



## Batch Absorption, Regeneration Studies of The Natural Zeolite and Evaluate Their Performance of Removal Efficiency Over Multiple Cycles

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**ABSTRACT:** Characteristic zeolites are broadly utilized in different natural remediation due to their physio-chemistry properties such as tall ion-exchange capacity, adsorption productivity, tall ion-exchange capacity, cost-effectiveness and plenitude in nature. Zeolite are overwhelmingly successful in expelling overwhelming metals from wastewater. This paper centering on the bunch motor and recovery thinks about to assess the execution of the zeolite evacuation productivity amid consequent recovery cycles to guarantee their maintainable utilize. Recovery tests were conducted chemical treatment utilizing 200 ml of 0.5M NaCl. All through the recovery tests, it was watched as a common drift that the adsorption capacity of common zeolite that has been recovered was diminished somewhat with each rehashed three recovery cycles. Zeolite gems are hold their unique morphology with slight alter. The result from the ICP-OES appears that the effectiveness of overwhelming metal evacuation was upgraded with the application of pre-treatments. The comes about have too appeared that driving drive for adsorption is exceptionally tall within the introductory organize of the adsorption prepare and a slower adsorption rate takes after. Characteristic zeolites had a tall evacuation capacity of overwhelming metals and other properties did not alter in a critical way, indeed after a number of recovery cycles.

**KEYWORDS:** Natural zeolite; Adsorption; Wastewater treatment; Regeneration; Ion exchange.

### 1. INTRODUCTION

Zeolite may be a microporous aluminosilicate mineral, has been broadly examined as an particle trade fabric due to its tall cation trade capacity and effective regenerative properties (Salih et al, 2025; Alshameri et al, 2014; Wijesinghe et al, 2016). Zeolites are taken a toll successful in operational, copious in supply, ease of utilize, naturally neighborly, simple to recover and recuperate their adsorption properties. This make zeolite more adaptable to utilize in several applications such as catalyst, adsorption and particle trade procedure industry, pharmaceutical, farming, gas adsorption and contamination control (Cadard, et al, 2022; Yuna, et al, 2016; Eroglu et al, 2017; Cataldo et al, 2006; Jarosz et al, 2022 ;Margeta and Farka, 2020; Salih et al, 2021). Overwhelming metal harmfulness can happen through different sources, such as sullied water, discuss contamination, nourishment, mechanical work situations, or indeed certain solutions (Salih et al, 2018). Overwhelming metals are well-known to cause different wellbeing maladies counting influencing numerous organs and frameworks within the body. A few of the common wellbeing issues caused by presentation to overwhelming metals incorporate neurological clutters, kidney harm, cardiovascular issues, cancer, respiratory issue, bone harm, skin issues and influence the safe framework (Ali et al, 2019; Tchounwou et al, 2012; Senila et al, 2019a ; Rahmany-samani et al, 2023; Hlodal et al, 2015). Particle trade and adsorption are common operations utilized for evacuation of overwhelming metals from wastewater. Over the final decades, the utilize of particle trade and adsorption forms in treating wastewater, with the potential utilize of normal zeolite have broadly seriously considers by analysts (Oztas et al, 2008; Blanchard *et al*, 1984; Argun, 2008; Inglezakis et al, 2007; Polat et al, 2004; Gunay et al, 2007). As a portion of economical advancement and squander administration programs, it ought to endeavor to diminish squander materials and change over them into valuable items by reusing

or reusing strategies (Mohammed, et al, 2020; Crini, 2006; Fatah et al, 2025). This could be valuable for decreasing the sum of crude materials utilized and working costs, decreasing the volume of squander materials and diminishing their natural affect. In later decades, numerous ponders have been conducted on altering characteristic zeolites to extend their particular surface range, reestablishing the zeolite or move forward adsorption capacity utilizing different strategies utilizing warm or chemical pre-treatments, alteration with surfactant and alteration by metal oxides ( Salih, et al, 2020; Wijesinghe et al, 2016; Mohammed, et al, 2020; Fatah et al, 2025). The recovery of adsorbent is one of the variables that can be taken into consideration since that produces the forms more prudent and diminishes the natural affect (Salih, et al, 2019b). For these reasons, there's a solid motivation to get it the instruments driving to the reuse and recovery of adsorbents, which makes the strategy cheaper and cleaner. Recovery is the method of re-establishing the zeolite adsorption capacity after it has immersed. Chemical treatment in recovery handle already considered, which ordinarily includes the utilize of NaCl and water to remove sorbed cations from the zeolite surface (Stone and Holliday, 1944; Salih, et al, 2025). The recovery cycles for characteristic zeolite can shift depending on components just like the sort of overwhelming metal, the Physico-chemical properties of zeolite, and the strategy utilized for regeneration (Guo et al, 2007; Katsou et al, 2011; Zhang et al, 2017; Salih, et al, 2020). Hence, the execution of common zeolites assessment amid ensuing recovery cycles considered. This work is additionally to examine the impact of the number of recovery cycles on the evacuation of overwhelming metals from wastewater arrangements utilizing normal zeolite as a moo fetched fabric. This paper has focused on the efficiency of natural clinoptilolite in the removal of heavy metals cations copper ( $\text{Cu}^{2+}$ ), iron ( $\text{Fe}^{3+}$ ), lead ( $\text{Pb}^{2+}$ ) and zinc ( $\text{Zn}^{2+}$ ) from synthetic industrial wastewater, to achieve allowable limits. The adsorption behaviour of natural clinoptilolite and a number of regeneration cycle for removal efficiency of natural zeolite were also investigated.

## **2. MATERIALS AND METHODS**

### **2.1. Batch kinetic studies**

Group dynamic thinks about were performed in arrange to explore the conduct of adsorbents and get it the evacuation instruments included within the adsorption handle. Active ponders are basic forms that are utilized to obtain data approximately the method elements such as the adsorption rate, contact time and mass exchange parameters counting outside mass exchange coefficients and intraparticle diffusivity (Connors, 1990; Salih, et al, 2018). Therefore, kinetic considers were utilized in this think about to assess the reasonableness of common zeolite for expelling overwhelming metals from arrangement.

#### **Batch Adsorption Procedure**

Bunch tests were carried out utilizing 4g measurements of normal zeolite. The adsorbent was blended with 100 ml arrangement of the suitable multi-component arrangement. An tumult speed of 150 rpm was utilized for 360 minutes and tests were collected each hour and examined. The molecule measure of the zeolite tests utilized was  $250 \text{ \AA}$ . The pH was balanced to  $4 \pm 0.1$ . This explore was at first carried out for  $\text{Cu}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Pb}^{2+}$  and  $\text{Zn}^{2+}$  particles utilizing settled starting metal concentrations in open discuss at room temperature. Concentration of the overwhelming metals some time recently and after the adsorption prepare measured, utilizing strategies like inductively coupled plasma mass spectrometry (ICP-MS). The information gotten from the active adsorption tests were utilized to decide the rate expulsion of metal particles from arrangement was moreover decided utilizing the condition underneath:

$$\text{Percentage Adsorbed (\% Removal Efficiency)} q_e = \{(C_o - C_e) \times 100\} / C_o$$

where,

$q_e$  amount of adsorbate adsorbed per unit weight of adsorbent (mg/g)

$C_o$  and  $C_e$  are the initial and final metal ion concentrations in solution (mg/l) respectively,  $V$  is the solution volume (l) and  $m$  is the weight of the zeolite used (g).

### **2.2. Regeneration in batch studies**

Regenerative capability is one of the characteristics that are considered when utilizing zeolite as an adsorbent for any down to earth application (Richardson et al, 2002). Zeolite can be recovered and reused with small alter in its adsorption proficiency after recovery. Characteristic zeolite can be reused for a number of cycles some time recently the structures are depleted, which makes the forms temperate and empowers a diminish within the volume of squander fabric. In this consider, the recovery of common zeolite was carried out utilizing Chemical treatment. The adsorption capacity of the common zeolite was moreover explored after each run. Utilized zeolite tests were recovered and blended with multi-component arrangements of the overwhelming metal particles for cycles of 360 minutes each. The tests were washed with deionised water after each recovery cycle in arrange to expel overabundance NaCl entangled inside the zeolite structure. Three adsorption cycles were carried out for all of the overwhelming metal particles.

#### **Regeneration Procedure**

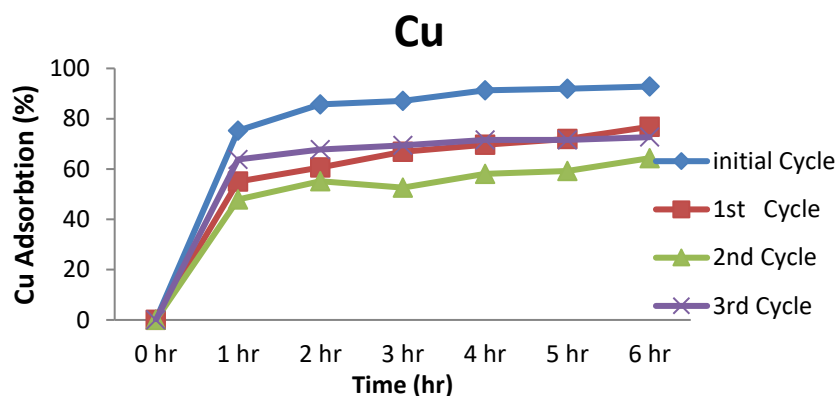
Recovery of the characteristic zeolite utilizing Chemical treatment was performed utilizing 200 ml of 0.5M NaCl. After each run, the zeolite test was disturbed within the stripping arrangement employing a attractive stirrer at 150 rpm for 45 minutes. The tests were flushing twice in deionised water for 15 minutes and dried in open discuss at room temperature. They were at that point re-

used for the following test run and the recovery was rehashed 4 times in arrange to watch the adsorption efficiencies with time. The adsorption/ desorption cycle was performed to decide the reversibility of the responses and reusability of characteristic zeolite (Oztas et al, 2008; Han et al, 2006; Salih, et al, 2025; Yaqub, 2024b). Rehash the absorption-regeneration prepare for numerous cycles (4 cycles) to assess the zeolite execution over time and degree the expulsion proficiency in each cycle and survey any lessening in execution.

### 3. RESULTS AND DISCUSSION

#### 3.1. Kinetic study results

The comes about of the motor tests were utilized to degree the adsorption capacities from arrangements. Multi-component arrangements were blended with common zeolite and unsettled for 360 minutes at room temperature. The beginning concentrations of the blended component arrangements were 141, 41, 336 and 88 mg/l for the copper ( $\text{Cu}^{2+}$ ), press ( $\text{Fe}^{3+}$ ), lead ( $\text{Pb}^{2+}$ ) and zinc ( $\text{Zn}^{2+}$ ) particles separately. In common the comes about appear that the most elevated adsorption rate of  $\text{Cu}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Pb}^{2+}$  and  $\text{Zn}^{2+}$  particles took put within the to begin with hours taken after by a slower adsorption rate afterward on. The primary hour is an starting arrange of adsorption when higher rates of adsorption take put; this may be due to the accessibility of more adsorption destinations and the reality that the metal particles trade effortlessly on the surface of the zeolite grains (Inglezakis et al, 2007; Yaqub, 2024a; Salih, et al, 2025). The driving constrain for adsorption is exceptionally tall within the initial organize of the adsorption handle and this moreover comes about in the next starting adsorption rate. After that, a slower adsorption rate takes after due to slower dissemination of the metal particles into the insides channels. Subsequently, these metal particles involve the replaceable positions inside the gem structure of the normal zeolite (Amarasinghe and Williams, 2004; Yaqub, 2019; Myroslav et al, 2006; Abdulrahman et al, 2025; Fatah et al, 2025). (Fig.1) appears that when the common zeolite was initially used at concentration 141.41 mg/l, the stacking capacity for  $\text{Cu}^{2+}$  particles was 3.26 mg/g, and after the primary recovery with 0.5M NaCl stripping arrangement, the loading capacity diminished to 2.70mg/g. The moment stacking capacities for  $\text{Cu}^{2+}$  particles after recovery were around 2.26 mg/g (lower than the primary recovery run and the beginning run) and the third run had a stacking capacity of 2.55 mg/g, which was marginally lower than the beginning run but higher than the moment recovery run (Fig.1). With an beginning stacking capacity of  $\text{Fe}^{3+}$  particles of 1.02 mg/g (Fig.2) it was watched that the primary and moment recovery cycles recorded stacking capacities were lower than the beginning run. The ensuing third recovery cycles recorded stacking capacities somewhat higher than the introductory run. Agreeing to (Fig.3) it is clear that the introductory stacking capacity of  $\text{Pb}^{2+}$  particles was 8.38 mg/g. The ensuing to begin with, moment and third recovery cycles recorded stacking capacities comparative to the introductory run. The starting stacking capacity of  $\text{Zn}^{2+}$  particles was 1.48 mg/g (Fig.4). It was watched that the primary and moment recovery stacking capacity diminished to 1.37 and 1.32 mg/g. The third run had a stacking capacity of 1.64 mg/L, which was marginally higher than the beginning run. Desorption forms took put due to the relocation of the overwhelming metal ions from adsorption locales on the zeolite structure within the case of  $\text{Na}^+$  particles from NaCl arrangement. All through the recovery tests, it was watched as a common drift that the adsorption capacity of normal zeolite that has been recovered was diminished somewhat with each rehashed three recovery cycles. This concurs with the comes about gotten by Argun (2008) with respect to the recovery of characteristic zeolites, as he found that the zeolite capacity diminishes with each rehashed recovery cycle (Mohammed, et al, 2020; Yaqub, 2025; Salih, et al, 2018). This diminish in zeolite adsorption capacity may show up for two reasons: to begin with, it may be that a few metal particles, once they are traded onto the zeolite, are inflexibly settled or gotten to be blocked off to the approaching particles, and hence the particle trade locales decrease with time. The moment conceivable component may be due to the expulsion of oxygen iotas from the cross section, when strongly held cationic species are expelled amid the recovery step. This would result within the pulverization of the zeolite grid and misfortune of adsorption/ particle trade destinations.



**Fig. 1: The percentage of removal efficiency of natural zeolite for the removal copper ions before and after subsequent regeneration.**

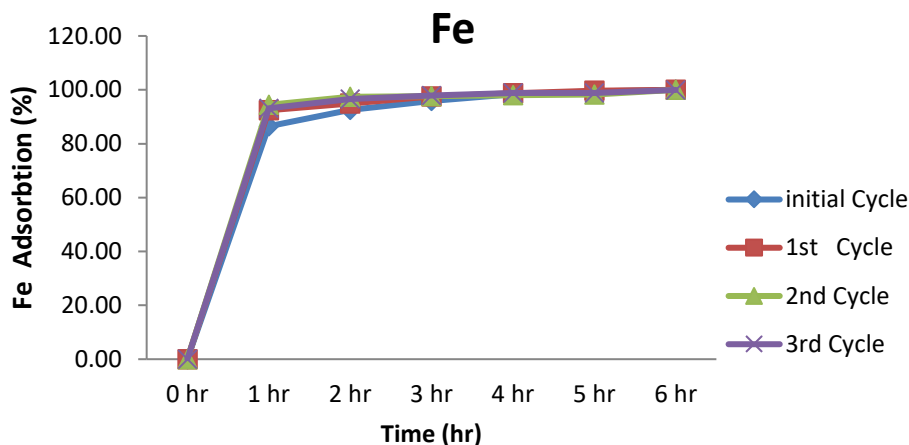


Fig. 2. The percentage of removal efficiency of natural zeolite for the removal Iron ions before and after subsequent regeneration.

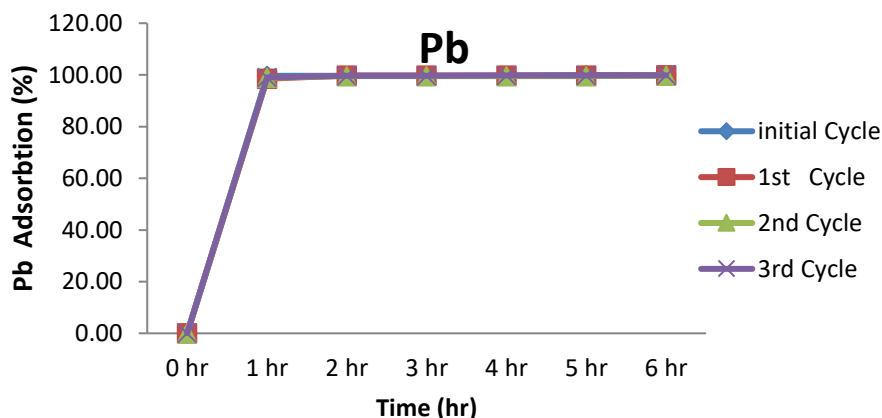


Fig. 3. The percentage of removal efficiency of natural zeolite for the removal lead ions before and after subsequent regeneration.

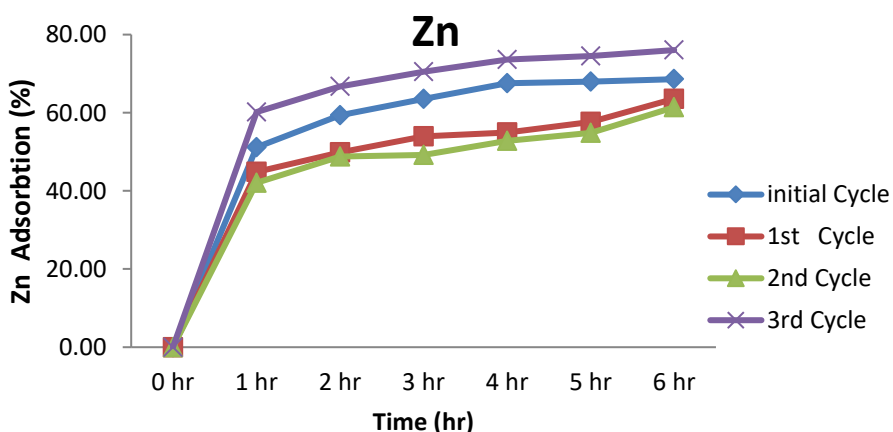


Fig. 4. The percentage of removal efficiency of natural zeolite for the removal zinc ions before

### 3.2. Zeolite characterization result

The surface morphology and microstructure of diverse common zeolite tests some time recently and after different medicines were examined utilizing Checking Electron Microscopy (SEM). Zeolites was recovered to re-establish their adsorption or catalytic properties, ordinarily through chemical medicines. (Fig.5) appear the micrographs of the ‘as received’ normal zeolite tests. The pictures were taken beneath the taking after SEM explanatory conditions: EHT =10.00 kV and Flag A = SE1, WD 6.5mm at amplification of 1000x, 5000x. The micrographs clearly appear that the characteristic zeolite structure have a well-defined microporous structure.

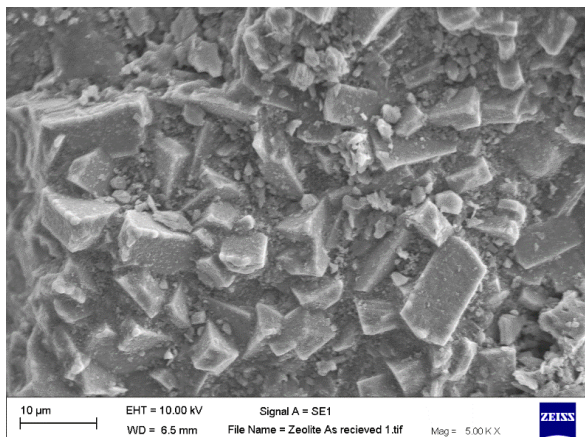


Fig. 5. Show the micrographs of the “as received” natural zeolite samples.

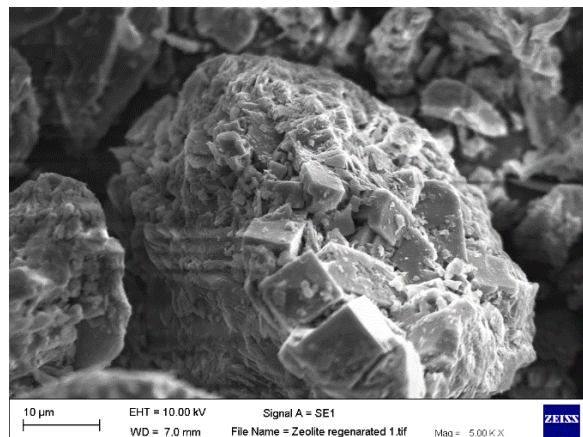


Fig.6. SEM micrographs of first regeneration of natural zeolite at 5000x magnifications.

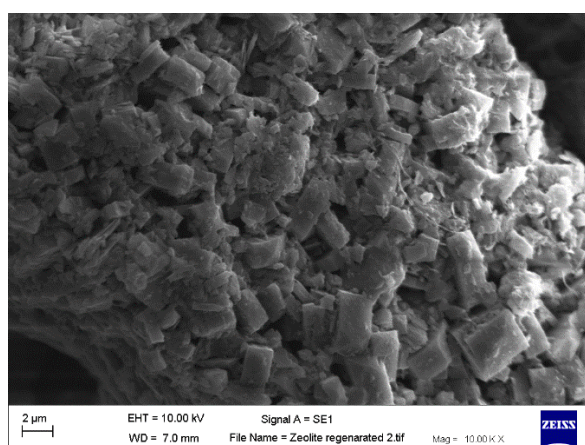


Fig.7. SEM micrographs of second regeneration of natural zeolite at 10000x magnifications.

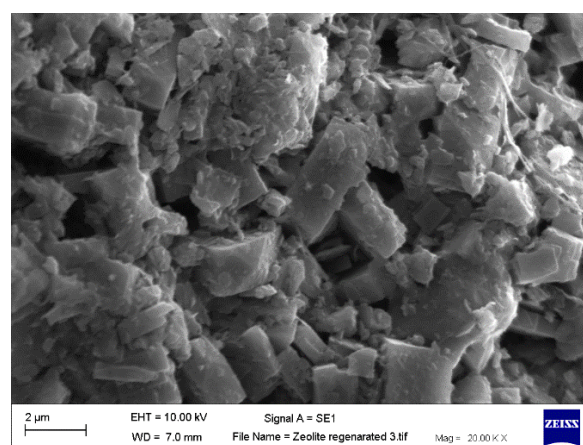


Fig. 8. SEM micrographs of third regeneration of natural zeolite at 20 000x magnifications

(Fig. 6, 7 and 8) shows the micrographs of the “regenerated” natural zeolite samples of three regeneration cycles. The images were taken under the following SEM analytical conditions: EHT=10.00 kV and Signal A = SE1, WD 7 mm at a magnification of 10000x, 20000x and 5000x. The SEM clearly show that the natural zeolite pores collapse or blockage and Regeneration processes caused zeolite particles to agglomerate. Acid treatments have altered the saple structure and leading to dissolution of certain components, which can be observed as changes in surface texture.

## CONCLUSION

Throughout the regeneration experiments, it was observed zeolite can be regenerated and reused while the adsorption capacity of natural zeolite that was decreased slightly with each repeated three regeneration cycles. Natural zeolites performance declines over multiple regeneration cycles.

Comparing SEM images of as resived natural zeolite with those of regenerated zeolite can provide a clear understanding of the impact of regeneration cycles on the material’s structure and morphology have changed such as structural degradation, pore blockage, or loss of adsorption

After analysing the removal efficiency, regeneration capacity, and adsorption characteristics of natural zeolite. It can be conclude that natural zeolite is a sustainable and effective solution for wastewater treatment over extended periods of use.

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