Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

CHLORIDE REMOVAL FROM MDEA VIA CO-PYROLYSIS OF LEONARDITE AND MG-AL CALCINED LAYERED DOUBLE HYDROXIDE ALONG WITH SODIUM STABILIZATION, USING ECO-FRIENDLY ADSORBENT

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Abstract

The chlorine content in Methyl diethanol Amine (MDEA) solution used for the gas sweetening purpose, caused operational difficulties such as amine loss and foaming multiplicity as well as corrosion rate enhancement. This study represents the chloride uptake capacity of Leonardite char activated by calcined double-layered hydroxide in a Methyldiethanol amine solution. The adsorption Kinetic and isotherm were fitted to pseudo-second order and Freundlich models respectively. The effect of adsorbate dosage and co-pyrolysis temperature on MDEA dechlorization was tested. Batch equilibrium experiments at ambient conditions revealed that the new absorbent uptake capacity might be up to 41.2 mg/g of Leo/MgAl-CLDH (co-pyrolysis temperature: 500°C). For pH stabilizing after amine dechlorization, zein protein coated on the sand as a low-cost and biodegradable metal adsorbent was investigated. The FTIR spectral analysis shows stretching vibrations due to the bonding of zein with sand which indicates the success of bio-coating.

Keywords: Amine dechlorization, Cation Stabilizing, CLDH, Leonardite char, zein Coated Sand

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1 INTRODUCTION

Besides further development and new techniques for sour gas sweetening purposes such as nanofluid contacting, poly membrane, and catalytic conversion[1-3] still MDEA adsorption is widely used worldwide. This method suffers from high energy consumption and the effectiveness of reaction medium by ionic pollutants. The concentration of anionic spices in amine solution, especially chloride, leads to an increase in the rate of heat-stable salts formation as an undesirable factor which comes with amine loss, foaming multiplicity, and rising corrosion rate.[4-7] As a result, the investigation of new, economical, and eco-friendly techniques for MDEA dechlorization is justifiable. Techniques such as electromagnetic (EM) and electrodialysis (ED) for HSS (Heat Stable Salt) separation, are generally costly.

While EM is highly dependent on the amine flow rate through cells,[8] the temperature and amine concentration in ED, are limiting agents.[9] The integrated hydrate-based gas separation and chemical absorption for the sweetening of natural gas with high H_2S and CO_2 contents was presented by Liu et al.[10] The technic meets the sale gas specification and consumed less energy than traditional method. This method suffers from low operating temperature range. Solvent extraction for removal of formate, glycolate, and oxalate by 2-ethyl-1-hexanol as a diluent was reported to be very well in MDEA and AMP CO_2 capturing systems.[11] The conventional method for separating heat-stable salts from amine solutions is thermal reclaiming. Which is done in the form of atmospheric pressure and vacuum conditions. Thermal reclaiming must be considered a conventional but high-energy

Eur. Chem. Bull. 2023,12(3), 2685-2692

2685

Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

consumption method that consists of stripping and neutralization steps to reduce amine carbamate loading and recover protonated amine.[12]

Ion exchange has been extensively used for amine dechlorination and HSS separation because it avoids design and operation complexities. The pH of the industrial amine solution has a significant impact on the effectiveness of commercial polymers used to remove organic acids and dechlorinate the solution.[13,14] To increase the effectiveness of resin renewal, HyungCho et al.[15] investigated the use of zirconium pentahydroxide solution in place of traditional NaOH. The alginate/clay composite and bentonite were used by Edathil et al.[16] to remove organic seasonings from an industrial amine system.

Natural sorbents such as Leonardite charcoal have great potential as a suitable option in the dechlorination of MDEA solution due to their non-toxicity, availability, reasonable price and finally being environmentally friendly. Kurkova et al.[17] have proposed that the adsorption potential of natural coal such as Leonardite is due to the oxygen functional group in the coal structure. Humic is the dominant substance in the Leonardite structure[18] which is responsible for the active carboxylic and hydroxylic functional group as well as loctonic and phenolic which is connected to aliphatic or aromatic carbon in the structure. Surface oxygen causes electrostatic interaction between Leonardite and the adsorbate.[19]

By converting the included organic elements to activated carbon, carbonized leonardite would have better adsorption qualities. By using Leonardite char made at 4500° C, Chammui et al.[20] looked into the sorption of arsenic from a sample of groundwater. It investigated how conflicting anions affected arsenic sorption. To adsorbe organic pollutants from heavy oil waste water and regulate COD and total salts, Tang et al.[21] used lignite-activated coke. On carbonized Leonardite, the Congo red sorption was suggested by Ausavasukhi et al.[19] The reaction nature is endothermic and spontaneous, and the sorption process adheres to the pseudo-second-order model.

Another attractive choice for anionic pollutants removal from an aqueous solution might be based on layered double hydroxide (LDH) minerals, especially after reconstruction by calcination (CLDH). Lv[22] investigated the use of calcined MgAl-CO3 layered double hydroxide for drinking water deflouridation. The exothermic nature of the reaction was also proposed as well as adsorption pH dependent. Theiss et al.[23] also emphasized the LDH thermally activation to improve the anionic spices (BF_4^-, Cl^-, Br^-, …) adsorption capacity. As described by Bontchev et al.[24] the term "memory effect" which deals with LDH crystal structure collapsing and being amorphous by heating at a certain temperature and then recovering by imprisoning target anions in the solution, justifying the CLDH adsorption potential. Gibb et al.[25] proposed the use of dual adsorbent to salinize groundwater. Naturalizing the groundwater after dechlorization by CLDH was performed via Na^+ adsorption on acid-treated clinoptiolite. Xu et al.[26] investigated the effect of humic acid on borate sorption via Mg-CLDH and Ca-CLDH to provide a prediction model to study the effect of different reactive groups in the humic acid structure. Phosphate and fluoride removal from water by synthesizing mesoporous MgAl-LDH composites was proposed by Li et al.[27] The sorbent, due to high magnetic properties could be easily recovered from solution and regenerated by $NaNO_3$-NaOH solution for further usage. Co-pyrolysis of rice husk and Mg-Al layered double hydroxide to form a new eco-friendly adsorbent to remove phosphate from the aqueous system and the effect of pyrolysis temperature and Mg:Al molar ratio on adsorption properties of synthesized adsorbent has been reported.[28]

We used zein protein as a low-cost and environmentally friendly metal adsorbent because the solution's pH increased after dechlorination due to a rise in sodium ion concentration and the need to stabilize the amine pH in the range advised by the designer of the gas sweetening process. Zein is a protein found in maize that is alcohol-soluble but insoluble in water, acetone, and anhydrous alcohols other than methanol.[29] Zein's amino and carboxyl groups give it the ability to accept and remove other substances.[30] The use of zein in the manufacture of membranes with the ability to absorb heavy metals for industrial wastewater treatment has been reported. Forward osmosis membranes based on polyethyleneimine and zein were made which have a high potential for water permeation while having selective rejection ability towards heavy metal ions.[31] Thanawatpoontawee et al.[32] studied iron-loaded zein beads for arsenic (V) removal from water. The effects of iron loading concentration and solution pH on absorption efficiency were investigated. Deng et al.[33] prepared a zein/gelatin nanofibrous membrane for

Eur. Chem. Bull. 2023,12(3), 2685-2692

2686

Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

copper absorption. Nano-hydroxyapatite improves the membrane's mechanical properties. Teng et al.[34] improved zein oil sorption capacity and mechanical properties by adding polyvinylidene fluoride (PVDF) to zein fiber via electrospinning.

This study presents chloride removal from MDEA solution by Leonardite char activated with Mg-Al calcined double hydroxide via co-pyrolysis s as a low-cost and efficient adsorbent. Due to active negatively charged groups in the Leonardite structure, the binding affinity of the carboxylic group to anionic species especially Cl$^-$ is limited. The addition of CLDH to the adsorbent greatly reduces this limitation. To stabilize the concentration of the released sodium ion and adjust the pH of the solution within the range recommended, sand covered by zein protein has been used as a biodegradable and low-cost absorbent.

2 MATERIALS AND METHODS

2.1 Adsorbents suppliers and specification

Pure Methyl diethanol amine (MDEA) solvent originated from Himsorbent^{TM}, Sodium chloride was obtained from Scharlau^{TM}. Zein was purchased from SIGMA^{TM}. The sand was collected from Siraf port beach Bushehr-Iran, washed with distilled water, and dried before experiments. $Mg(NO_3)_2.6H_2O$ and $Al(NO_3)_3.9H_2O$ were obtained from Xilong chemical. Na_2CO_3 and NaOH were purchased from MeRCK. The purchased chemicals all are in analytical grade.

The Metrohm^{TM} 848 Titrino Plus was used to determine Cl$^-$ concentrations. This might be interpreted as an automatic titration method that detects the formation of AgCl precipitation with potentiometer variation detection using the titroid electrode. The AVATAR thermo Fourier transform infrared spectroscopy determined the success of bio coating on sand using FTIR analysis. The inoLab pH 7110 WTW was used for pH measurement, and DOMEL^{TM} CENTRIC 90 centrifugal was used for solubility and separation studies. A vacuum quartz furnace (Zhengzhou TCH instrument, SK-G03123K) was used for co-pyrolysis purposes.

2.2 Adsorbents synthesis

Leonardite char activated by hydrotalcite $[Mg_6Al_2(OH)_{16}.4H_2O]$ synthesized based on

Zhao et al.[35] constant pH method. LDH by Mg/Al ratio of 3 was prepared. 9.6 gr $Mg(NO_3)_2.6H_2O$ and 3.2 gr $Al(NO_3)_3.9H_2O$ was dissolved in 22.5 ml deionized water to form solution A. NaOH (20.48gr) and Na_2CO_3 (6.4gr) was added to 22.5 ml deionized water to prepare solution B. to motivated Leonardite by LDH and co-pyrolysis, based on the idea taken from Chammui et al.,[20] 2,5 gr Leonardite (sieve size:80 µm) added to 20 ml stirring deionized water. Solution A and B were then added simultaneously dropwise to the Leonardite mixture at such a rate that, the pH of the mixture was constant at 9.5. The mixture must keep at 60º C for 24 hours. Samples were then collected and washed with deionized water and dried in an electrical oven at 110º C for 24 hours. Samples were co-pyrolysis in a vacuum quartz furnace (Zhengzhou TCH instrument, SK-G03123K) at 400º C, 500º, C, and 700º C for 2 hours. The heating rate and N_2 flow were selected based on the manufacturer's recommendation of 15º C/min and 100 ml/L.

1 gr zein was added to 50 ml of a 60% ethanol solution and stirred for 12 hours. The solution received 10 g of sand and was left stirring overnight. The absorbent must be washed with distilled water and dried.

3. RESULTS AND DISCUSSION

The test samples were prepared from fresh MDEA with a concentration of 45% by weight and the determined amounts of sodium chloride with analytical grade (MeRCK, Germany) was added to it. Batch adsorption experiments were performed by mixing a predetermined amount of adsorbent with saline amine solution at 120 rpm and a desirable temperature .After 24 hours and reaching equilibrium, the aqueous phase is separated by a centrifuge. The removal percentage R and the equilibrium concentration q_e were calculated as
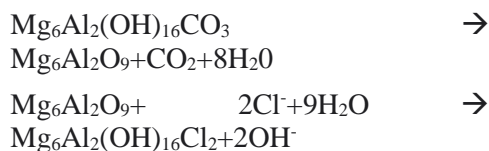
$$R = [(C_0 - C_e)/C_0] \times 100$$

$$q_e = (V/m)(C_0 - C_e)$$

where C_0 (mg/L) is the chloride initial concentration in solution and C_e (mg/L) is its equilibrium concentration. m (g) denotes the Leo-CLDH mass and V (L) is the volume of the solution.

Based on the flow dynamics, the adsorption mechanism can be of the film or cavity penetration type. In this regard, the equilibrium data obtained from adsorption experiments are matched with pseudo first or second-order

*Eur. Chem. Bull. **2023**,12(3), 2685-2692*

2687

Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

models. The adaptation of equilibrium data with adsorption isotherm models will also determine the distribution of chloride ions between the adsorbent and the liquid phase. The calcination reaction changed the MgAl-CO$_3$ LDH structure and a mix of Mg-Al oxide (Mg$_2$ Al$_2$ O$_9$, CLDH) formed. The corresponding chemical reaction is expressed by

Mg$_6$Al$_2$(OH)$_{16}$CO$_3$ $\rightarrow$ Mg$_6$Al$_2$O$_9$+CO$_2$+8H$_2$0

Mg$_6$Al$_2$O$_9$+ 2Cl$^-$+9H$_2$O $\rightarrow$ Mg$_6$Al$_2$(OH)$_{16}$Cl$_2$+2OH$^-$

Leo-CLDH equilibrium uptake capacity was determined by adding 5 g/L of adsorbent to 100 mL of initial chloride concentration (5-500 mg/L) solution in 250 mL Erlenmeyer flasks. The mixture was then shaken at 120 rpm and the desired temperature in a mechanical shaker.

3.1 Adsorption isotherm

Langmuir and Freundlich models were applied to the data using the nonlinear least squares method. The Langmuir model assumes monolayer adsorption on homogenous surfaces, whereas the Freundlich model assumes multilayer adsorption on heterogenous adsorption sites.[25]

The equilibrium isotherms of the Leo-CLDH were studied at 25, 35, and 45 °C. The data obtained from the experiments were matched with the Langmuir and Freundlich isotherm model to determine which model best describes the adsorption isotherm.[36]
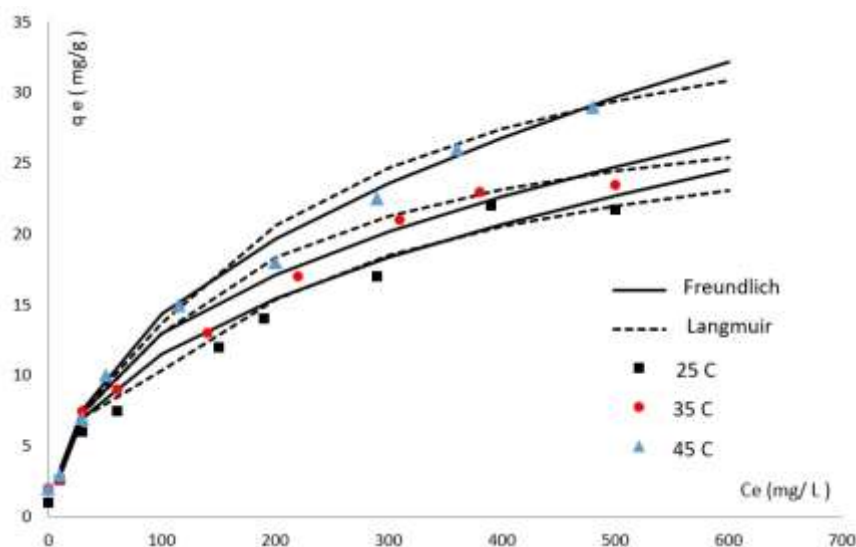
Langmuir isotherm model $\quad q_e = \frac{q_m K_L c_e}{1 + K_L c_e}$

Frenudlich isotherm model $\quad q_e = K_f c_e^{\frac{1}{n}}$

Where K$_L$ and K$_f$ are the constants of Langmuir and Freundlich models, respectively. q$_m$ is the maximum chloride uptake capacity and n represents the heterogeneity factor. Figure 1 shows isotherm data and fitted models of chloride adsorption and corresponding parameters are listed in Table.1. Considering the correction coefficient at different temperatures according to Table.1, the Frenudlich equation shows more agreement with the experimental data than the Langmuir equation. In addition, with increasing temperature from 25 ° C to 45 ° C, the adsorption capacity increases.

Table 1. Isothermal parameters of chloride adsorption onto Leo-CLDH.

Temperature	Langmuir			Frenudlich		
	K$_L$	q$_m$	R^2	K$_F$	n	R^2
°C	L/mg	mg/g		[(mg/g)(L/mg)]$^{1/n}$		
25	0.006	30.87	0.9612	1.70	2.4	0.9944
35	0.008	31.62	0.9624	2.09	2.6	0.9953
45	0.006	41.2	0.9700	1.91	2.3	0.9942

Eur. Chem. Bull. 2023,12(3), 2685-2692

2688

Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

FIGURE 1 Isotherm data and fitted models of Cl adsorption (startup Cl concentration 5-500 mg/L, LO-CLDH dosage: 5 g/L).
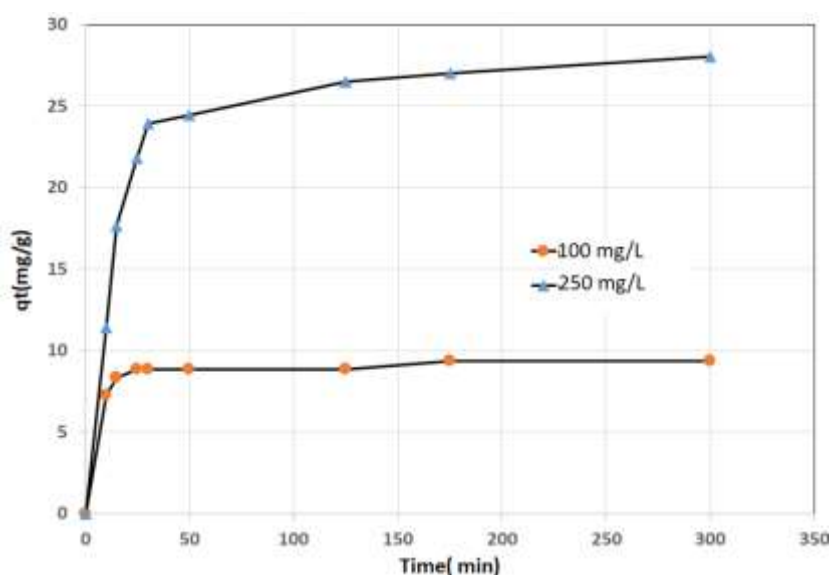
3.2 Adsorption Kinetics

The batch experiments to determine chloride adsorption kinetics were investigated by different initial chloride concentrations (100 mg/L or 250 mg/L). As can be seen in Figure 2, for both cases, the same equilibrium behavior is observed after one hour after the start of the experiment. To interpret the mechanisms involved, two different kinetic models were used and their corresponding equations are as follows:

Pseudo first-order model $\quad q_t = q_e(1 - e^{kp_1})$

Pseudo second order model $\quad q_t = \frac{k_{p2}q_e^2 t}{1 + k_{p2}q_e t}$

Where K_{p1} and K_{p2} represent the constant rate for the pseudo-first order model (min^{-1}) and pseudo-second-order model (g/(mg min)), respectively. q_t and q_e are the uptake amount of chloride per unit adsorbent weight at a certain time t and equilibrium (mg/g), respectively.

The kinetic parameters are summarized in Table.2. The obtained correlation coefficients (R^2) of the pseudo-second order model (0.9997 and 0.9994) are higher than the pseudo-first order model (0.9927 and 0.9788). Thus the pseudo second order equation seems to control the reaction kinetics. On the other hand, a higher constant value of the reaction rate at a lower concentration of chloride indicates that the adsorbent has a higher adsorption rate at a lower concentration.

FIGURE 2 Adsorption kinetics of chloride adsorption onto Leo-CLDH (Initial Cl concentration=100/250 mg/L, adsorbent dosage 10 g/L).

Table 2. Kinetic parameters of chloride adsorption onto Leo-CLDH.

C_t (mg/L)	Pseudo first order			Pseudo Second order		
	K_{p1}(min^{-1})	q_e (mg/g)	R^2	K_{p2}(g/mg.min)	q_e (mg/g)	R^2
100	0.2768	8.84	0.9927	0.3318	8.97	0.9997
250	0.0964	23.92	0.9788	0.0269	24.32	0.9994

3.3 Effect of adsorbents dosage

The chloride removal efficiency at different adsorbent dosages (1–15 g/L) is shown in Figure 3. The chloride uptake capacity

decreased from 13.2 mg Cl/g (at the dosage of 1 g/L) to 4.1 mg Cl/g (at the dosage of 15 g/L), while the adsorption efficiency increased from 24.8% to 92.4%. As can be seen in Figure 3, the

Eur. Chem. Bull. **2023**,*12(3), 2685-2692*

2689

Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

absorption efficiency increases with the increase of the adsorbent dose from 1 g/L to 7 g/L. Then its upward trend slows down a bit and finally reaches its maximum value at a dose of

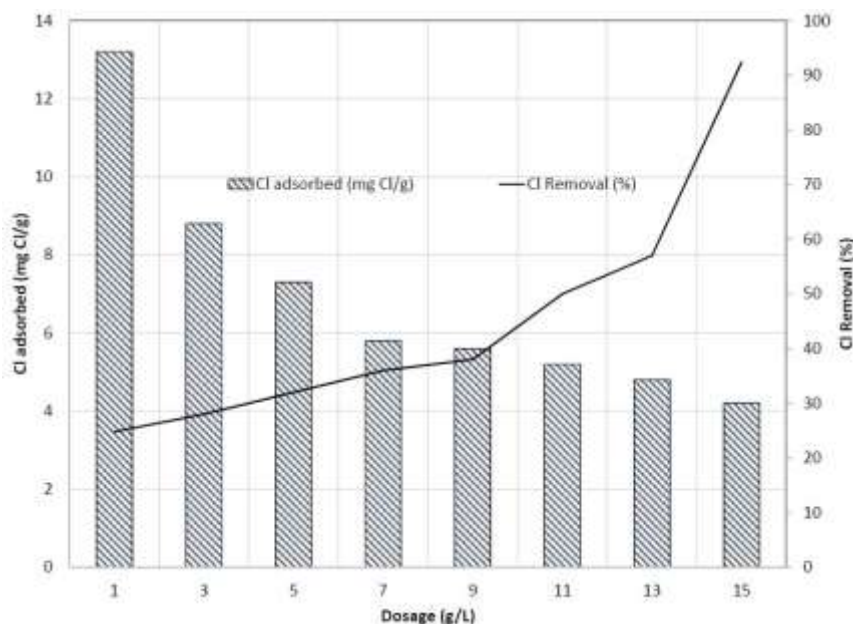15g/L. This phenomenon is related to the higher surface area provided by the more dosage of adsorbent.[37]

FIGURE 3 The effect of adsorbent dosage on Cl adsorption (Initial Cl concentration=50 mg/L).

3.4 Effect of co-pyrolysis temperature

The effect of co-pyrolysis temperature (300–700 C) on the chloride uptake capacity of Leo-CLDH was investigated, and the results are shown in Figure 4. It increases with increasing co-pyrolysis temperature and reaches the highest chloride removal of 97.6% at 500 C. However, chloride removal is then reduced as the co-pyrolysis temperature is increased,

reaching a minimum of 73.4% at 700 C. These outcomes are in line with earlier research on CLDH's phosphate absorption. The porosity and contact surface of the adsorbent were originally increased by raising the pyrolysis temperature. As the temperature rises further as a result of the breakdown of the crystal structure, the porosity decreases and the absorption index falls.[28]
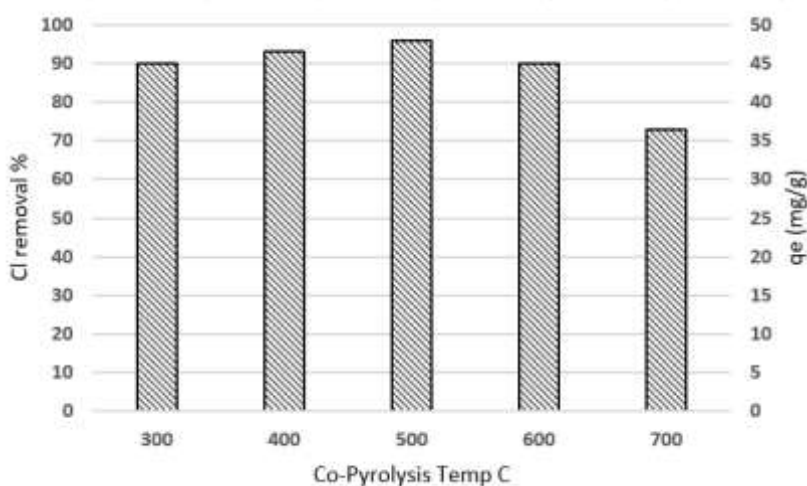

FIGURE 4 The effect of co-pyrolysis temperature on Cl adsorption (Initial Cl concentration=50 mg/L).

3.5 pH stabilization

Amine dichlorination causes an increase in the

pH of the solution from an initial value of 9.5 to 12.4 due to the release of sodium ions. To stabilize the pH of the solution in the initial range,

*Eur. Chem. Bull. **2023**,12(3), 2685-2692*

2690

Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

a varring amount of zein protein coated on the sand (ZCS) is added to 50 mL of dechlorinated amine agitated with a mechanical shaker at 200 rpm for 24 hours. The equilibrium pH versus the mass of ZCS is shown in Figure 5.
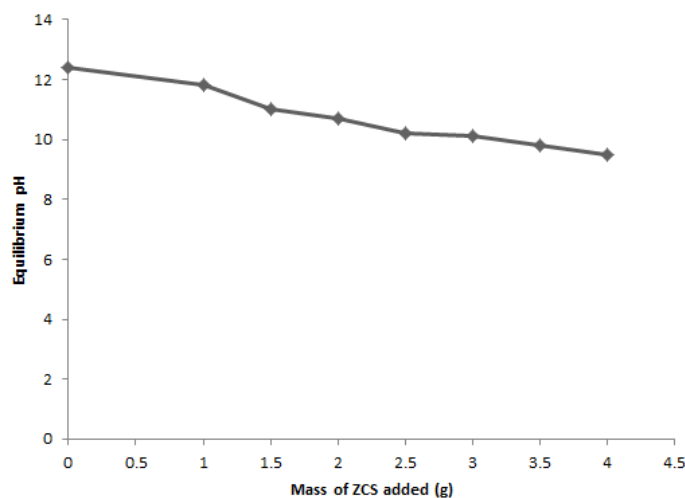

FIGURE 5 The equilibrium pH as a function of the mass of ZCS.

The spectra of zein-coated sand are shown in Figure 6. Stretching vibration at 2925 cm^{-1} and 3500 cm^{-1} is due to (N-H) and (O-H) bonds of amino acids (amide A band) belonging to the zein structure.[38] The stretching of carbonyl (C=O) at 1470 cm^{-1} is related to the amide groups of peptide.[39]
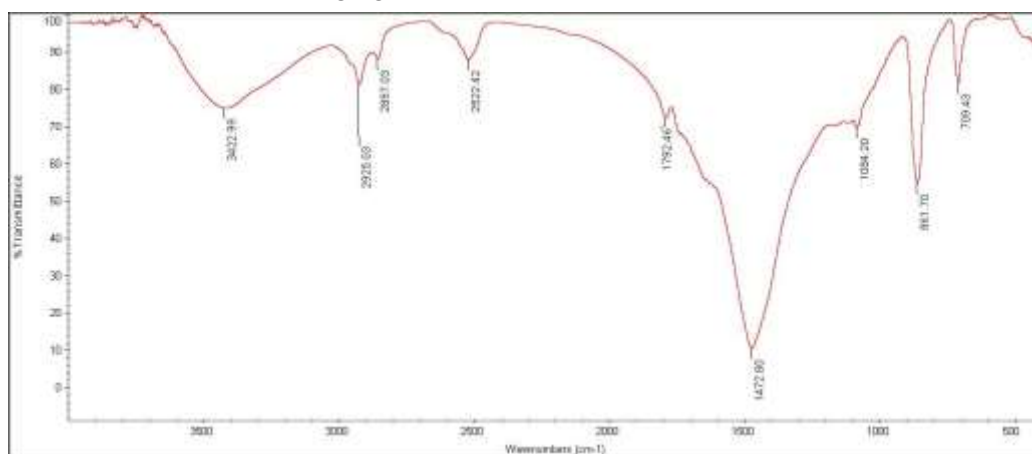

FIGURE 6 The FTIR spectra of zein-coated sand (ZCS).

4. CONCLUSION

This study presents chloride removal from MDEA solution by Leonardite char activated with Mg-Al calcined double hydroxide via co-pyrolysis s as a low-cost and efficient adsorbent. Due to active negatively charged groups in the Leonardite structure, the binding affinity of the carboxylic group to anionic species especially Cl$^-$ is limited. The addition of CLDH to the adsorbent greatly reduces this limitation. The adsorption Kinetic and isotherm were fitted to pseudo-second order and Freundlich models respectively. The effects of adsorbate dosage and co-pyrolysis temperature on MDEA dechlorization were tested. Batch equilibrium experiments at ambient conditions revealed that the new absorbent uptake capacity might be up to 41.2 mg/g of Leo/MgAl-CLDH (co-pyrolysis temperature: 500° C). To stabilize the concentration of the released sodium ion and adjust the pH of the solution within the range recommended, sand covered by zein protein has been used as a biodegradable and low-cost absorbent.

Eur. Chem. Bull. 2023,12(3), 2685-2692

2691

Chloride removal from MDEA via co-pyrolysis of Leonardite and Mg-Al calcined layered double hydroxide along with sodium stabilization, using eco-friendly adsorbent

Section A -Research paper

preparing the required materials.

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

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Eur. Chem. Bull. **2023**,*12(3), 2685-2692*

2692