

CHARACTERIZATION OF NATURAL ZEOLITE AND EVALUATION OF ITS ADSORPTION CAPACITY

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Abstract

The demand for zeolites has been steadily increasing due to zeolite has various range of applications that contribute significant economic value including catalysis, wastewater treatment, industries, agriculture, gas separation, and other environmental remediation. The structure and properties of the zeolite samples were characterized by using different analytical techniques which discloses information on the physiochemical, phase identification, elemental composition, surface morphology, texture, and microstructure of materials surface morphologies, thermal stability of zeolite product. All are related to **removal efficiency of heavy metals** ions from solution. The result confirm that the natural zeolite was identified as clinoptilolite, with minor impurities such as quartz and clay minerals. Zeolite crystals showed an irregular, plate-like morphology with a rough and porous surface. Visible pores and channels indicated a high surface area, suitable for adsorption. Occurrence of exchangeable cations (Na^+ , K^+ , Ca^{2+} , and Mg^{2+}) enables the zeolite to effectively adsorb and exchange cations such as Fe^{3+} , Cu^{2+} , Pb^{2+} and Zn^{2+} from wastewater.

Keywords: Adsorption, Natural Zeolite, Wastewater, Heavy Metals, Ion Exchange.

DOI: 10.31039/bjir.v2i4.26

1. INTRODUCTION

Natural zeolites are highly multipurpose materials used in different applications such as catalysis, gas separation, mining, wastewater treatment and in some other various fields of industry and agricultural remediation (Kesraoui Ouki *et al.*, 1994; Papageorgiou *et al.*, 2006; Bekkum H. *et al.* 1991; Chen and Poon, 2009; Salih *et al.*, 2018).

The adsorption and particle trade handle are the foremost broadly utilized methods that have pulled in expanding intrigued in logical community and diverse companies, which contribute critical financial (Yaqub, 2019; Salih *et al.*, 2020; Salih *et al.*, 2020). Moreover, manufactured zeolites were created due to their amazing adsorption capacities and higher exhibitions, compared to common zeolites, but they are as a rule exceptionally costly. Different zeolites from kaolinite or other fiery debris and have made incredible advance in union of 4A, mordenite, X, Y zeolites, etc. (Inglezakis and Grigoropoulou, 2004; Salih *et al.*, 2019; Pandey *et al.*, 2009). In the mean time, numerous adjusted zeolites have been created that can be appraised as having a middle adsorption capacity, whereas they don't fetched as much as manufactured zeolite and have distant better; a much better; a higher; a stronger; an improved" > a higher adsorption capacity than characteristic zeolite (Colella, 2007; Colella and Shrewd, 2014; Yaqub, 2019; Salih *et al.*, 2020). Numerous considers have appeared that zeolites have tall adsorption capacities for evacuation of poisons from arrangements and can reach adsorption

balance level in brief periods of time (Salih *et al.*, 2020; Blanchard *et al.*, 1984; Erdem *et al.*, 2004; Inglezakis *et al.*, 2004). The numerous employments of common zeolite, counting the expulsion of overwhelming metal particles in wastewater treatment are based on its physical and chemical properties. These properties clarify their wide run of agrarian and mechanical applications (Salih *et al.*, 2021; Rivera *et al.*, 2000). As detailed by Colella (1999) the application of normal zeolites for wastewater treatment is primarily based on their particle trade and adsorption properties. It is additionally well established that particle trade within the case of zeolites takes put among cations and as it were their adjustment can give them with anion sorption properties (Salih *et al.*, 2018; Colella, and Shrewd, 2014). The take-up of metal cations from arrangements by the zeolites is influenced by a various of components such as the temperature, the starting arrangement pH, the nearness of competing particles, the molecule measure, the measurements of the hydrated broken down species compared to the opening of their channels and the outside surface movement (Colella, 2007; Salih *et al.*, 2019). Zeolites are steady strong and can stand up to tall temperatures since they have tall softening focuses over 1000°C. They moreover stand up to tall weights and don't oxidize in discuss and break down in water or other inorganic solvents (Dyer, 1988; Dyer and Zubair, 1998; Yaqub, 2025; Gottardi and Galli, 1985; Guinier, 1963). There are numerous sorts of common zeolite that have been distinguished within the world; there are exceptionally common shapes such as common zeolite, mordenite, stilbite, analcime phillipsite, chabazite, and laumontite, though paulingite, offretite, mazzite and barrerite are much rarer shapes (Blanchard *et al.*, 1984). Galli (1983) contends that natural zeolite is the foremost plenteous of the characteristic zeolites. Its characteristic unpleasant morphology appears an open reticular structure of simple get to, shaped by open channels of 8- to 10-membered rings. Argun (2008) has moreover recommended that characteristic zeolite has good adsorption capacities and is broadly utilized within the world. In any case, distinctive sorts of zeolite have diverse adsorption capacities based on their physicochemical properties (Salih *et al.*, 2018; Inglezakis, 2007; Yaqub, 2024; Mohammed *et al.*, 2020). This paper depicts the characterisation of the normal zeolite to supply data on: (a) structure and morphology; (b) chemical composition; (c) capability to sorb and hold particles; and (d) capability to chemically change over those particles.

1.1. Characteristics of adsorbents

The foremost critical properties for any adsorbent are capacity, selectivity, regenerability, energy, compatibility and cost. The good adsorbents have to be have the taking after imperative

characterises: (Ruthven, 1984; Richardson *et al.*, 2002; Gedik and Imamoglu, 2008; Inglezakis *et al.*, 2004; Misaelides, 2011; Salih *et al.*, 2018; Mohammed *et al.*, 2020):

1. High porosity, uniform molecular-sized channels and sweeping specific surface zone.
2. High adsorption capacity in a wide run of adsorbate concentrations.
3. High molecule exchange capacities.
4. High catalytic properties and accessibility through pores to allow certain particles section in the midst of adsorption and to break down others.
5. Simplicity and moor gotten in operation and practical methodology.
6. Easy to recuperate and reuse for various times.
7. High softening point, warm relentlessness and essential relentlessness toward drying out.
8. Low brought of securing and does not display additional defilement into the environment, irrelevant waste period.

2. MATERIALS AND CHARACTERIZATION

2.1 Characterisation of natural zeolite

Knowledge of the characteristics of natural zeolite, raw materials used in synthesis are very important. The technological properties of zeolite products depend on the physical, chemical and mineralogical characteristics of the starting materials; this also controls the overall processing treatment of polluted effluents.

The comprehensive characterization of natural zeolite using various analytical techniques such as X – Ray Diffraction (XRD), X – Ray Fluorescence (XRF), Thermogravimetric Analysis (TGA), Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS) and Inductively Coupled Plasma-Optical Emission Spectrometers (ICP-OES) provides critical insights into its structural, chemical, and physical properties. These properties directly influence the zeolite's adsorption capacity, making it a versatile material for applications in heavy metals removal from wastewater.

2.2. Analytical techniques

2.2.1. Scanning Electron Microscopy (SEM)

SEM may be a capable imaging method utilized to think about the surface morphology, surface, and microstructure of materials at tall determination. For normal zeolites, SEM gives profitable bits of knowledge into their gem shape, measure, porosity, and surface highlights. The surface morphology of the diverse normal zeolite tests was decided beneath the taking after expository conditions: EHT = 10.00 kV and 20.00 kV, Signal A = SE1 and VPSE, WD = 6.0, 6.5, 7.0 and 8.5mm at different magnifications.

2.2.2. Energy Dispersive Spectroscopy (EDS)

EDS is an explanatory procedure was utilized to distinguish the natural composition and chemical characterization of distinctive characteristic zeolite tests. EDS regularly coupled with SEM, may be a method utilized to analyze the essential composition of materials. For common zeolites, EDS gives profitable data approximately the chemical composition, counting the system components (Si, Al) and interchangeable cations (Na^+ , Mg^{2+} , K^+ and Ca^{2+}).

2.2.3. X – Ray Diffraction (XRD)

XRD expository strategy was utilized for stage recognizable proof of the normal zeolite tests and characterisation of the heterogeneous strong blends to decide the relative plenitude of crystalline compounds and get data on the unit cell measurements. XRD was utilized to recognize zeolite by comparing the diffraction design with known reference designs from databases just like the Universal Zeolite Affiliation (IZA) or the ICDD (Universal Middle for Diffraction Information).

2.2.4. X – Ray Fluorescence (XRF)

XRF was utilized as explanatory strategy for deciding the basic composition of zeolites. Zeolites are aluminosilicate minerals, and their chemical composition can contrast depending on the sort of zeolite. XRF can recognize and measure the major and follow components display in zeolites, such as silicon (Si), aluminum (Al), sodium (Na^+), potassium (K^+), calcium (Ca^{2+}), magnesium (Mg^{2+}) and other components that will be portion of the zeolite structure or display as pollutions.

2.2.5. Thermogravimetric Analysis (TGA)

TGA may be a procedure utilized to degree the weight changes in a fabric as a work of temperature or time. The warm stability of the common zeolite was obtained this is often to degree changes within the physiochemical properties while expanding temperature continually. TGA is especially valuable for analyzing water substance, warm soundness, and decay conduct, and pick up data approximately the mass misfortune amid the warm treatment handle.

2.2.6. Inductively Coupled Plasma-Optical Emission Spectrometers (ICP-OES) analysis

The overwhelming metal particle concentrations within the wastewater arrangement were decided utilizing ICP-OES. The standard arrangements for metal examination utilizing the ICP were arranged from standard metal arrangements to decide the sum of Fe^{3+} , Cu^{2+} , Pb^{2+} and Zn^{2+} show within the arrangements some time recently and after the particle trade.

3. RESULTS AND DISCUSSION

3.1. Scanning Electron Microscopy (SEM) results

The surface morphologies of the different natural zeolite samples before and after modification were analysed. Fig (1) show the micrographs of the “as received” natural zeolite samples. The images were taken under the following SEM analytical conditions: EHT =20.00 kV and Signal A = VPSE, WD 8.5mm at a magnification of 2000x. The micrographs clearly show that the natural zeolite structure contains a number of different diameter size porous surface, Irregular, plate-like crystals with a size range of ($1\text{ }\mu\text{m} \leq d \leq 3\text{ }\mu\text{m}$) due to their framework structure. SEM show the roughness, porosity surface features of zeolite crystals with visible channels and cavities. Some areas show smooth surfaces, possibly due to secondary mineral coatings.

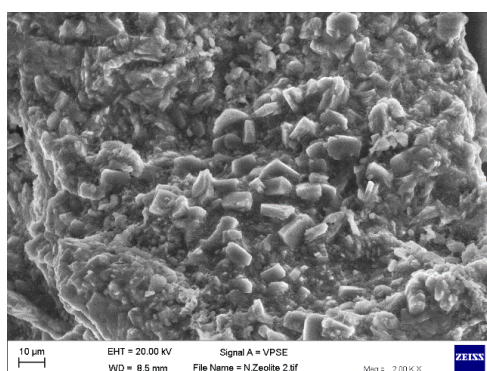


Fig. 1: Showing the SEM micrograph of as-received natural zeolite at a magnification of 2000x

(Fig. 2) appears the characteristic zeolite beneath the taking after SEM explanatory conditions: EHT = 20.00 kV, Flag A = VPSE, WD 8.5mm at a amplification of 2000x. The micrographs generally appear well characterized precious stones of common zeolite (SBU is 6-6). SEM pictures unveil the shape and estimate of zeolite gems, which is plate-like.

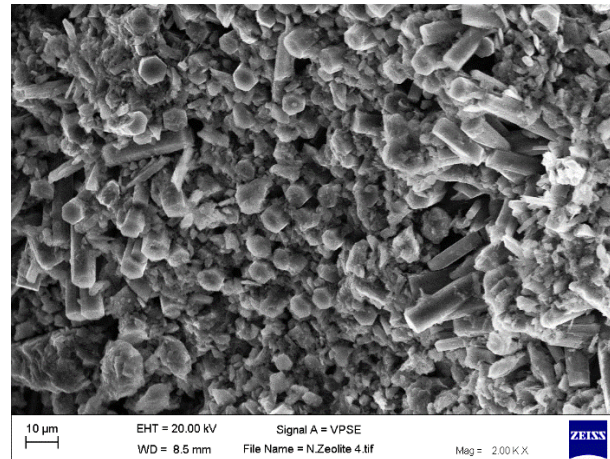


Fig. 2: Showing the randomly oriented crystallites and in particular indicating the crystalline nature of natural zeolite, at a magnification of 2000x.

The impact of chemical pre-treatment was dissected when 0.5M of NaCl acidic arrangement was utilized for the chemical pre-treatment of zeolite to evacuate all of the undesired unstable matter (undesirable squander fabric) and get a clean surface, as appeared in (Fig. 3) beneath the taking after SEM expository conditions: EHT =10.00 kV, Flag A = SE1, WD 6.5mm at a amplification of 20000x. The micrographs underneath appear a clean surface and well-defined gem structures of normal zeolite.

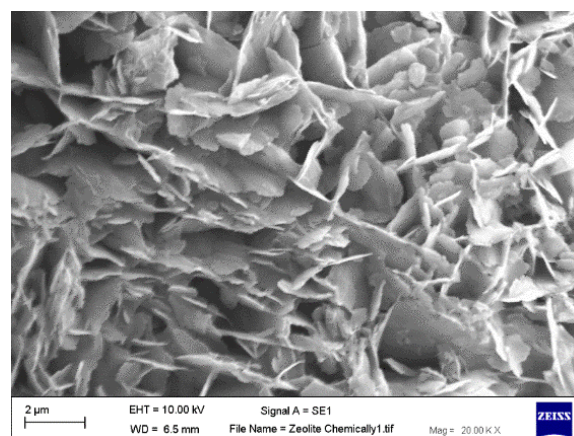


Fig. 3: SEM micrographs of chemical pre-treatment of natural zeolite at different magnifications: (a) 20000x

SEM analysis for acid-treated zeolites shows a cleaner surface with fewer impurities (e.g., clay minerals or amorphous phases). The surface appear smoother or more uniform compared to untreated zeolites due to chemical treatments as well as shows unclogged blocked pores. The pore size distribution appear more uniform after treatment.

3.2. Energy dispersive spectroscopy (EDS) results

An electron pillar was arbitrarily coordinated onto diverse parts of the normal zeolite tests in arrange to induce a more precise investigation. As appeared in Fig. (4) it is discernible how the diverse parts of the test were examined.

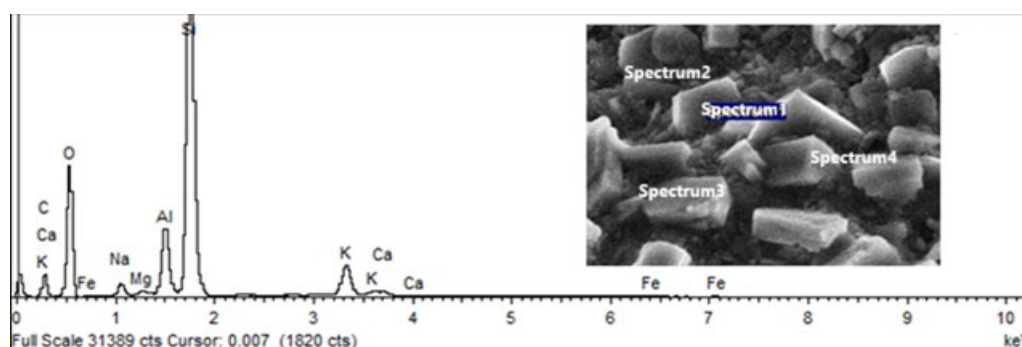


Fig. 4: EDS analysis showing the elemental composition and the scanned image for natural zeolite.

The results of the EDS analysis also shows that the predominant exchangeable cations in the natural zeolite structure were found to be Na^+ , Mg^{2+} , K^+ and Ca^{2+} this is indicating a mixed-cation zeolite. Cations influence the ion-exchange capacity and catalytic properties of the zeolite.

The Si/Al ratio affects properties such as hydrophilicity, acidity, and thermal stability.

The **Si/Al ratio** is a critical parameter for zeolites, calculated as:

$$\text{Si/Al ratio} = \frac{\text{Weight\% of Si} / \text{Atomic weight of Si}}{\text{Weight\% of Al} / \text{Atomic weight of Al}}$$

$$Si/Al \text{ ratio} = \frac{21.22/28.09}{3.93/26.98} = 5.2$$

A high Si/Al ratio (>5) indicates a more hydrophobic and thermally stable zeolite and low ion-exchange capacity and acidity.

Table 1: EDS analysis showing the elemental composition and the scanning method for natural zeolite.

Element	Na	Mg	Al	Si	K	Ca	Ti	Fe
zeolite	%	%	%	%	%	%	%	%
Spectrum 1	1.18	0.27	3.92	22.14	3.13	0.36	0.0641	0.37
Spectrum 2	1.25	0.33	4.01	22.13	2.47	0.32	0.0651	0.44
Spectrum 3	1.31	0.38	3.78	20.05	1.9	0.31	0.0636	0.33
Spectrum 4	1.57	0.16	4.02	20.54	2.04	0.24	0.0626	0.23
average	1.33	0.29	3.93	21.22	2.39	0.31	0.06	0.34
Std.deviation	0.17	0.09	0.11	1.08	0.55	0.05	0.00	0.09

3.3. X – Ray Diffraction (XRD) results

The procedure was carried out and data were collected in the range 0-50 degrees, with a scanning step of $^{\circ}2\theta$. The crystalline patterns were compared with the standard line patterns from the powder diffraction file database supplied by the International Centre for Diffraction Data (ICDD). The intensity and sharpness of XRD peaks are indicators of the crystallinity of the zeolite. Crystalline zeolites show sharp, well-defined peaks, while amorphous materials produce broad peaks. The XRD result showed that the sample contained natural zeolite in the majority (Fig.5) Strong peaks around $^{\circ}2\theta = 5.2^{\circ}, 10.1^{\circ}, 11.2^{\circ}, 16.6^{\circ}, 19.3^{\circ}, 24.1^{\circ},$ and 30.4° (Cu K α radiation).

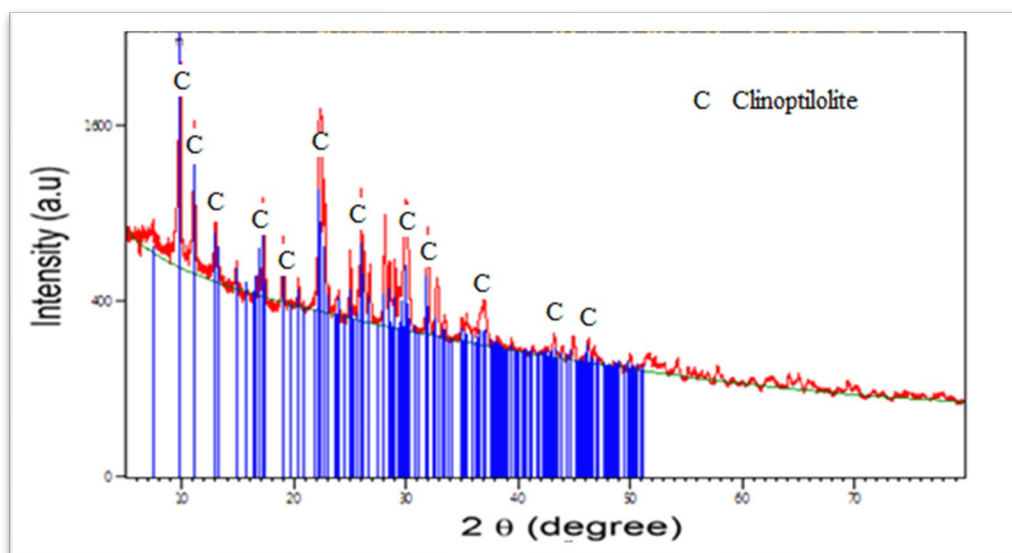


Fig. 5: XRD analysis showing the mineralogical analysis of Natural zeolite (Natural Zeolite).

3.4. X-Ray Fluorescence (XRF) result

The comes about of the XRF examination are displayed in Table (2). Zeolites test was composed fundamentally of Silicon (Si) and Aluminum (Al), Cations Such as sodium (Na^+), potassium (K^+), calcium (Ca^{2+}), or magnesium (Mg^{2+}), which adjust the negative charge of the aluminosilicate system. Zeolites test was contain follow sums of components like press (Fe), titanium (Ti). The result appears that SiO_2 , Al_2O_3 , Na_2O and K_2O are primary components of common zeolite with higher rate compared to EDS investigation result. These oxides speak to the interchangeable cations within the zeolite structure. Components like FeO , TiO_2 , or others were showing in little sums. The comes about of the EDS

examination too appeared that the major interchangeable cations within the common zeolite structure were Na^+ , Mg^{2+} , K^+ and Ca^{2+} .

Table 2: Chemical composition (wt.%) of the natural zeolite.

Element	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	K_2O %	CaO %	TiO_2 %	Fe_2O_3 %
wt.%	2.12	0.39	4.03	30.97	2.11	0.56	0.09	0.14

The Si/Al ratio was also calculated from the SiO_2 and Al_2O_3 content, the results was calculated as $(\text{SiO}_2\%/60.08) / (\text{Al}_2\text{O}_3\%/101.96) = (30.97/60.08) / (4.03/101.96) \approx 13.37$, high Si/Al ratio (>10) which is indicate a more hydrophobic and thermally stable zeolite and lower ion-exchange capacity and acidity.

3.5. Thermogravimetric Analysis (TGA) result

Comes about gotten from TGA/DTG appear that, common zeolite tests were ceaselessly losing weight after warming up to 1000°C Fig. (6). The most reasons of this are due to lack of hydration and dehydroxylation forms. The DTA bend appears an endothermic crest at $50\text{--}150^\circ\text{C}$ (Sprynskyy, 2010). Agreeing to Perraki and Orfanoudaki (2004) weight misfortunes at $20\text{--}100^\circ\text{C}$ and $100\text{--}200^\circ\text{C}$ are due to hygroscopic water and freely reinforced water, individually (Dyer, 1988; Gottardi and Galli, 1985). In the first step, when the natural zeolite was heated, the intact water was eliminated at below 200°C . this is due to the removal of adsorbed water (physisorbed water) from the pores and surface. In the hydrated phases, dehydration occurs at temperatures mostly below about 400°C and is largely reversible. Dehydroxylation of the zeolite framework, leading to the removal of more strongly bound water (chemisorbed water) and some structural water (hydroxyl groups). This stage may overlap with the decomposition of any impurities (e.g., carbonates or organic matter). In the second stage hydroxyl groups were removed at temperature above 400°C (Colella and Wise,

2014). Then dehydroxylation took place in the range 400-600 °C (Ribero *et al.*, 1984). Salih, (2018) reported two forms of water molecules and hydroxyl groups existing in the structure of silicate minerals such as natural zeolite. Dehydroxylation of the zeolite framework, leading to the removal of structural water. This phase may overlap with the decomposition of any impurities (e.g., carbonates or organic matter). Framework collapse phase appear at above 800°C. The zeolite structure begin to break down, leading to further weight loss or phase transformation.

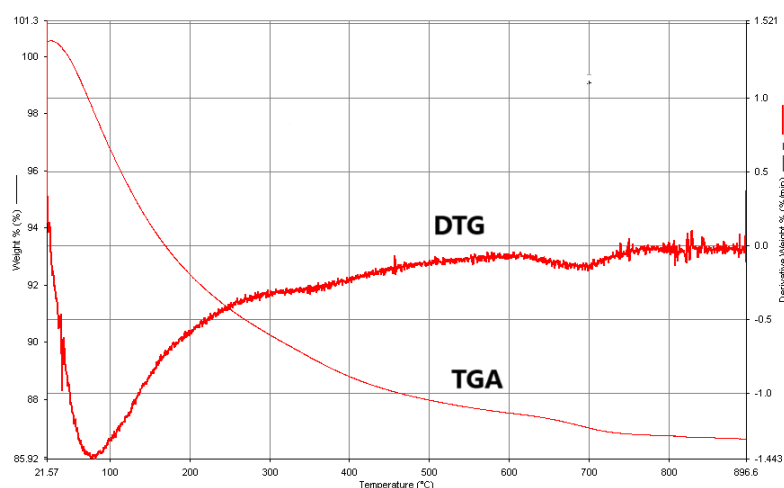


Fig. 6: Thermogravimetric analysis (TGA/DTG) of natural zeolite between 20-900°C.

According to the findings, natural zeolites are typically stable up to 400–600°C, after which they undergo framework collapse and dehydroxylation. Water from the zeolitic materials was responsible for any loss below 400°C. Thirteen weight percent was the total loss determined by the thermogravimetric analysis. Other authors (Al-Dwairi, 2009; Ugal *et al.*, 2010; Perraki and Orfanoudaki, 2004) also found similar values. Natural zeolite's DTG pattern shows a new peak around 750–800 °C, which could be related to the synthesis of sodalite and suggest the presence of impurities such carbonates or organic materials.

3.6.Inductively Coupled Plasma-Optical Emission Spectrometers (ICP-OES) results

The behavior of adsorbents, variables influencing the rate of adsorption, and the maximum effective capacity of natural zeolite towards the removal of Fe^{3+} , Cu^{2+} , Pb^{2+} , and Zn^{2+} ions are all displayed in the results of Inductively Coupled Plasma-Optical Emission Spectrometers (ICP-OES). The three adsorbent masses used in the kinetic studies were 2g, 4g, and 8g. A 100 ml solution of the suitable multi-component solution was combined with the adsorbent. For 360 minutes, samples were taken and analyzed hourly at an agitation speed of 150 rpm. The

zeolite samples that were used had a particle size of 250 μm . A pH adjustment of 4 ± 0.1 was made. The data unequivocally demonstrate that an increase in adsorbent mass led to an increase in heavy metal ion adsorption. The reason for this is that more adsorption sites are available per mass of adsorbent surface as the adsorbent mass grows, which raises the overall amount of metal removed. While the proportion of adsorbed Fe^{3+} and Pb^{2+} reached 90% in the first hour, the majority of the removal of Cu^{2+} , Fe^{3+} , Pb^{2+} , and Zn^{2+} ions takes place in the early stages (Fig. 7). Because there are more active sites for ion exchange and adsorption when zeolite mass increases, the total adsorption capacity for heavy metals often increases as well.

The information gotten from the motor adsorption tests were utilized to decide the evacuation capacity, q_e (mg/g) of the diverse adsorbents utilizing the taking after condition:

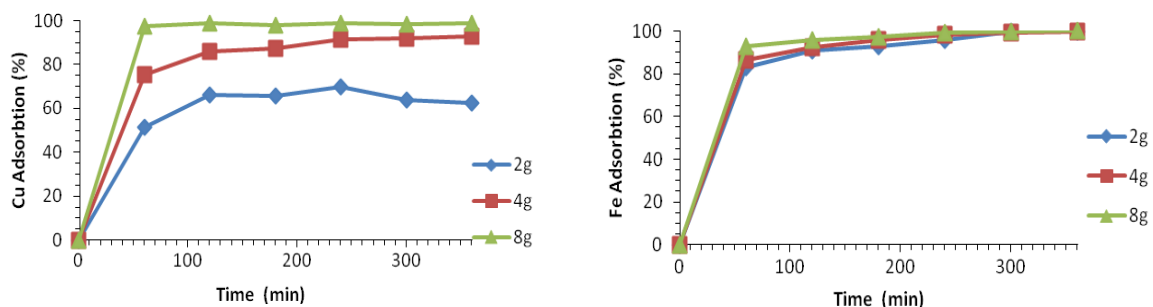
$$q_e = (C_o - C_e) \times V / m$$

The percentage removal of metal ions from solution was also determined using the equation below:

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

where,

q_e sum of adsorbate adsorbed per unit weight of adsorbent (mg/g) C_o and C_e are the starting and last metal particle concentrations in arrangement (mg/l) separately, V is the arrangement volume (l) and m is the weight of the zeolite utilized (g).



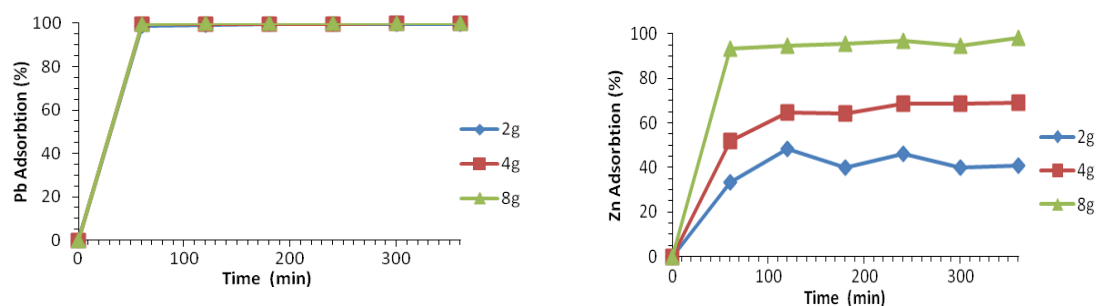


Fig. Error! No text of specified style in document..3: Shows the effect of the mass of natural zeolite on the adsorption of copper, iron, lead and zinc from solution.

4. CONCLUSION

The characterization results confirm that the natural zeolite was identified as clinoptilolite, with minor impurities such as quartz and clay minerals. The high crystallinity of the zeolite ensures a stable framework for adsorption processes. The zeolite crystals showed an irregular, plate-like morphology with a rough and porous surface. Visible pores and channels indicated a high surface area, suitable for adsorption.

Presence of exchangeable cations (Na^+ , K^+ , Ca^{2+} , and Mg^{2+}) enables the zeolite to effectively adsorb and exchange cations such as Fe^{3+} , Cu^{2+} , Pb^{2+} and Zn^{2+} from wastewater. The Si/Al ratio of 5.2 indicates moderate hydrophilicity, making the zeolite suitable for adsorbing polar molecules. Increasing the mass of zeolite generally increases the total adsorption capacity for heavy metals due to the availability of more active sites for ion exchange and adsorption. Chemical pre-treatment can enhance the adsorption capacity by removing impurities and increasing surface area. The framework remained stable up to 400°C , after which dehydroxylation and framework collapse occurred.

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