

# Effect of Polyurethane on the Structure and solubility of Gypsum Soil

Atheer G. Ibrahim <sup>1,\*</sup>, Maysam Th. Al-Hadidi <sup>2</sup>

<sup>1</sup>National Centre for Water Resources Management, Ministry of Water Resources, Baghdad, Iraq

<sup>2</sup>Water Resources Engineering Department, University of Baghdad, Baghdad, Iraq

\*Corresponding Author E-mail: [AtheerGurjee1993a@gmail.com](mailto:AtheerGurjee1993a@gmail.com)

## Abstract

Gypsum soil is a problematic soil when its gypsum particles are contact with water. The objective of the research is studying the effect of adding polyurethane on the structure of the gypsum soil. This study is continuing to a previous study which explain the process of the treating gypsum soil that subject to water flow in a flume in an experimental canal. The samples of this study have been taken for testing them which stored for four years since 2018, along this period the samples were stored in a room temperature. In this study, these samples subjected to XRD, XRF, and SEM tests to explain the structure of the soil, before and after the treatment. The gypsum content test is done and the results are 41%. The results of XRD test proved that there was low effect of gypsum in the treated soil which have low gypsum index at 2-Theta value due to presence of polyurethane, while the results of XRF test explained that the concentration of elements in treated soil is greater than untreated soil and the reduction in concentrations of the elements Fe, Ca, K, S, and Zn are 83%, 32%, 54%, 85%, and 95%, respectively. In SEM test, the results showed that the gypsum soil became more cohesive due to the addition of the polymer material specially at the zoom 1.00 and 5.00 Kx.

**Keywords:** XRD, XRF, SEM, Gypsum soil, Polyurethane.

## تأثير مادة البولي يوريثان على بنية وذوبان التربة الجبسية

اثير كرجي ابراهيم<sup>1\*</sup> , ميسم ثامر الحديدي<sup>2</sup>

<sup>1</sup> المركز الوطني لادارة الموارد المائية – وزارة الموارد المائية – بغداد

<sup>2</sup> قسم هندسة الموارد المائية، جامعة بغداد، بغداد، العراق

\*ايميل المؤلف المرسل: Atheergurjee1993a@gmail.com

### الخلاصة

تعتبر التربة الجبسية من التربة التي تسبب مشاكل عندما تلامس جزيئاتها الجبسية الماء. الهدف من البحث هو دراسة تأثير إضافة مادة البولي يوريثان على بنية التربة الجبسية. هذه الدراسة هي استكمال لدراسة سابقة توضح عملية معالجة التربة الجبسية الخاضعة لجريان الماء في قناة مختبرية. تم أخذ عينات هذه الدراسة لفحصها وتم تخزينها لمدة أربع سنوات منذ عام 2018، وخلال هذه الفترة تم تخزين العينات في درجة حرارة الغرفة. في هذه الدراسة تم إخضاع هذه العينات لاختبارات XRD و XRF و SEM لتوضيح بنية التربة قبل وبعد المعالجة. تم إجراء اختبار محتوى الجبس وكانت النتائج 41%. أثبتت نتائج اختبار XRD انخفاض تأثير الجبس في التربة المعالجة حيث كان مؤشر الجبس المنخفض بقيمة 2-ثباتا بسبب وجود مادة البولي يوريثان، في حين أوضحت نتائج اختبار XRF أن تركيز العناصر في التربة المعالجة أكبر من التربة غير المعالجة حيث كانت نسبة الانخفاض في تركيز العناصر Fe و Ca و K و Zn و S هي 83% و 32% و 54% و 85% و 95% على التوالي. وفي اختبار SEM أظهرت النتائج أن التربة الجبسية أصبحت أكثر تماسكا نتيجة إضافة مادة البولي يوريثان خاصة عند قيم التكبير 1.00 و 5.00 Kx.

الكلمات المفتاحية: SEM, XRF, XRD, تربة جبسية, بولي يوريثان

## 1. Introduction

Gypsum soil is a type of soils which has many problems specially when contact with water. (Al-Hadidi & Ibrahim, 2018) used polymer material to reduce solubility and improve the hardness of the gypsum soil with gypsum content 41%. They proved that best cover ratio is 10% which gave small corrosion about 3% after 28 days.

(Ibrahim, et., al., 2022) proved that there are many ways to control the solubility of the soil and hardening it such as clinker additive, fuel oil, bentonite and kaolinite, acrylate, nano Materials, cutback asphalt, and cement.

(Buck & Van Hoesen, 2002) used soils from southern New Mexico, and proposed a new name paedogenic gypsum morphology called snowball morphology. This gypsum can occur in (0.5 – 1) mm, by using Scanning Electron Microscopy (SEM) test. When there is a combination between the morphology and other development indicators of the soil, the snowball may be used to calculation geomorphic surfaces and relative ages of soil.

(Jha & Sivapullaiah, 2014) used X-ray analysis for soil with variable contents of gypsum, the results showed a formation of new peaks and when there was addition of gypsum, the unconfined compressive strength of soil will deteriorate.

(Al-Barrak & Rowell, 2006) used gypsum soil from Saudi Arabia to determine the gypsum dissolution by formation of calcite coatings during leaching process. The results showed that there was dissolving in the initial gypsum content but in the case of sandy clay loam, between one-quarter of gypsum and one-third, there was no dissolve. The case of sandy clay about one-fifth could not dissolve. Chemical tests including SEM showed the formation of calcite coatings on particles of the gypsum.

(Weindorf, et. al., 2009) used gypsum soil from Southern New Mexico and West Texas. The samples which collected from previous sites were scanned XRF test. It was a comparison between three sets of data: soil characterization data, quantitative X-ray diffraction, and portable XRF. The best correlation of XRF data for gypsum was between quantitative X-ray diffraction and PXRF ( $R = 0.96$ ), the results also showed that PXRF provided 6% results of soil characterization data.

(Yilmaz & Civelekoglu, 2009) treat the expansive clay soil with adding gypsum as a treatment material. Gypsum quantities (2.5, 5, 7.5 and 10%) by mass and XRD test were used. After 7 days, they proved that gypsum content can change the swell percent, strength parameters, and plasticity for untreated and treated soil.

(Kordlaghari & Rowell, 2006) used three soils from UK, Iran, and Saudi Arabia which the PH and Organic matter values were (7.8, 7%), (8.2, 1.4%), and (7.8%, 1%) respectively. The soil from Saudi Arabia was shaken by the solution  $\text{KH}_2\text{PO}_4$  for 24 hours in 10mm  $\text{CaCl}_2$ . Scanning electron micrographs (SEM) test was approved that there was disappearing in gypsum small crystals and forming of calcium phosphate.

(Ebailila et. al., 2022) Studied gypsum effect with varying concentrations (0, 3, 6 and 9%) by weight, equivalent to the contents of sulphate (0, 1.4, 2.8 and 4.2, respectively, of sulphate soil performance which stabilised two levels of lime (4 and 6% by weight). The results showed that the expansion and the strength were proportional to the lime and sulphate content.

(Al-Jassim & Al-Hadidi, 2020) studied the effect of cement material on gypsum soil in irrigation canals by restationing system. The results show that this system reduce canal scouring in untreated soil by 56.6% and 82% in treated soil. In spite of that, the soaking and wetting could affect gypsum soil, but when there is using improving material, this effect could be reduced, (Ahmed, et. al., 2020), (Al-Nedawi & Al-Hadidi, 2020).

(Danoosh & Al-Hadidi, 2022) studied the effect of rationing system on earth canal stability during rapid drawdown of the water. They used a canal called Birmana as a case study in Iraq. The software Geo-Studio was used and the results of a minimum factor of safety were 1.2, 1.159, 1.142, and 1.161 for Spencer, Morgenstern, Bishop, and Janbu methods.

(Toma & Al-Hadidi, 2022) studied soaking and wetting effect on gypsum soil by using polyurethane material. The results were a 10% increase in the durability of the soil and there was a decrease in the collapse potential when the number of wettings increased.

There was not any study that explains the effect of polyurethane on gypseous soil, so the objective of this study is to show the structure and the solubility of gypseous soil after treatment with polyurethane material as an additive.

## 2. Methodology

The methodology of this research is represented by taking the samples from a previous study made by authors (Al-Hadidi & Ibrahim, 2018) as mention above and made a lot of tests such as XRD, XRF, and SEM in order to check if there was a change in structure of the treated soil with polyurethane compared with untreated soil and if the soil components changed or kept with no great change.

## 2.1. Physical properties

Physical properties of gypsum soil and polyurethane are necessary to understand the nature of this soil such as gypsum content, optimum water content, and soil classification according to (USCS), and D50 which known as the portions of particles with diameters smaller and larger than this value are 50%, Tables (1) and (2) show these properties.

Table (1): polyurethane properties. (After Al-Hadidi & Ibrahim, 2018)

Chemical composition	Isocyanate + polyol
Color	yellow
Viscosity ( <i>mPa.s</i> )	650-700
Specific gravity ( <i>gm/cm<sup>3</sup></i> )	1.18
Coagulation time (s)	30-1800
PH	6-7

Table (2): Soil physical properties. (After Al-Hadidi & Ibrahim, 2018).

Gypsum content (%) by weight	41
Maximum dry unit weight ( <i>kN/m<sup>3</sup></i> )	17
Optimum water content (%)	12
Specific gravity	2.56
$e_{max}$	0.92
$e_{min}$	0.50
$D_{10}$	0.17
$D_{30}$	0.38
$D_{60}$	1.1
$C_c$	0.69
$C_u$	6.11
Soil Classification According to (USCS)	Sand poorly graded soil

## 2.2. X-ray diffraction test

X-ray diffraction test which is a widely used analysis method to evaluate the crystallinity and structure of solid samples. In this technique, the phenomenon of crystal X-ray diffraction results from a scattering process in which the X-rays are scattered by the electrons of the atoms in the sample without changing the wavelength, (Harris and Norman White, 2008). This test

was used for two samples, treated gypsum soil with polyurethane chemical material and untreated gypsum soil.

### **2.3. X-ray Fluorescence test**

X-ray Fluorescence test which is a non-destructive analytical technique that bombards the sample with high-energy X-rays, causing the sample atoms to be ionized and to knock electrons out of their orbits. The ejected electrons are replaced by electrons falling from other orbits, and the incident electrons emit X-rays with a unique energy level depending on the orbit they came from and how far they fell. By measuring the energy and quantity of X-rays emitted, it is possible to determine the elements present in the sample. XRF can measure elements from magnesium to uranium (atomic numbers 12 to 92), (Wu, et., al., 2012).

For polymeric materials, samples are analysed as they are taken unless they are very thin, in which case they are stacked together. If the sample is a powder or granule, it can be packed into sample containers to obtain a stronger X-ray signal. Because XRF cannot measure light elements such as carbon, nitrogen, oxygen, fluorine or sodium (Wu, et., al., 2012).

This test was used for two samples, treated gypsum soil with polyurethane and untreated gypsum soil.

### **2.4. SEM Test**

SEM analysis is a powerful research tool that uses a focused electron package to produce highly detailed and complex images of the surface topography of a sample, (Collin and Courtois, 1988).

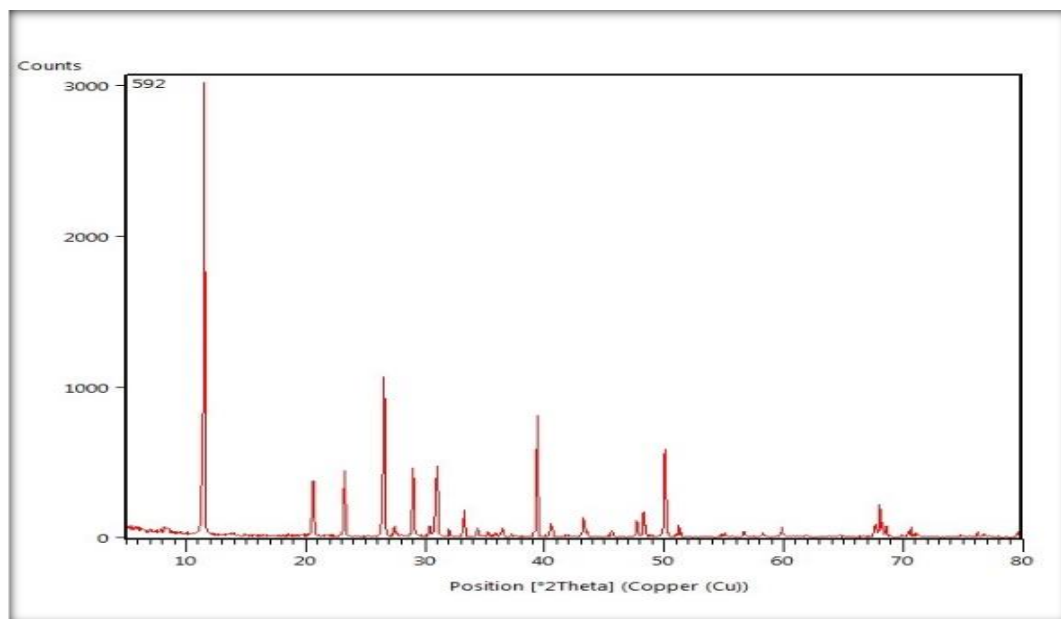
Scanning electron microscopy (SEM) magnifies a specific area of a sample using a package of focused, high-energy electrons. The sample is under vacuum to ensure that the electron package remains focused and does not interact with airborne particles. When the electron package hits the sample, it causes secondary electrons to be released from the detected sample to provide an image based on the surface topography, (Kobayashi and Ugai, 2012).

SEM analysis is more powerful than optical microscopy, as this powerful electron microscope has a magnification capacity of up to 500,000 times. This test was used for two samples, treated gypsum soil with polyurethane chemical material and untreated gypsum soil, (Kobayashi and Ugai, 2012).

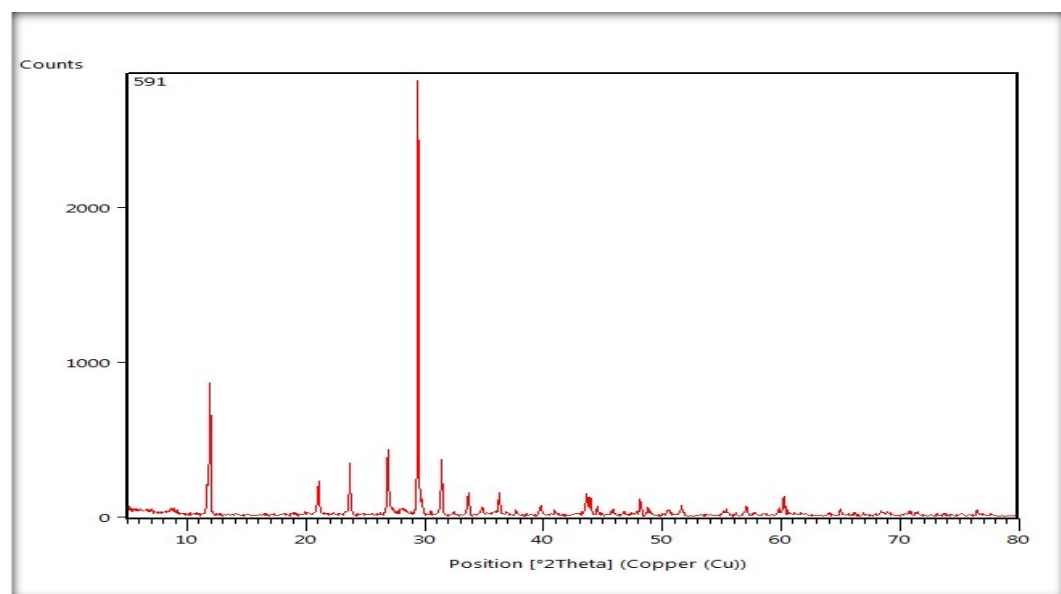
### 3. Results and Discussions

#### 3.1 XRD Test Results

The results of XRF test are shown in the Figure (1). gypsum content index in untreated soil is high and reached to (2 Theta = 11.589) where the index of gypsum (I %) at this angle equalled to 100%. This gypsum content is different for treated soil according to figure (1-B) which explain the low effect of gypsum particles at the same (2 Theta value), and this is the effect of polymer when adding to the gypsum soil.



A: Untreated soil

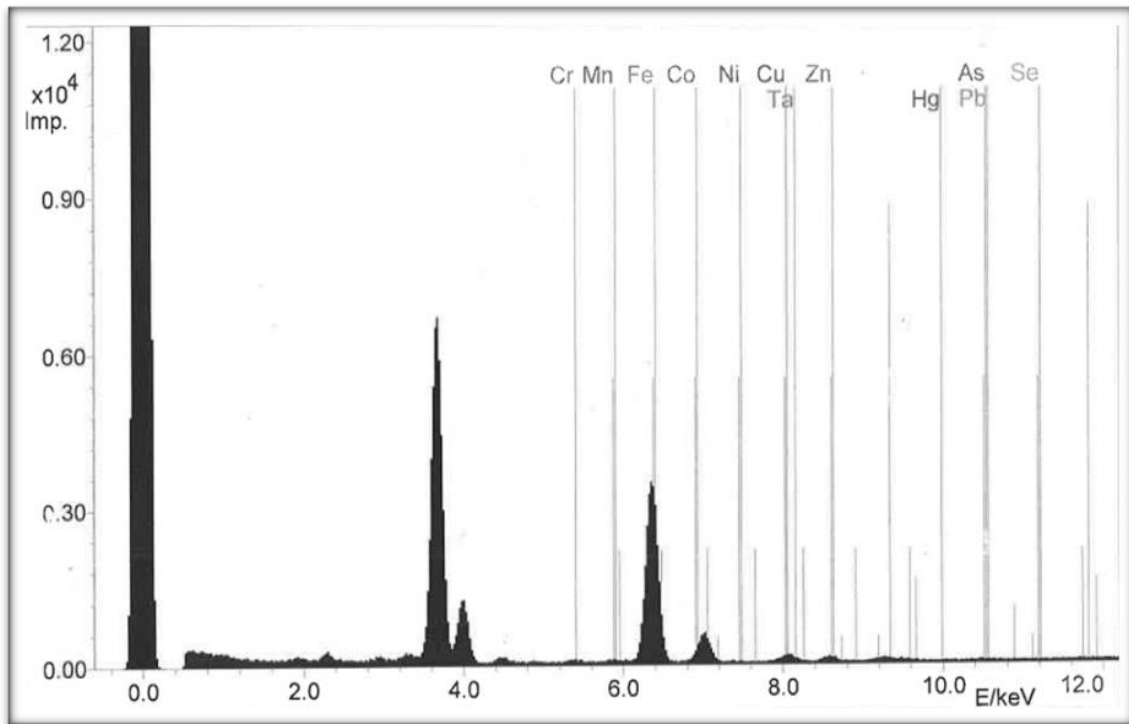


B: Treated soil

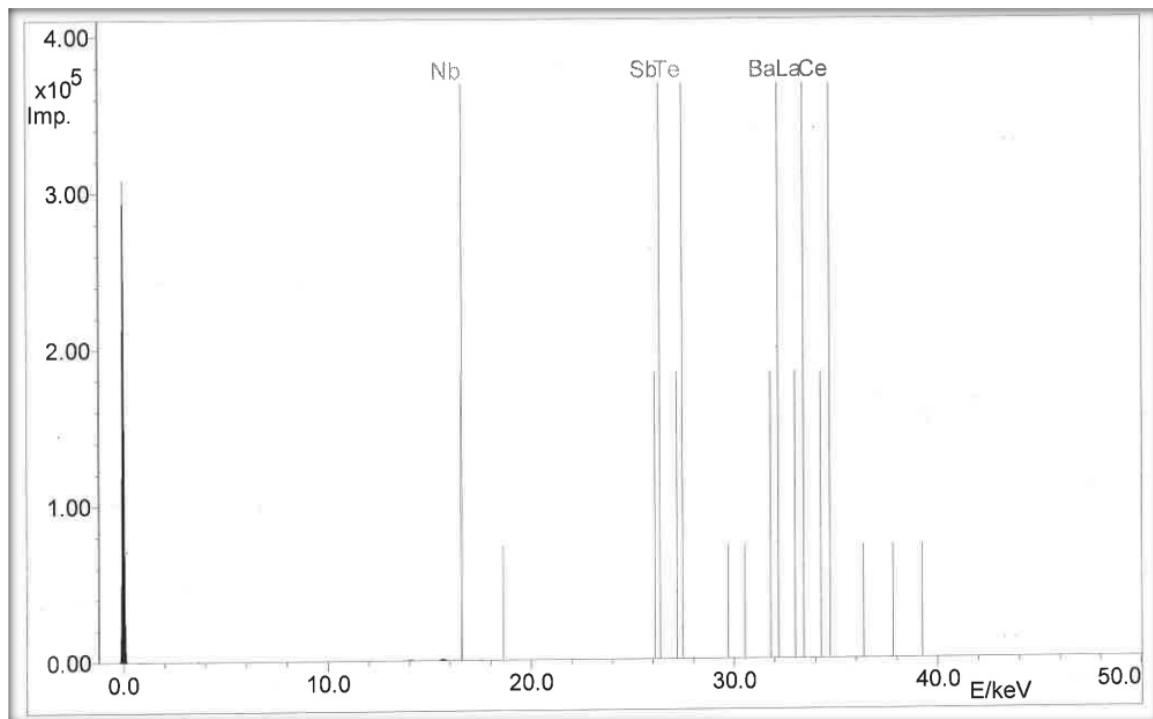
Figure (1): X-ray diffraction test for treated and untreated soil.

### 3.2 XRF Test Results

The results of XRF test are shown in the Figure (2) and (3).



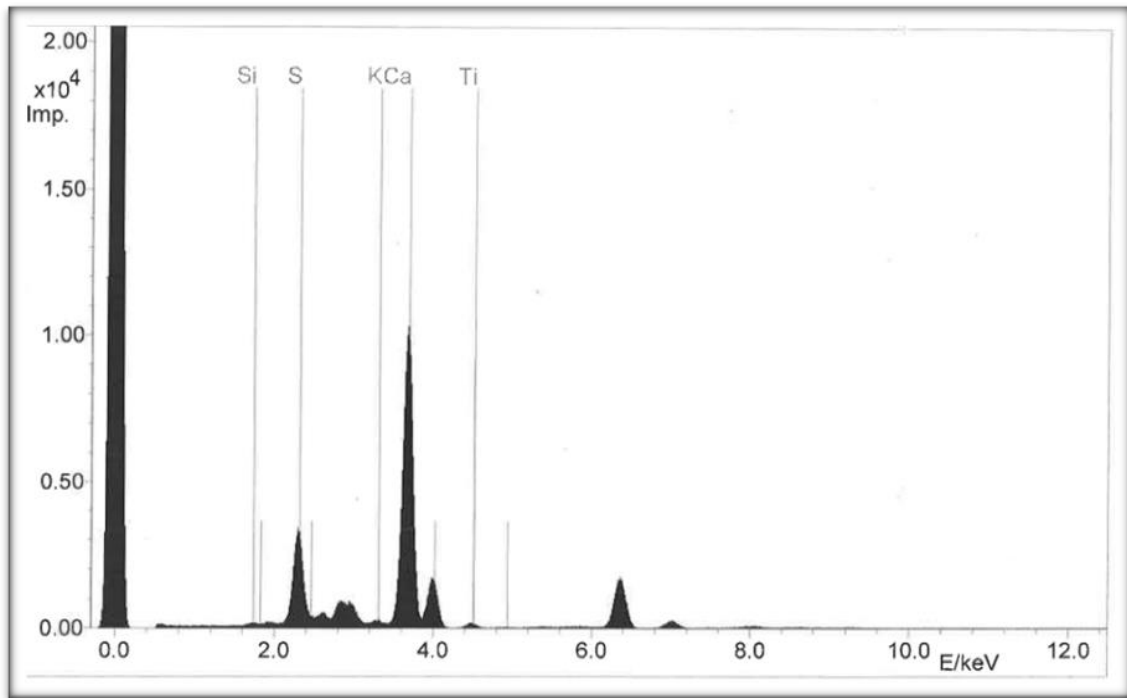
(A): Concentration of elements from Cr to Se.



(B): Concentration of elements from Nb to Ce.

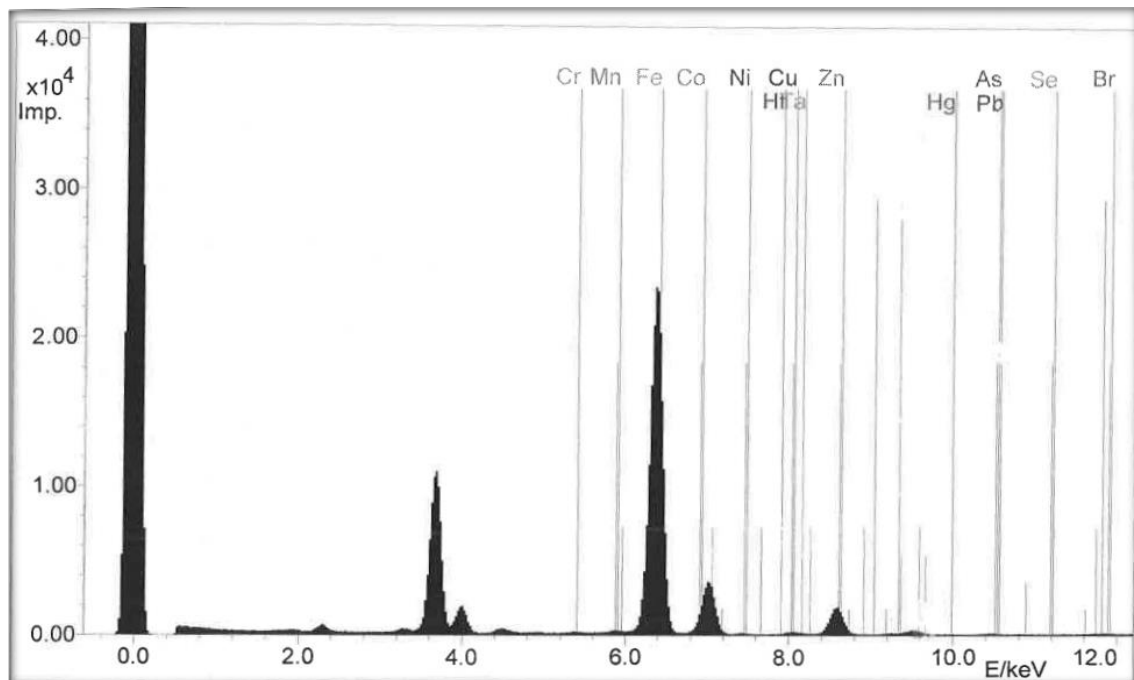
Figure (2): XRF Test for untreated soil.





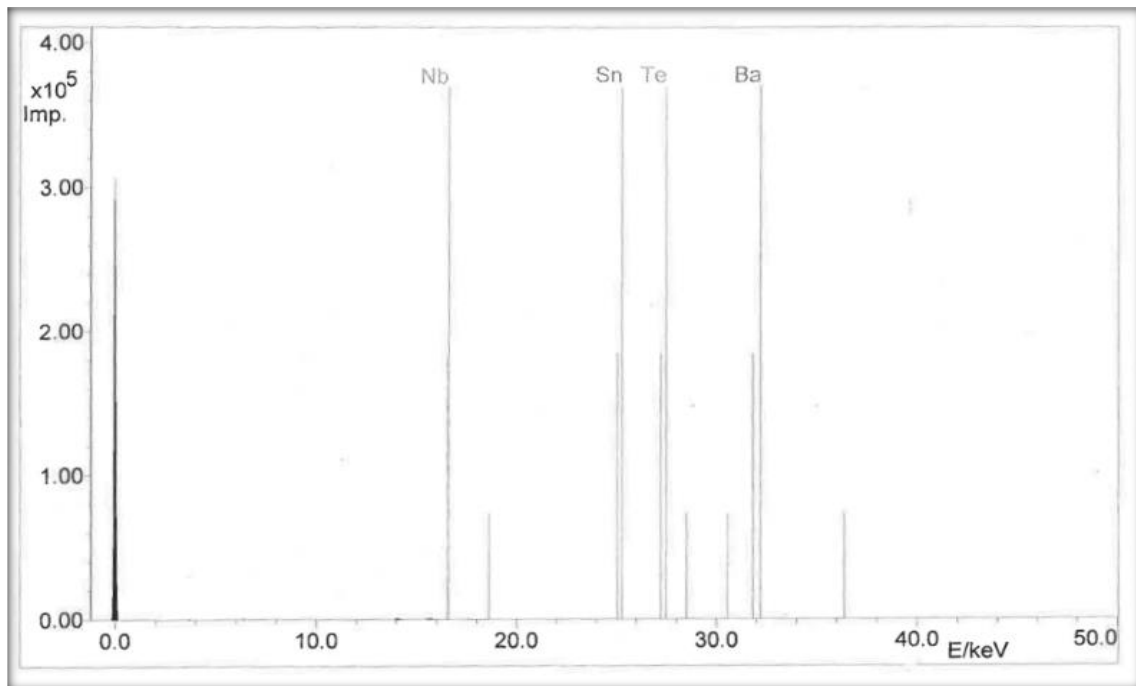
(C): Concentration of elements from Si to Ti.

Figure (2): XRF Test for untreated soil.(continued)

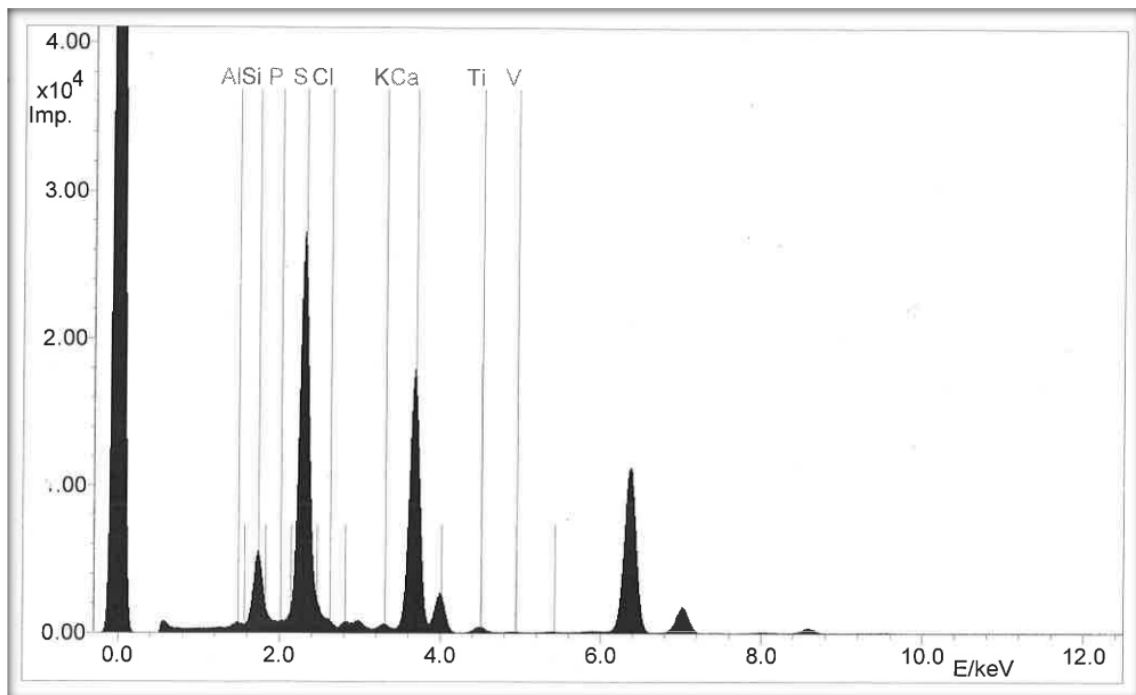


(A): Concentration of elements from Cr to Se.

Figure (3): XRF Test for treated soil.



(B): Concentration of elements from Nb to Ce.



(C): Concentration of elements from Si to Ti.

Figure (3): XRF Test for treated soil (Continued).

For made this study, three samples were stored from previous study as mention before, the first was the untreated soil before putting it in the channel and exposed to the water, the second was the untreated soil after exposed to the water in the channel and the third sample, was the treated

soil. The first sample could not be tested because it was damaged, so the XRF test not made for this sample. Table (3) shows the results of XRF test for the second and the third samples.

Table (3): Results of XRF test.

Element	Concentration (horizontal Axis)	
	Treated soil	Untreated soil after exposed to the water
Fe	2.335	0.3932
Ca	6.525	4.440
K	0.2352	0.1073
S	1.3	0.1855
Zn	0.049	0.00206

From Table (3), it has been observed that the concentration of the elements Fe, Ca, K, S, and Zn in the treated soil is greater than in the untreated soil and this was an index to the stability of the soil structure. Logically, the percentages of these elements in original soil (first sample) were the same or larger than in the treated sample.

The results of this test showed the proportions of elements in each sample, so the appearance of some elements in the treated soil in large proportions means that they were present in the original soil before treatment in an amount equal to or higher than these proportions. Since the examination of soil before treatment was not be capable due to its was damaged, so the percentage of these element could be considering in a proportion equal to what was present in the treated soil, based on the law of conservation of mass, which states that the mass can neither be created nor destroyed (Vermaa et.al.,2016).

The difference in the elements of components between the two samples is shown in Table (4).

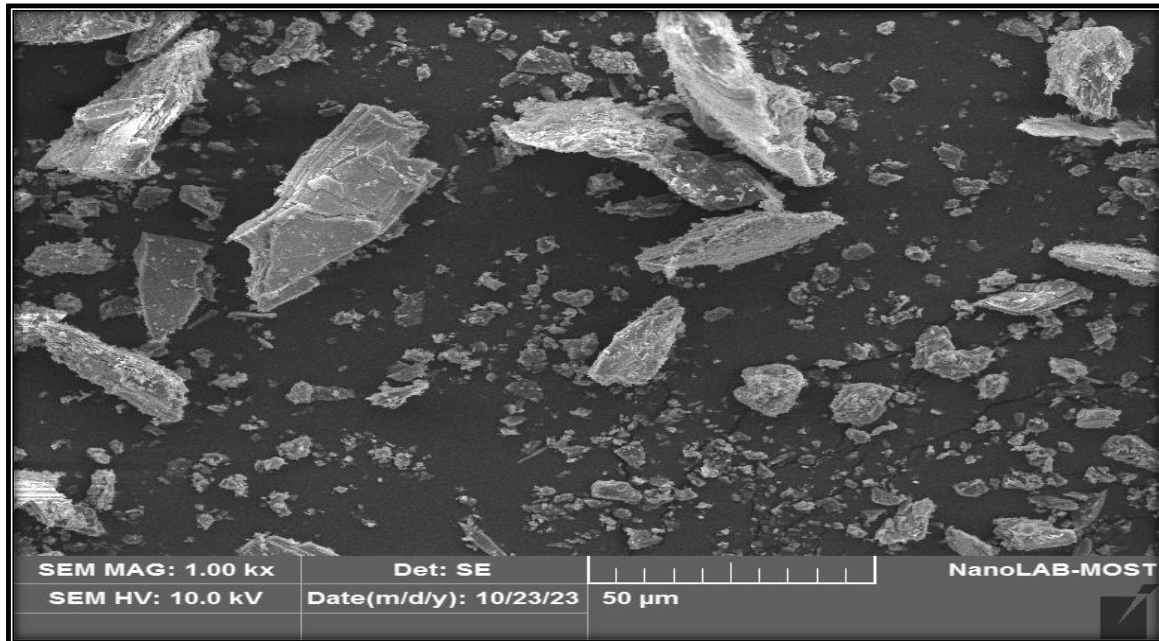
Table (4): Elements difference ratio.

Element	Difference ratio %
Fe	83
Ca	32
K	54
S	86
Zn	95

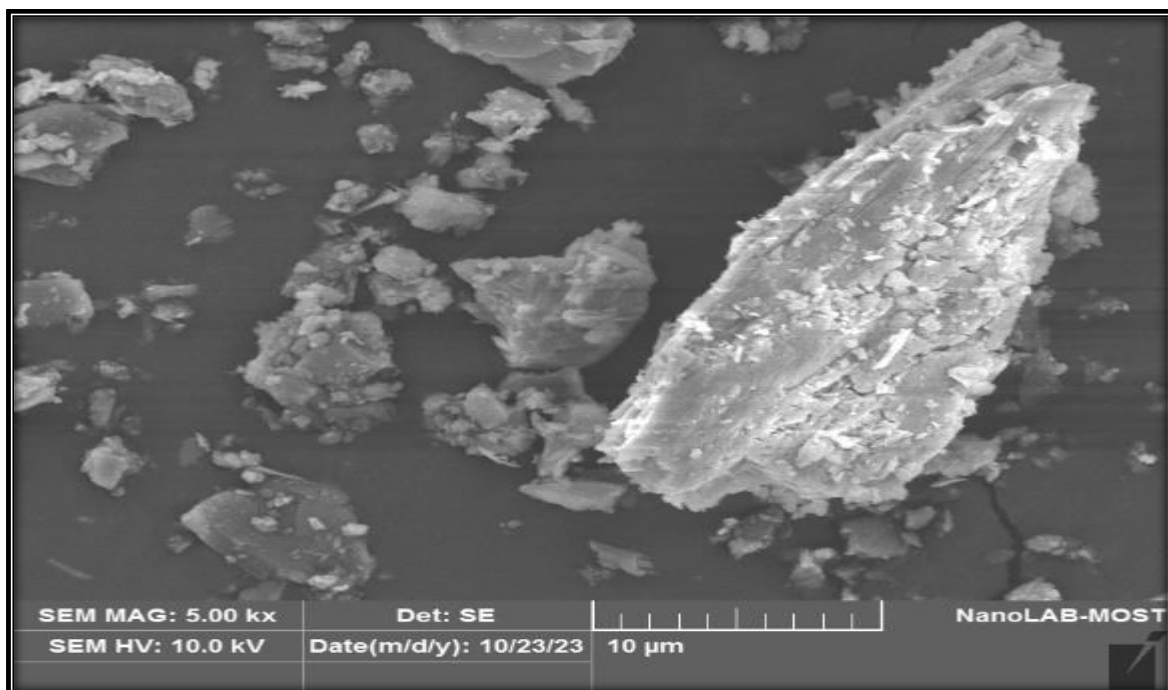
As shown in table (4), the difference in the elements of components between the treated and untreated sample was very high and that give evidence that the treatment done well and give very good results which mean that the addition of polyurethane maintains the elements of gypsum soil from the erosion.

### 3.3 SEM Test Results

The results of SEM for this study are shown in the Figures (4) and (5). The photos (A, B, C) in both figures refers to zoom equal to 1.00, 5.0 times the origin photo, and 35.0 times the origin photo, respectively.

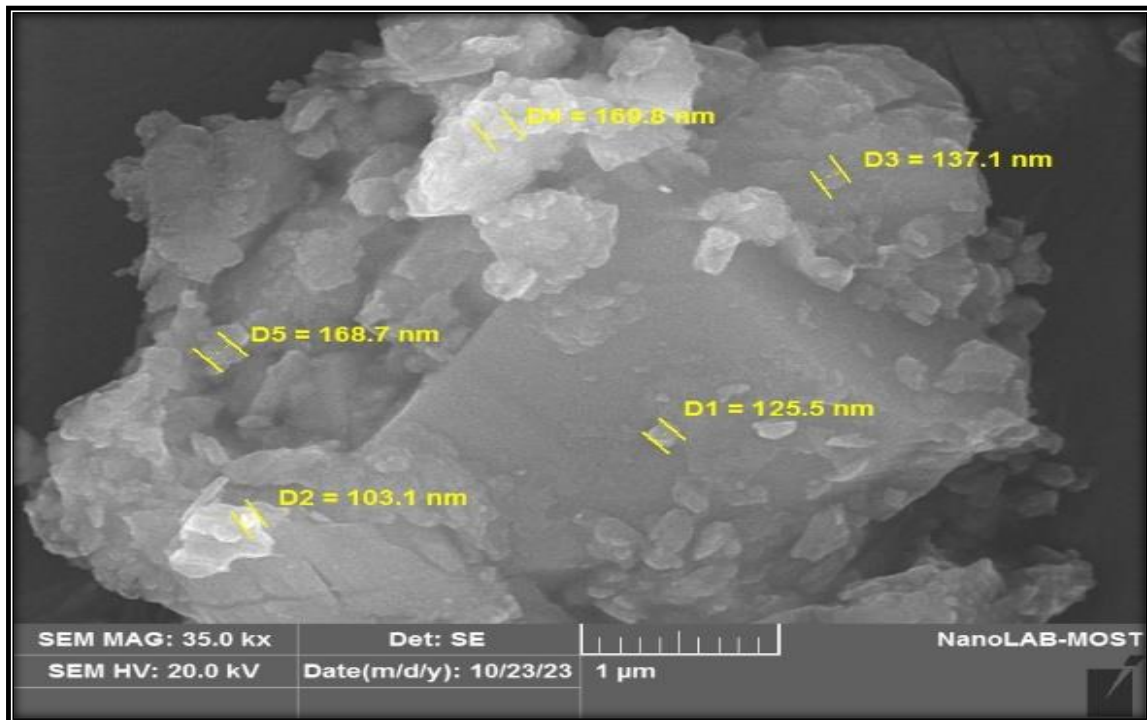


A: SEM at zoom 1.00



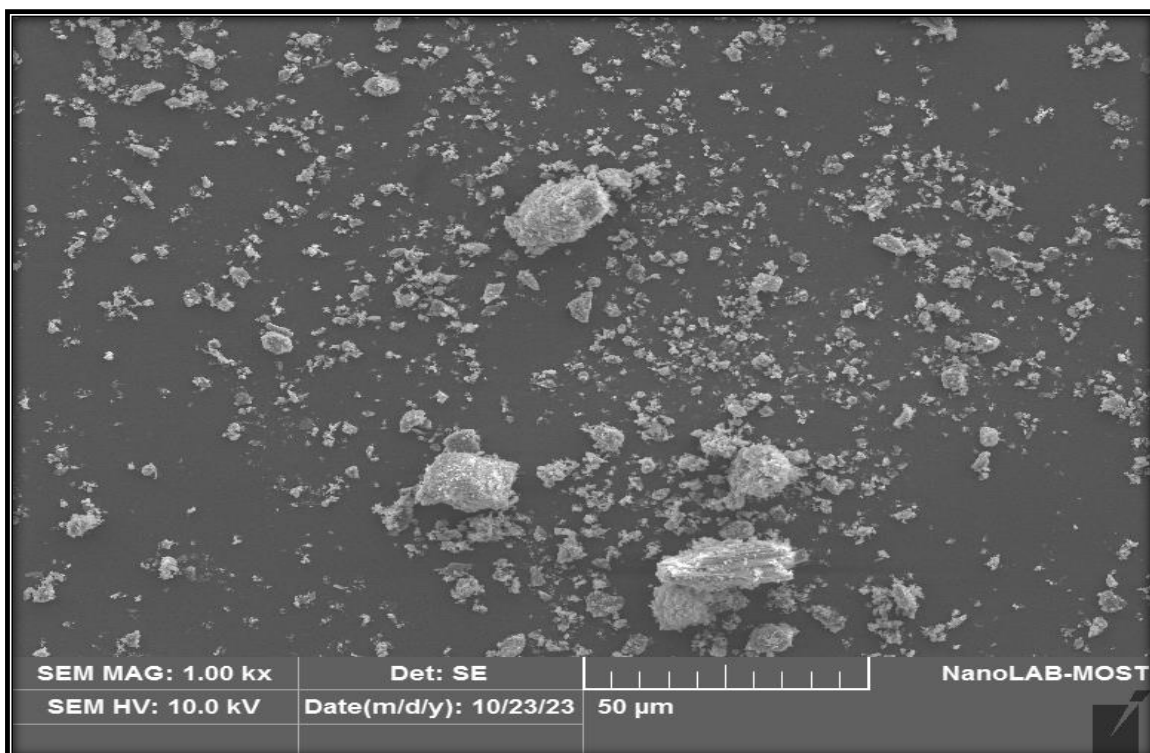
B: SEM at zoom 5.00 times the original photo.

Figure (4): SEM test for treated soil.



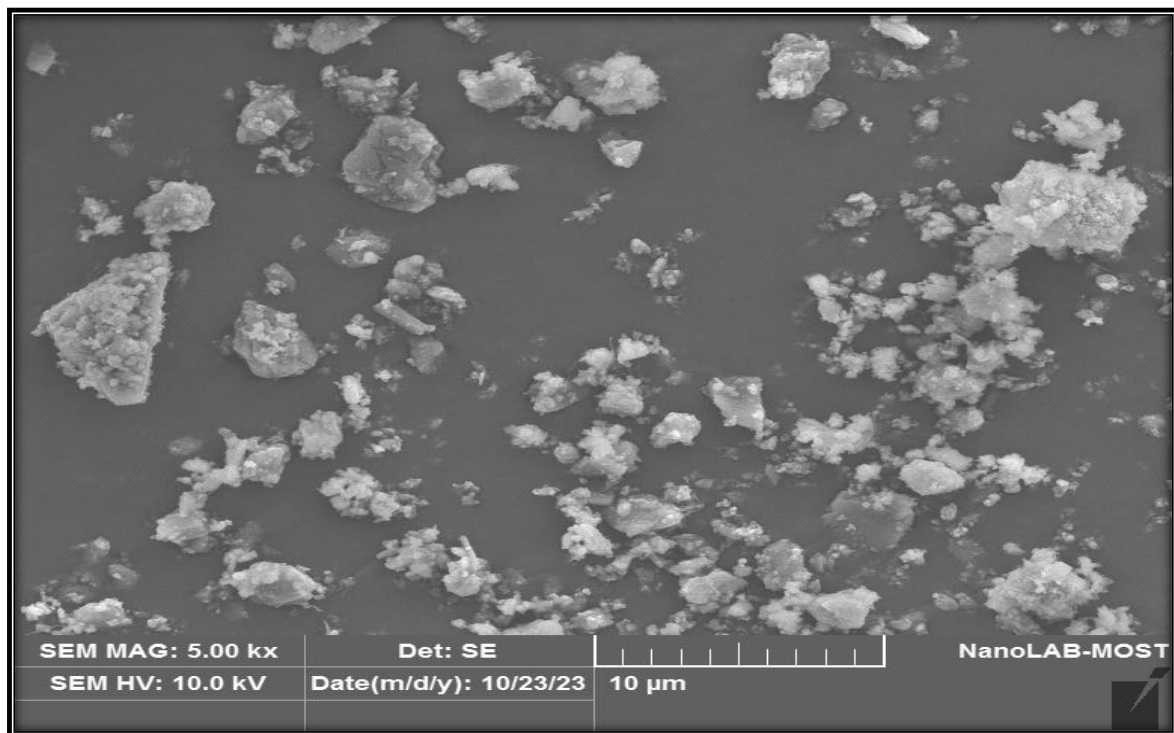
C: SEM at zoom 35.0 times the original photo.

Figure (4): SEM test for treated soil (continued).

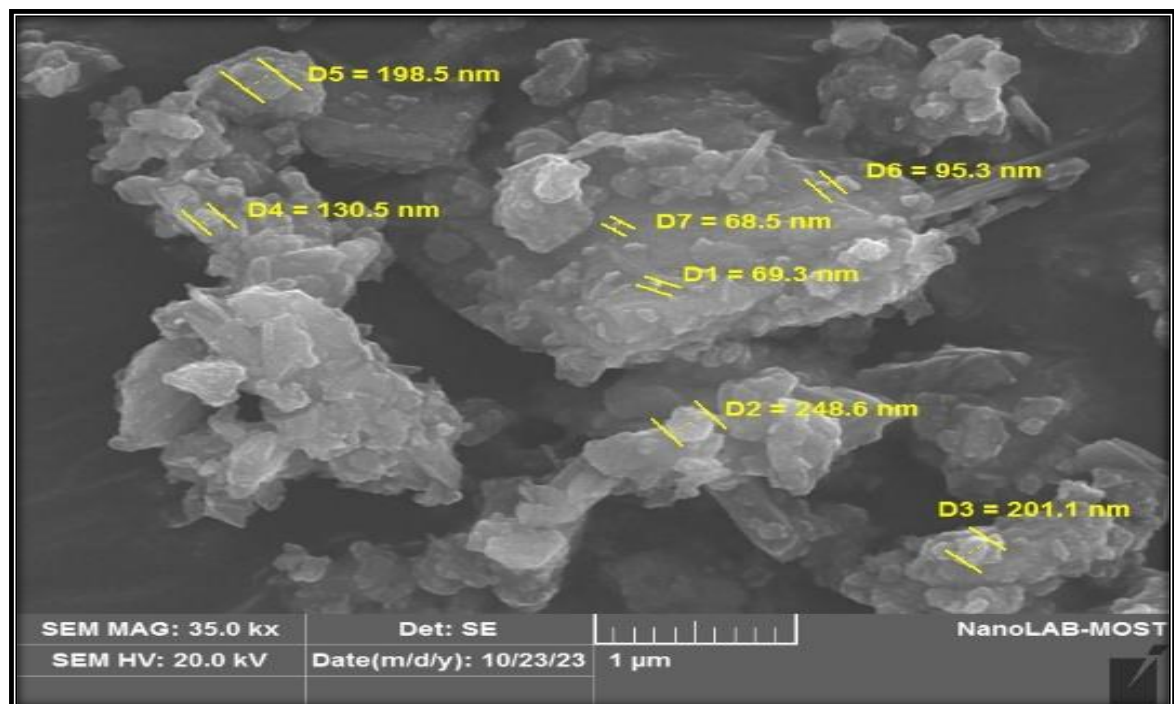


A: SEM at zoom 1.00

Figure (5): SEM test for untreated soil.



**B:** SEM at zoom 5.00 times the original photo.



**C:** SEM at zoom 35.0 times the original photo.

Figure (5): SEM test for untreated soil (continued).

From Figures (4 and 5), figure (A) at zoom 1.00 Kx for both samples, (B) for both samples at zoom 5.00 Kx, and (C) for both samples at zoom 35.0 Kx. It was noticed from this figures

that the treated soil is a cohesive granular soil. After being treated with polymer, these granules are very cohesive, and this leads to reducing the pores between the soil particles due to the formation of the chemical that filled these voids, while in untreated soil from Figure (5 A) was not cohesive and the pores were large.

### **3.4 Comparison with other studies**

The study of (Al-Barrak and Rowell, 2006) proved that after 1 hour extract, the gypsum soil of 40% gypsum has intensity 3000 for 2 theta 11.589 which the same value of this study for gypsum soil before treated. The study (Al-Barrak and Rowell, 2006) didn't used polyurethane as an additive material, so the comparison After treatment is not valid.

## **4. Conclusions**

- 1) After 4 years of creating the samples of treated and untreated soil, polyurethane was highly efficient material for treatment of gypsum soil from the effect of erosion.
- 2) The effect of this polymer maintains the element such as Fe, Ca, K, S, and Zn in treated soil.
- 3) Cohesive granular increased in the treated soil with polyurethane.

## References

- Ahmed, A., Kobayashi, M., and Ugai, K. ,2012, *Performance assessment of clay soil stabilized with recycled gypsum based on SEM and XRD*. In 2nd International Conference on Transportation Geotechnics (ICTG) International Society of Soil Mechanics and Geotechnical Engineering (ISSMGE).
- Ahmed, B. A., Al-Hadidi, M. T., and Mohammed, D. W. , 2020, *Improvement of the Gypseous soil properties by using Copolymer and Styrene-butadiene rubber*. In IOP Conference Series: Materials Science and Engineering (Vol. 737, No. 1, p. 012084). IOP Publishing.
- Al-Barrak, K., and Rowell, D. L. ,2006, *The solubility of gypsum in calcareous soils*. Geoderma, 136(3-4), 830-837.
- Al-Hadidi, M. T., and Ibrahim, A. G. ,2018, *Improvement of Gypsum Soil by Using Polyurethane to Reduce Erosion and Solubility of Irrigation Canals*. International Journal of Engineering and Technology, 7(4.20), 372-376.
- Al-Jassim, W. S., and Al-Hadidi, M. T. ,2020, *Impact of rationing on the properties of cement-treated gypsum canals*. Association of Arab Universities Journal of Engineering Sciences, 27(3), 15-30.
- Al-Nedawi, N. M. and Al-Hadidi, M. T. ,2020, *Finite element analysis of seepage for Hemrin earth dam using Geo-Studio software*. Diyala Journal of Engineering Sciences, 66-76.
- Buck, B. J., and Van Hoesen, J. G. ,2002, *Snowball morphology and SEM analysis of pedogenic gypsum, southern New Mexico, USA*. Journal of Arid Environments, 51(4), 469-487.
- Collin, J. P., & Courtois, B. (1988). Device testing and SEM testing tools. In *Testing and Diagnosis of VLSI and ULSI* (pp. 469-506). Dordrecht: Springer Netherlands.
- Danoosh, A. H., and Al-Hadidi, M. T. ,2022, *Numerical simulation to the effect of applying rationing system on the stability of the Earth canal: Birmana canal in Iraq as a case study*. Journal of the Mechanical Behavior of Materials, 31(1), 729-738.
- Ebailila, M., Kinuthia, J., and Oti, J. ,2022, *Role of gypsum content on the long-term performance of lime-stabilised soil*. Materials, 15(15), 5099.
- Harris, W. I. L. L. I. E., and Norman White, G. ,2008, *X-ray diffraction techniques for soil mineral identification*. Methods of soil analysis part 5—Mineralogical methods, 5, 81-115.
- Ibrahim, A. G., Al-Hadidi, M. T., Hussien, H. H., and Neamah, N. K. ,2022, *A Review Study on Gypseous Soils Stabilized with Different Additives in Iraq*. Journal of Water Resources and Geosciences, 1(1), 125-141.



- Jha, A. K., and Sivapullaiah, P. V. ,2014, *Role of gypsum on microstructure and strength of soil*. Environmental Geotechnics, 3(2), 78-89.
- Kordlaghari, M. P., and Rowell, D. L. ,2006, *The role of gypsum in the reactions of phosphate with soils*. Geoderma, 132(1-2), 105-115.
- Toma, N. M., and Al-Hadidi, M. T. ,2022, *The effect of soaking and wetting on the properties of the gypsum soil treated with polyurethane*. Association of Arab Universities Journal of Engineering Sciences, 29(2), 01-08.
- Vermaa, D., Singhb, V., and Kishwanc, J. ,2016, *Material conservation–an approach to prevention of pollution*. Octa Journal of Environmental Research, 4(4), 312-318.
- Weindorf, D. C., Zhu, Y., Ferrell, R., Rolong, N., Barnett, T., Allen, B. L., and Hudnall, W. ,2009, *Evaluation of portable X-ray fluorescence for gypsum quantification in soils*. Soil science, 174(10), 556-562.
- Wu, C. M., Tsai, H. T., Yang, K. H., and Wen, J. C. ,2012, *How reliable is X-ray fluorescence (XRF) measurement for different metals in soil contamination*. Environmental Forensics, 13(2), 110-121.
- Yilmaz, I., and Civelekoglu, B. ,2009, *Gypsum: an additive for stabilization of swelling clay soils*. Applied clay science, 44(1-2), 166-172.