# AN X-RAY DIFFRACTION AND INFRARED SPECTROSCOPY STUDY OF TWO TYPES OF ZEOLITES SATURATED WITH ZN

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## ABSTRACT

**This research was included a method for preparing synthetic zeolite using araw clay mineral components found in nature (kaolin) and it was prepared by the hydrothermal method in the presence of sodium hydroxide. Natural zeolites were also used and saturated with zinc adopting the method of columns through $ZnSO_4.7H_2O$ solution. Several measurements were taken for the zeolites before and after the saturation process, the results obtained were ascribed to the X-ray diffraction (XRD) which showed a well crystallization rate while the chemical analysis of the elements showed that they contain a high percentage of silicon and aluminum, which gives evidence, is a balanced and equal reaction. The infrared spectrum (IR) showed the change in the frequency band of 1002 $cm^{-1}$, which expresses its sensitivity to the occurrence of the substitution process within the structural composition of the zeolite, and confirmation of this is the disappearance of the weak shoulder from the right side of the diffraction 1002 $cm^{-1}$, which represents the voids in the structural composition of the mineral, which was accompanied by the widening of the diffraction itself, it confirms the occurrence of replacement of zinc in those voids.**

**Key words: natural zeolite, synthetic zeolite, kaolin, slow release fertilizers, Zn deficiency**



# دراسة حيود الاشعة السينية والتحليل الطيفي للأشعة تحت الحمراء لنوعين من الزيولايت المشبع بالزنك

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**المستخلص:**

**يحتوي البحث على طريقة لتحضير زيولايت صناعي بالاستفادة من مكونات خام معدنية طينية موجودة في الطبيعة (الكاولين) وتم تحضيره بالطريقة الهيدرو حرارية بوجود هيدروكسيد الصوديوم, كما استخدم الزيولايت الطبيعي وتم تشبيعهما بعنصر الزنك بطريقة الاعمدة من خلال محلول محلول $ZnSO_4.7H_2O$. واجريت العديد من القياسات على الزيولايت قبل وبعد التشبيع وهي نتائج حيود الاشعة السينية (XRD) والتي بينت نسبة تبلور جيدة,في حين بين التحليل الكيميائي للعناصر احتوائها على نسبة عالية من السليكون والالمنيوم ويعطي دليل على تفاعل متوازن ومتكافيء , وطيف الاشعة تحت الحمراء (IR) والذي بين التغيير الحاصل في حزمة التردد 1002سم$^{-1}$ والتي تعبر عن حساسيتها لحدوث عملية الاحلال Substitution ضمن التركيبة البنائية للزيولايت ,وتأكيداً على ذلك هو زوال الكتف الضعيف weak shoulder من الجهة اليمنى للحيود 1002سم$^{-1}$ والذي يمثل الفراغات الموجودة في التركيب البنائي للمعدن والذي رافقه اتساع الحيود نفسه الامر الذي يؤكد حدوث احلال للزنك في تلك الفراغات.**

**الكلمات المفتاحية: الزيولايت الطبيعي، الزيولايت الصناعي، الكاولين، اسمدة بطيئة التحررالزنك، نقص الزنك**



**Received:24 /6/2023, Accepted:23/10/2023, Published:December 2025**

## INTRODUCTION

Calcareous soils have a deficiency for most micronutrients, including zinc. The fertilizers used to treat zinc deficiency are mineral fertilizers and organic chelated compounds. However, Many researchers have used these fertilizers to treat zinc deficiency in Calcareous soil(3,24,21).soils these fertilizers are not highly efficient in treating the deficiency. Therefore, we will try to manufacture a slow-release zinc fertilizer from the zeolite mineral. Zeolite is a sedimentary volcanic mineral mainly consists of aluminous silicates which has a crystalline structure in the form of three-dimensional networking (15) Zeolite is formed in natural conditions in the presence of alkaline water containing a high percentage of salts that interact with volcanic ash forming a crystalline formation (18,20). Natural zeolite can be obtained from deposits or through its manufacture. The general crystal structure of zeolite minerals consists of the association of the tetrahedra units in the form of double or symmetrical circular rings, and each of these rings is composed of 4, 5, 6, 8 or 12 tetrahedra units that are linked together giving the tubular shape of the mineral. The size of those pipes or channels within the metal composition depends on the number of the tetrahedra units involved in the formation of the circular rings within the general structure and the size of these channels increases with the increase in the number of the tetrahedra units participating in the formation of the minera building rings. Zeolite mineral contains pores with a molecular size of (4Aº). It has a crystalline structure consisting of ring chains of tetrahedra that are connected to each other through positive ions such as sodium and potassium. There are 50 types of natural zeolite that have been identified and about 150 types that have been formed synthetically. The most well-known types of natural zeolite are shabazit, analcite, philistite, clinopetlite, mordenite, ironite and ferrite. It is found in many countries around the world such as Italy, Turkey, Russia, South Africa, Syria, Jordan and Yemen. Quantities of this mineral are used around the world estimated at 3 million tons annually, of which 18% is natural zeolite and the rest is a synthetic mineral (19). The structural composition of open zeolite gives it unique and distinctive characteristics compared to other silicate minerals (8,1). and synthetic zeolites can be manufactured in the form of powder or granules (11, 14), previous studies revealed that the preparation of synthetic zeolites from chemical sources of silica and alumina is more expensive compared to the conversion of raw materials (i.e., natural sources of silica). and alumina) using the hydrothermal method (23,12). synthesized zeolite A using rice husks as a source of silicon and sodium hydroxide (NaOH)(13). The extraction of silica in the form of sodium silicate through the decomposition of rice husk ash at temperatures 600Cº(16,22). also used coal ash to manufacture zeolites successfully. The clay mineral ores (silica and bauxite) are an important and main source with economic feasibility for the production of the basic materials used in the preparation of zeolite because the silica ore contains a percentage of silica that can interact with the base, so it turns into sodium or potassium silicate, depending on the type of base, which is estimated at 31.2%. While bauxite ore contains 54.02% aluminum, which is converted to sodium aluminate. They are the two main elements for the preparation of zeolite by the hydrothermal method. (21, 3, 17) experimented to prepare synthetic zeolite from natural raw materials, which used kaolin as a raw material for the preparation of zeolit and characterized kaolin and zeolite prepared by X-ray (RXD)at temperature of 50Cº All these conditions led to obtaining industrial zeolite with a degree of crystallinity of 90% and cubic crystals of zeolite A. There are no discoveries of zeolite minerals in Iraq. Therefore, the study aims to manufacture zeolite from the kaolin mineral available in the Anbar desert in western Iraq and saturate it with zinc to be used as a slow-release fertilizer in Calcareous soils that suffer from its deficiency, and it is important to study X-ray diffraction and infrared spectroscopy of natural and industrial zeolite before and after its zinc saturation.

## MATERIALS AND METHODS

The natural zeolite of Jordanian origin was used in this experiment. As, the synthetic zeolite was manufactured from kaolin clay, which was prepared by the General Authority of Geological Survey. Kaolin clay is available in the Duwakhla region, west of Anbar. The mineral was identified by X-ray and IR at the Ministry of Science and Technology - Department of Environment and Water. Kaolin samples were taken, crushed and ground into powder, and sieved in a laboratory sieve with a diameter of 75 µm <dp< 125 µm. The sieved kaolin was treated with a thermal treatment at a temperature of 600 °C for 3 hours to convert it into metakaolin. This will change its composition as it is illustrated in the following equation:

$Al_2Si_2O_5$ $(OH)_4$ (kaolin) → $Al_2Si_2O_7$ (metakaolin) + $2H_2O$

Metakoline was treated with a 4M sodium hydroxide solution (5:1), placed in a volumetric flask with a capacity of 1000 ml, and mixed in a magnetic bar for 10 minutes until the mixture became homogeneous. The mixture was left for 24 hours at room temperature. For every 200 grams of metakaolin, 1000 ml of sodium hydroxide solution is added. After 24 hours, the solution is shaken and heated by a Magnetic stirrer heater (the volumetric flask is equipped with a thermometer to measure the temperature of the solution to set the temperature at 80 °C) and for two hours to prepare the synthetic zeolite from metakoline as shown in the following equation:

$6Al_2Si_2O_7$ (metakaolin)+12NaOH→Na12 $(AlO_2)$12$(SiO_2)$12 (zeolite A).$27H_2O$ + $6H_2O$

After heating the mixture, it was filtered through a Buechner funnel using a Section pump and washed with distilled water to remove excess alkalinity. The washing process was repeated several times until the pH reached 10.5. They are dried in an electric oven at a temperature of 100°C for four hours, then ground and sifted through a sieve with a diameter of 63 µm (4).

**Measuring the properties and specifications of natural and synthetic zeolite**

Conducting several measurements on zeolite is to identify its properties and specifications, namely: chemical analysis, X-ray diffraction (XRD) and infrared (IR) spectroscopy.

**Zinc saturation of zeolite**

500 grams of natural and synthetic zeolite were taken, and two solid polyethylene columns with a length of 60 cm and an inner diameter of 7.1 cm were used in this study. The columns were fixed on a wooden board with a height of 30 cm to facilitate the process of carrying the tubes and to facilitate collection of the solution filtrate. A perforated plastic cover wasfixed at the bottom of the columns. It is connected to a rubber tube with a glass beaker for the purpose of collecting the solution. Filter paper and a layer of washed coarse sand with a height of 2 cm are placed on the lid. The columns are filled with natural and synthetic collected in a glass beaker each column separately in order to measure the concentration of zinc in the filtrate solution and thus know the ability of the zeolite to saturate with Zn fixed at the bottom of the columns. It is connected to a rubber tube with a glass beaker for the purpose of collecting the solution. Filter paper and a layer of washed coarse sand with a height of 2 cm are placed on the lid separately in order to measure the concentration of zinc in the filtrate solution and thus know the ability of the zeolite to saturate with Zn. (10) This method is similar to the method of saturating humic acids with micronutrients.

## RESULTS AND DISCUSSION

**X-ray examinations of the natural and natural zeolite mineral saturated with zinc :**

After analyzing natural zeolite and natural zeolite saturated with zinc, the results listed in Table 1 were obtained. The results of the chemical analysis of the elements present in the natural zeolite showed that it contains silica at a high percentages (44.172%), and medium percentages of (MgO, $Al_2O_3$, CaO, $Fe_2O_3$) (11.40, 10.07, 18.06, 10.67) respectively, and small percentages of $Na_2O$, $K_2O$, TiO, which are 2.35, 2.83, 1.76, respectively.

**Table 1. Chemical analysis of elements present in natural zeolite and natural zeolite saturated with zinc calculation in form of oxide**

| Metal Oxides | % Ratio |
|---|---|
| **Natural Zeolite** | |
| $Na_2O$ | 2.35 |
| MgO | 10.67 |
| $Al_2O_3$ | 18.06 |
| $SiO_2$ | 44.17 |
| $K_2O$ | 1.76 |
| CaO | 10.07 |
| TiO | 2.83 |
| $Fe_2O_3$ | 11.40 |
| **Natural Zeolite Saturated with Zinc%** | |
| MgO | 9.11 |
| $Al_2O_3$ | 14.52 |
| $SiO_2$ | 42.41 |
| $SO_3$ | 1.38 |
| $K_2O$ | 1.89 |
| CaO | 9.47 |
| TiO | 2.80 |
| $Fe_2O_3$ | 13.03 |
| ZnO | 3.90 |

to the columns in the form of a solution of $ZnSO_4.7H_2O$ at a concentration of 5000 mg $L^{-1}$. The percentages of clay and non-clay minerals present in natural zeolite and natural zeolite, each separately, and sifted through a sieve with a diameter of 63 µm. Zinc was added The solution is added manually gradually to the columns until each quantity is finished, then the zeolite in the columns is washed with distilled water twice, and the filtrate of the solution is zeolite saturated with zinc were calculated, as it is noted that the zeolite included minerals containing silicon at a high percentage in its composition, such as chlorite $(Si¸Al)_4$ $O_{10}(OH)_8$ and quartz SiO, which totaled (65.26%). which corresponds to the chemical analysis of the mineral, which showed that it contained high levels of silica, and when the zeolite was When the natural zeolite was saturated with zinc, the proportions of the elements were similar, with the presence of zinc in the form of ZnO at a rate of 3.90%.saturated with zinc, the percentage of dolomite mineral increased to 20.04%, and calcite mineral did not appear, as shown in Table 2 .

**Table 2. Percentages of clay and non-clay minerals for natural zeolite and natural zeolite saturated with zinc by X-ray (XRD)**

| Minerals | Percentage Ratio |
|---|---|
| **Natural Zeolite** | |
| Chlorite | 28.42 |
| Kaolinite | 3.16 |
| Quartz | 36.84 |
| Mica | 4.21 |
| Halite | 3.16 |
| Dolomite | 10.53 |
| Calcite | 13.68 |
| **Natural Zeolite Saturated with Zinc** | |
| Chlorite | 25.07 |
| Kaolinite | 9.25 |
| Quartz | 31.38 |
| Mica | 5.20 |
| Halite | 9.06 |
| Dolomite | 20.04 |

The natural zeolite mineral sample was diagnosed by X-ray diffracted. (Fig. 3) showed the appearance of diffractions of 8.58, 4.23, 3.57, and 2.88 A°, which all belong to the group of zeolite minerals, especially the Chabazite mineral, which is one of the group members of zeolites. The d-spacing diagnosed in the examined sample is highly identical to the base distances of the Chabazite mineral mentioned by (11,8) opinions go with Dixon who diagnosed that the Chabazite mineral at the base distances were 8.93, 4.46, 3.88, 2.89 A°, the first diffraction appeared with high intensity (strong) at the d-spacing of 8.58 A° and the second with medium intensity at the d-spacing of 4.23 A°, which was largely identical to the natural zeolite sample examined in the current study. The variation in the values of the d-spacingrecorded in the examined sample and those recorded in previous studies showed a minor difference. Byrappa (8) Showed that this discrepancy may attribute to many causes including the discrepancy in the sizes of the prepared metal particles or the preparation and arrangement of the samples before the examination or the difference in the the diffraction of the examined metal before and after its saturation with a certain element. These results go along with the previous results of several studies; (8,11) that the diffraction of zeolite minerals is not affected by saturation with a certain type of cation, organic molecules, or even thermal treatments. And as shown by (11), the Chabazite mineral, which is one of the members of the zeolite mineral group, is considered to have a structural structure that is solid and open enough to accommodate and saturate with the elements Li, Na, Ag, Sr, Ba, k, $NH_4$, Rb, Ti Despite this, no discrepancy was found in the d-spaci of the diffraction of the mineral before and after saturation when examined by X-rays. ratio of Si / Al in the composition of the prepared mineral or the system and conditions of crystallization or the accuracy of the diagnosis of X-ray results. (Fig. 3), gave the same diffraction and at the same d-spaci recorded in the sample of natural zeolite examined before saturation with the zinc ion, as the X-ray examination technique was not able to show the variation obtained for the d-spaci of.

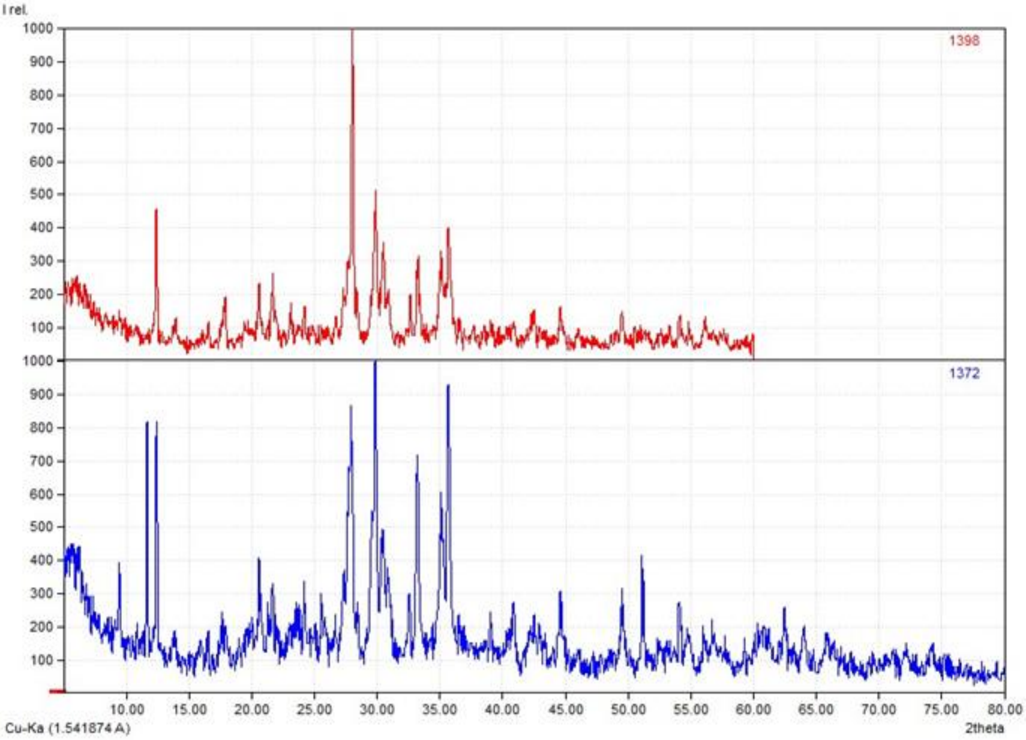


**Fig 1. X-ray diffraction of natural and natural zinc-doped zeolite)**

**Synthetic zeolite and synthetic zeolite saturated with zinc :**An analysis was conducted for the synthetic zeolite. Some results were obtained and listed in Table 3. The results of the chemical analysis of the elements found in the synthetic zeolite showed that it contains high percentages of silica (44.402%) and $Al_2O_3$ by 20.23%, and an average percentage of $Na_2O$ 18.939%, and small percentages of $Fe_2O_3$, TiO, which are 1.207 and 1.177%. when zeolite was saturated with zinc, these results obtained through an analysis were made for zinc oxide with a percentage of 3.66% and $SO_3$ with a percentage of 1.42%.

**Table 3. Chemical analysis of the elements present in industrial zeolite and synthetic zeolite saturated with zinc, calculated in the form of oxides**

| Synthetic zeolite Metal Oxides | % |
|---|---|
| $Na_2O$ | 18.94 |
| $Al_2O_3$ | 20.23 |
| $SiO_2$ | 44.40 |
| TiO | 1.21 |
| $Fe_2O_3$ | 1.18 |
| **zeolite saturated with zinc** | **%** |
| $Na_2O$ | 15.59 |
| $Al_2O_3$ | 25.36 |
| $SiO_2$ | 42.74 |
| $SO_3$ | 1.42 |
| CaO | 1.07 |
| TiO | 1.48 |
| $Fe_2O_3$ | 1.42 |
| ZnO | 3.66 |

The percentages of clay and non-clay minerals found in synthetic zeolite were calculated and listed in Table4.

**Table 4 . Percentages of clay and non-clay minerals for natural zeolites by X-ray diffraction (XRD)**

| Synthetic zeolite | |
|---|---|
| **Minerals** | **%** |
| **Chlorite** | **5.41** |
| **Kaolinite** | **19.82** |
| **Quartz** | **15.53** |
| **Mica** | **10.81** |
| **Halite** | **11.71** |
| **Dolomite** | **5.41** |
| **Sepiolite** | **13.68** |
| **Cristobalite** | **14.41** |
| **zeolite saturated with zinc Synthetic** | **%** |
| **Chlorite** | **3.19** |
| **Kaolinite** | **29.80** |
| **Quartz** | **16.49** |
| **Mica** | **12.37** |
| **Halite** | **4.12** |
| **Dolomite** | **2.06** |
| **Sepiolite** | **19.59** |
| **Cristobalite** | **12.37** |

It is noted from the results obtained that zeolites contain minerals that contain silicon at a high percentage in their composition, such as chlorite $(Si, Al)_4\ O_{10}\ (OH)_8$,quartz SiO, sepiolite $Mg_4Si_6O_{15}\ (OH)_2.\ 6H_2O$, and cristobalite, which is a form of silica, and totaled (48.75%) which corresponds to the chemical analysis of the mineral, which showed that it contains high levels of silica. As the results of synthetic zeolite saturated with zinc seem so close to the proportions of the unsaturated synthetic zeolite with zinc. A sample of synthetic zeolite was detected in the current study using X-ray diffraction, which gave the following diffraction 8.58, 4.23, 3.57, and 2.88 A° showed the first diffraction with a medium intensity at the base distance of 4.23A°, and that intensity depends on the content and degree of crystallinity, whose d-spacing was identical to those appearing in the natural zeolite sample which belongs to the Chabazite mineral. Sometimes, the diffraction of zeolite minerals may interfere with the diffraction of some common soil minerals, such as quartz, gypsum, halite, Palygorskile, Alkali feldspars, and mica. The samples examined (natural and synthetic) in the current study are pure not taken from soil samples, therefore, the diffractions identified for the zeolite minerals in the current study are ascribed to the Chabazite mineral, not as a result of its interference with other soil minerals. A sample of synthetic zeolite was diagnosed after loading it with Zn ion. The same diffraction and d-spacing obtained when examining the sample before saturation with zinc. These results are consistent with many studies (8,11) showed that the diffraction of zeolite minerals is not affected by the saturation process with a certain type of cation, organic molecules, or even thermal treatments. (11), and confirms the sample belong to the Chabazite mineral, which is one member of zeolite minerals, and has a solid and open structure provided to accommodate and saturate with elements Li, Na, Ag, Sr, Ba, k, $NH_4$, Rb, Ti. Despite this, no variation was found in the d-spacing of the diffraction of the mineral before and after saturation with Zn.

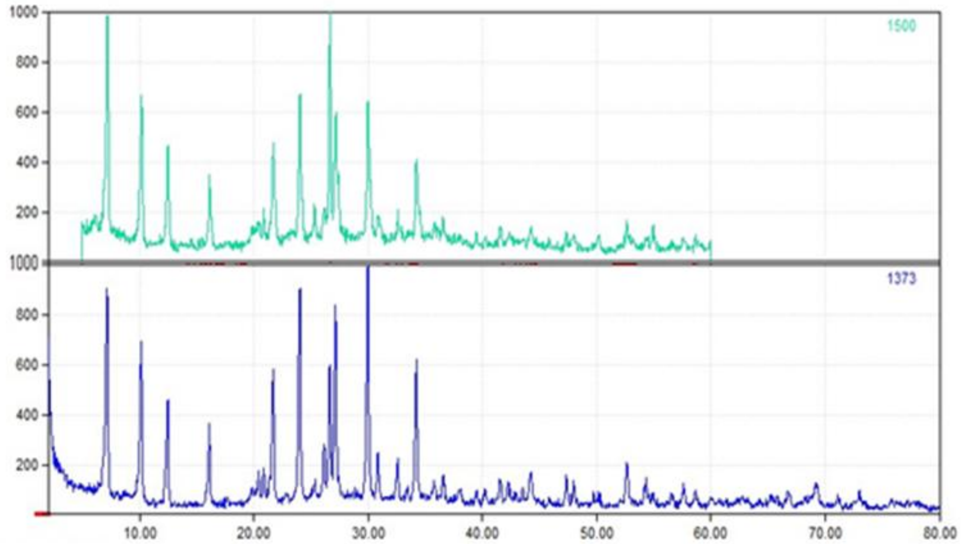


**Fig 2. The X-ray diffraction of industrial and Synthetic zeolite mineral saturated with zinc**

**Infrared spectrum examinations of the natural and natural zeolite mineral saturated with zinc:** (6 ،7 ،11) studies identified the main frequencies of the zeolite minerals spectrum bands. They showed the main frequencies of those minerals commonly occurring within the range of 400-1500 $cm^{-1}$. The aforementioned range generally contains two types of frequencies

1. The internal frequencies resulting from the $TO_4$ tetrahedra units, which are called the primary building unit (PBU), are present in all members of the group of zeolite minerals, in which they are sensitive to any change in the structural composition of these minerals.
2. The external frequencies resulting from the association of the tetrahedra units with each other, and are called the secondary building units (SBU), which they are sensitive to the occurrence of any addition (secondary building) to the general framework of the structural composition of zeolite minerals, such as the addition of structural rings to the building, or the presence of voids within the crystal structure.

**Natural zeolite samples:**

The existence of a sharp band at or around 1100 $cm^{-1}$ in general is an exclusive feature of the structural composition of zeolite minerals (12), which represents the Si-O bond, and this band is usually sensitive to the substitution process within the structural composition of zeolite minerals. The lack of broadening of the band at the frequency 1100 $cm^{-1}$ indicates its presence of the symmetric band for the $SiO_4$tetrahedra units .Therefore, the appearance of non-wide diffraction at frequency 1020$cm^{-1}$ in the examined sample (natural zeolite) indicates the presence of zeolite in the sample, and that it expresses the Si-O bond, and that the $SO_4$tetrahedra units constituting the mineral are present in the form of symmetric bands.

-The appearance of a weak shoulder on the right side of the diffraction 1020 $cm^{-1}$ in the sample expresses the presence of nests (voids) of the Al-OH bond (Nests of Al-OH), and these results in agreement with what (6).

-The appearance of band at a frequency of 462 $cm^{-1}$ represents the Si-O-Si bond, and that frequency is a distinctive feature of the zeolite minerals, which are characterized by the presence of four tetrahedra units that form the rings of the zeolite mineral.

-The presence of the two diffractions at frequencies 1641 and 1425 $cm^{-1}$ confirms the presence of the frequency band of water molecules in sample.

-The appearance of the frequency band at 3761$cm^{-1}$ represents the adsorption of the OH group on the surface of the zeolite (7).

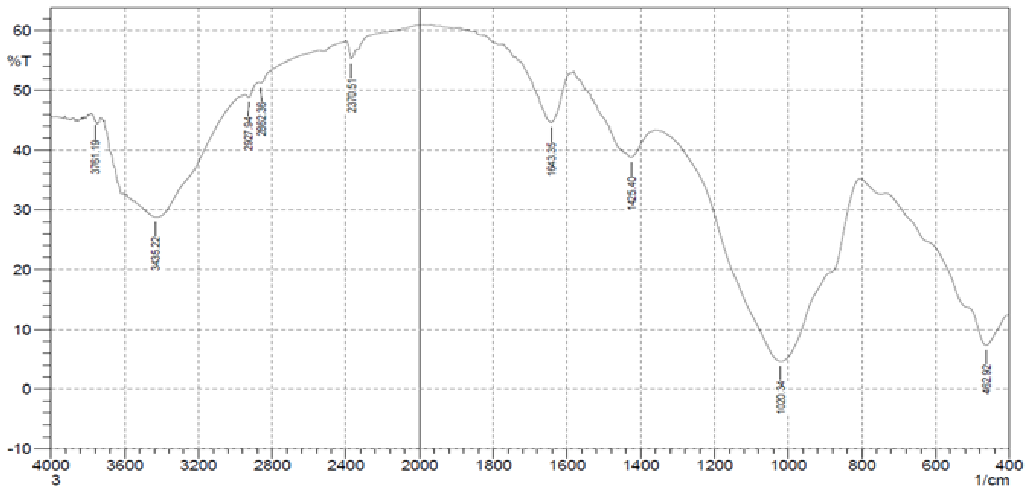


**Fig 3 . Infrared spectroscopy of natural zeolite**

**Natural sample saturated with Zn :**The most prominent change when the sample was examined is the appearance of wide bandat frequency of 1002 $cm^{-1}$ compared to the natural zeolite sample, which indicates that the tetrahedra units exist in an asymmetric band.

The change that occurred in the frequency band 1002 $cm^{-1}$expresses its sensitivity to the occurrence of the substitution process within the structural composition of zeolite, and the disappearance of the weak shoulder at the right side of the diffraction 1002 $cm^{-1}$ confirms presence of the in voids in the structural composition of the mineral, which was accompanied the widening of frequency band and confirmed the occurrence of zinc substitution in those voids.

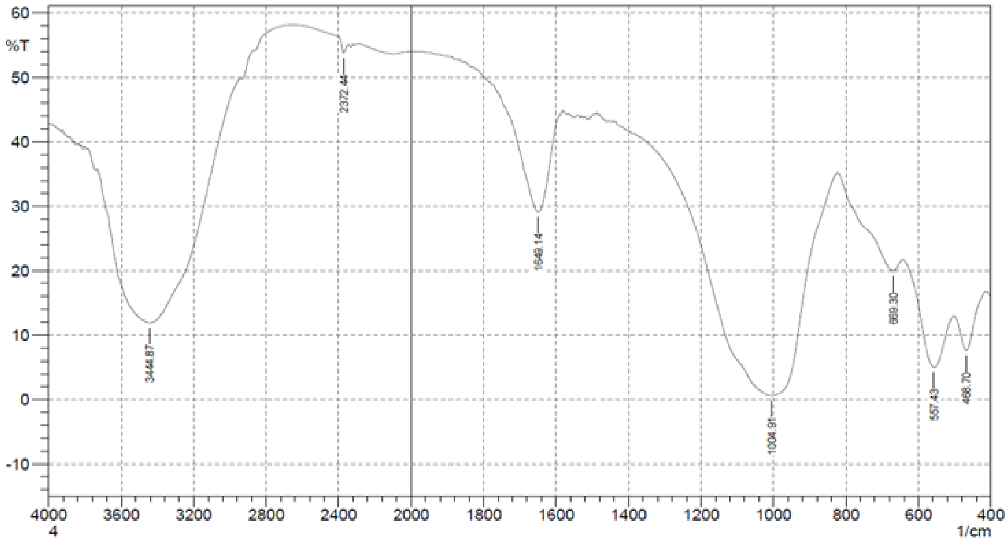


**Fig 4 . Infrared spectrum examination of natural zeolitesaturated with zinc**

**Synthetic Zeolite:**

The results of the infrared spectrum examination of the synthetic zeolite mineral showed that there is a medium expansion at frequencies of 468.7 and 555.5 cm$^{-1}$ in the C-X bond. It gave an average expansion at 1653.00 cm$^{-1}$, while the expansion in C=C band was very weak and appearead at bond at frequency of 2374.37 to 2929.87 cm$^{-1}$, a medium expansion occurred for the C-H bond, while the frequency 3437.15 cm$^{-1}$ gave a wide expansion for the O-H bond, Figure 5

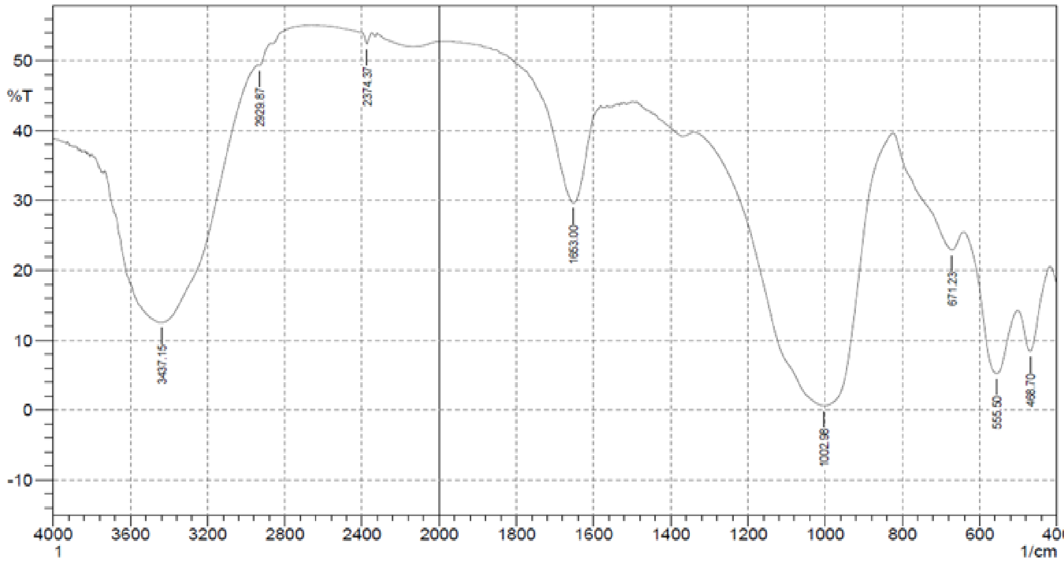


**Fig 5. Infrared spectrum examination of synthetic zeolites**

**Zinc-Saturated Synthetic Zeolite**

The results of the infrared spectrum examination of the synthetic zeolite mineral saturated with zinc showed that there was a medium band at the frequency of 464.84 cm$^{-1}$ which is belong to stretching of C-X band, while the wide bands at frequencies of 1020.34 cm$^{-1}$ and 1643 cm$^{-1}$ reflects a strong expansion of C-O bond, While for the C-C bond, a moderate expansion appeared at the frequency 1431.18 cm-1. The C=C bond had a weak and clear expansion in the C=N bond at the frequency 2376.30 cm-1, and at the frequency 2924.09 and 2856.58 cm$^{-1}$, the expansion was moderate in the bond, C-H and wide in the O-H bond. The frequencies (3435.22 and 3759.26 cm$^{-1}$) showed a strong expansion of the O-H bond, as shown in Figure 6.

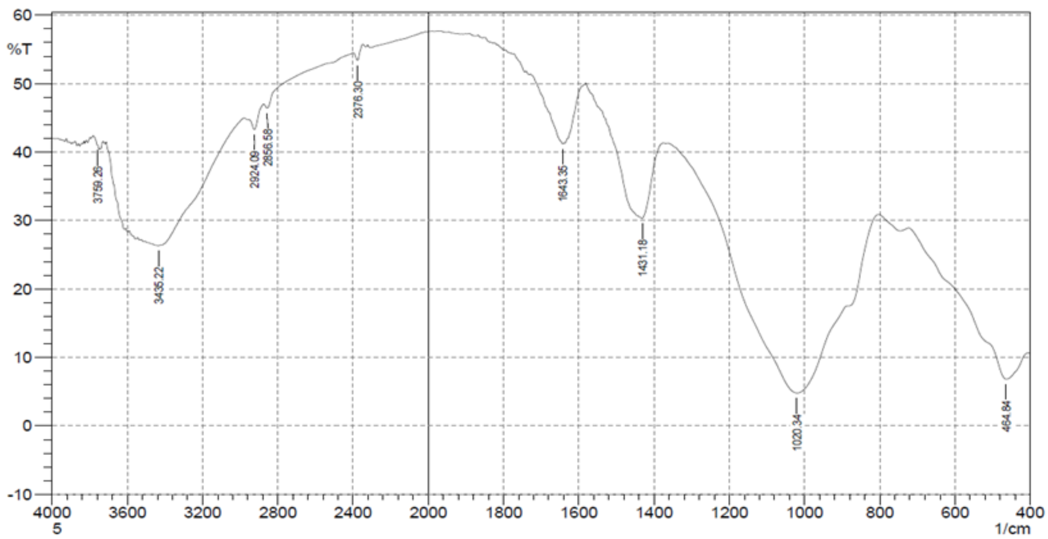


**Fig 6. Infrared spectrum examination of synthetic zeolite saturated with zinc**

While for the C-C bond, a moderate expansion appeared at the frequency 1431.18 cm-1. The C=C bond had a weak and clear expansion in the C=N bond at the frequency 2376.30 cm-1, and at the frequency 2924.09 and 2856.58 cm-1, the expansion was moderate in the bond, C-H and wide in the O-H bond. The frequencies (3435.22 and 3759.26 cm-1) showed a strong expansion of the O-H bond, as shown in Figure 6.

## CONCLUSIONS

X-ray and infrared spectroscopy have shown that synthetic zeolite can be prepared from natural kaolin with high efficiency. Natural and synthetic zinc zeolites are also efficient fertilizer sources in terms of their ability to provide zinc and protect it from precipitation and fixation.

## CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

## DECLARATION OF FUND

The authors declare that they have not received a fund.

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