorption (E) [15]by the relation [8].

$$E = \sqrt{\frac{1}{2K_{D-R}}} \quad (8)$$

2.3.6.2. Fitting of kinetic models

Kinetics adsorption experimental data for Na^+ on the adsorbents was examined by the pseudo-first-order and pseudo-second-order kinetic models [16].

Where,

- Pseudo 1st order model was shown in Eq. (9):

$$\ln(q_e - q_t) = \ln q_e - K_1 t$$
 (9)

Pseudo
$$2^{nd}$$
 order model was in Eq (10):

$$\frac{t}{q_{t}} = \frac{1}{K_{2} q_{e}^{2}} + \frac{1}{q_{e}} t \quad (10)$$

Where,

- the pseudo 1^{st} order adsorption constant rate ((g mg-1 min-1) was expressed by k_1

- the pseudo 2^{nd} order adsorption constant rate (min-1) is expressed by k_2 .

- the amount of Na+ adsorbed (mg g^{-1}) was expressed by qe and q_t, at equilibrium and at time t, respectively.

- By using the pseudo 2^{nd} order equation, the values of qe and K_2 can be calculated by plotting (t/qt) versus (t).

- the initial adsorption rate, h (mg g-1 min-1) can be calculated according to Eq (11):

$$h=K_2 q_e^2 (11)$$

where, the model of Weber-Morris equation) [17], can be used for The intra-particle diffusion rate calculation according to Eq.(12):

$$q_t = k_{ip}t^{1/2} + C_i$$
 (12)

- the amount of Na⁺ adsorbed (mg g⁻¹) at time t, was expressed by q_t (mg g⁻¹) at time t.

- the intra-particle diffusion rate constant at an stage i is expressed by K_{ip} .

- the square root of time t1/2 in minutes

- intercept of the linear expressed by C_i (mg g^{-1}) from the plot of Eq. (10).

3. Results and discussion

The raw Clinoptilolite obtained from biotraxx company, was reported to contain the following constituents: 68.17% SiO₂,11.05% Al₂O₃,0.64% Na₂O,3.93% CaO, and 1.11% K₂O, as well as major and trace constituents

[18]. After treatment with 5M HCl at 70°C, the minor constituents dropped to 0.06%, 0.03%, 0.02%, and 0.08%, respectively. Given the harsh treatment conditions, the lost ions are not assumed to be ion exchangeable, carbonate, or structural contents. To define the extractable ions of UnC, milder procedures were followed using 1M KCl [19]. The leached ions with KCl account for the exchangeable ions only. These results were analyzed by XRF instrumental and are illustrated in Table 1.

Table (1) Analysis of Na⁺ and K⁺ in K-C which leached with 1M KCl.

Compound	K-C
SiO ₂	74.10%
TiO ₂	0.11%
Al ₂ O ₃	12.84%
Fe ₂ O ₃	1.44%
MnO	0.04%
MgO	0.92%
CaO	1.58%
Na ₂ O	0.12%
K ₂ O	6.99%
P_2O_5	0.04%

The CEC value was modified to be (5.12. m_{eq} per 100 g) for Na⁺ ion.

3.1. Adsorption studies

3.1.1. Effect of initial concentration

The effect of the initial concentration on the adsorption isotherms of Na⁺ on the three designed materials as K-C, Ch₁₀K-C and $Ch_{20}K-C$ was shown in Figures (1), (2) and (3), where figures (2)and (3) presented the modification of Clinoptilolite by chitosan at different concentration. The characteristic of **S**3 according adsorption isotherms type classification proposed for liquid-solid interactions by Giles et al. [19]. The large negative contributions of the solvent or a second solute give indication about the activation energy for desorption of the solute which concentration-dependent.

which is expected for ion exchange on microporous solids. In addition, it was reported that the dielectric constant of Clinoptilolite decreases while its equilibrium volume increases with the increase in the electrolyte concentration, exposing a larger surface for the ion exchange process [20].



Figure 1. Adsorption isotherms of Na⁺ on K-C



Figure 2. Adsorption isotherms of Na^+ on Ch10K-C.



Figure 3. Adsorption isotherms of Na^+ on Ch20K-C.

The adsorption isotherms were studied two adsorption isotherm models: Freundlich and Langmuir models. Where Table (2) summarizes the parameters of the Freundlich and Langmuir models. The R2 values indicated the low fitness of the studied adsorption models for Na+ on K-C and N-K especially, using Langmuir isotherm model, which may be explained given the ion exchange nature of the process that does not resemble an adsorption process as illustrated in Eq. (13): K^+ hydrated Clinoptilolite + Na⁺ hydrated, aq $\overrightarrow{}$ Na⁺ hydrated- Clinoptilolite + K^+ hydrated, aq (13).

In contrary, Freundlich fitted well with the K/Na ion exchange process giving linear correlation coefficient (R2) > 0.979.

Table2. Isotherm model special characteristicsfor the adsorption of Na^+ on mentionedClinoptilolite models.

Freundlich isotherm parameters						
Adsorbent	l/n	$K_{\rm F} ({\rm mg/g})$	\mathbf{R}^2			
K-C	0.773	0.1621	0.9796			
Ch ₁₀ K-C	0.8525	0.0959	0.9834			
Ch ₂₀ K-C	0.7577	0.1725	0.9966			
Langmuir isotherm parameters						
Adsorbent	$q_{max} (mg g^{-1})$	$K_1 (mg/g)$	\mathbf{R}^2			
K-C	20.74688797	0.006262424	0.9558			
Ch ₁₀ K-C	-6.779661	-0.001598	0.5608			
Ch ₂₀ K-C	-89.28571	-0.000289	0.0058			

However, the best models that describe the adsorption of Na^+ on K-C, $Ch_{10}K$ -C and $Ch_{20}K$ -C are the Freundlich model and indicate that these adsorbents are physically adsorbed. The values of 1/n indicate favorable adsorption of Na^+ on the previous Clinoptilolite models within the studied ion concentrations.

This indicates the noticeable enhancement of the kaolin CEC that may be explained by the better polarizability of the heavier and larger size of the hydrated sodium ion compared with potassium or ammonium hydrated ions. By Contrast the adsorption capacity of Na⁺ on K-K to several lately reported adsorbents (Table 3), it can be ended that K-K has a modest capacity, However, it is specified by its quite inexpensive cost in addition to its simple treatment method and the importance of the exchanged potassium to plants.

3.1.2. Effect of time of shaking

The effect of time of shaking on the adsorption of Na^+ by the homo-ionized Clinoptilolite at the three different models was investigated at an initial metal concentration of 100 mg L⁻¹ during interval shaking time 10 – 1440 min (Fig. 4).

Which seen the removal of metal ions by different Clinoptilolite models was increased with increasing the time of shaking to reach its equilibrium at 120 min, yielding removal efficiencies average for all models of 18 to 26%.

Adsorption kinetics of Na⁺ ions by homoionized three Clinoptilolite models were studied following the pseudo-first order, pseudosecond order, and intraparticle diffusion models described above. From These results, The best description for kinetic model adsorption for Na^+ homo-ionized ions on the three Clinoptilolite is pseudo-second order kinetic model with a high correlation coefficient that's equal to one or other closed data to 1; This assumes that the taking of Na⁺ ions can be proceeded bv a heterogeneous sorption mechanism.

Table3. Comparability between the removal capacity of Na⁺ using K-K and N-K with other adsorbents.

Adsorbent	q _{max} mg g ⁻¹	t _{eq} min	Ref.	
activated coconut coir	43.7	20	[21]	
TOA	4.1	120	[22]	
activated carbon	134.2		[23]	
amorphous carbon	135.45	60	[24]	
activated zeolite	67.5%		[25]	
K-K	75.6	360	[26]	
N-K	17.5	360	[20]	
K-C	20.74	120	Present work	

 t_{eq} = equilibration time.

TOA = tetraoctylammoniummonensin.





Table 4- Kinetic model constants and correlation coefficients for Na⁺ adsorption on different Clinoptilolite models.

Pseudo-first order					
Adsorbent		q _{e, exp}	k1	q _{e,}	\mathbf{R}^2
		(mg/g)		$_{cal}(mg/g)$	
K-C		6.55	-0.0004606	0.357	0.168
					8
Ch ₁₀ K-C		5.25	13.818	0.88450	1E-05
				4405	
Ch ₂₀ K-C		5.74	0.00006909	2.42158	0.645
				65	5
Pseudo-second order					
Adsorben		Н	k ₂	q _{e,}	\mathbf{R}^2
t				_{cal} (mg/g)	
K-C	K-C -1.5246		-0.044947233	5.82411	1
				1823	
Ch ₁₀ K-C	-		-25.17263301	0.01148	0.463
	0.003319			3825	4
		722			
Ch ₂₀ K-C		-	-22.89667832	0.01545	0.700
	0.000547			7862	9
105		105			
Intraparticle diffusion					
Adsorbent k _{ip}		k _{ip}	Ci	\mathbf{R}^2	
K-C -		6.474	0.8861		
		0.0173			
Ch ₁₀ K-C 11		11.896	-36.976	0.5887	
Ch ₂₀ K-	C	9.244	-21.922	0.7961	

Conclusion:

Natural clinoptilolite was homo-ionized with K⁺ and coated by chitosan to obtain three different clinoptilolite modules C-K,

Ch₁₀K-C and Ch₂₀K-C, respectively. All the homo ionized clinoptilolite models were successfully utilized as adsorbents for the simultaneous removal of Na⁺ ions from aqueous solution and release of K^+ by batch adsorption method. The adsorption process was found to be dependent on initial concentration of Na⁺ ions and contact time. The metal uptake increases with increasing the initial concentration probably due the concomitant increase in clinoptilolite volume and decrease in the dielectric constant.

Optimum removal of metal ion was obtained at contact time of 120 min. The equilibrium data were best fitted with the Freundlich adsorption model. Pseudo-second order equation provided the best fit the to experimental data.

The maximum capacities (q_{max}) are found to be enhanced to 6.55, 5.25 and 5.74mg g^{-1} for K-C, Ch₁₀K-C and Ch₂₀K-C respectively.

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