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Comparison study between inherited and biogenic calcium carbonate formation on the surface roots of Eucalyptus trees using X-ray technique and field observations

Hashim H. Kareem¹ Kahraman H. Habeeb² Layth S. S. Al-Shihmani²

1: University of Misan , College of basic education , Dept. Of Science

2: University of Wasit , College of Agriculture , Dept. Of soil sciences & water resources.

<https://orcid.org/0000-0002-2720-7381>: Orcid

hashim.hanin@uomisan.edu.iq

Abstract:

This study aims to explain the mechanism of formation of calcite and its to shed light on the synthetic carbonate crystallization on the surface of roots due to this mechanism is poorly understood and highly controversial. It is accepted that calcite formed originally from parent rocks and not in soils, The origin of calcite in calcareous soil, in Aldiwaniya city, Iraq was investigated, by selecting two soil samples, one from the contact zone with root surface and the other sample from bulk soil and out of the root system, X-ray diffraction showed that the calcite has two types, first one is well crystallized and ordered, with only minor deviations in lattice unit cell parameters relative to typical calcite. The clay-sized calcite (active calcite) is enriched in A and C-horizons relative to biogenic calcite and this mineral exist in larger size separates, and it's clearly appears in the shape of white crystals around eucalyptus root surfaces as shown in graphs. The results of X-ray showed that the appearance of smectite mineral which was diagnosed by the peak 14.21° in air dry magnesium-saturated treatment which expanded to 16.28° when saturated with ethylene glycol, where clarity of peak was increased, Also, the results showed that the appearance of peak 14.21° in magnesium saturation air dried, as a shoulder on the peak of the mica (10.14°). In general, calcite minerals have characteristic x-ray diffraction peaks at the following angles: 29.5 , 31.7 , 34.5 , 35.7 , 47.3 , 56.4 , and 63.4 degrees.

Key words: calcareous soil, inherited calcium carbonate, eucalyptus roots, in situ formation, x-ray technique, clay minerals

Introduction:

All plants and trees account on soil, but trees and associated organisms also have a critical role in the genesis or transformation of soil (Taylor et al. 2009). Soil is produced from the degradation of bedrocks, which have many characteristics in the light of the source of the parent material, topography, climate and vegetation. Soil Carbon commonly comes from plants, the interactions between

plant roots, microorganisms and soil in the rhizosphere are very important issue in the study of the mineralogy of the soil. These interactions demands contact among many organisms associated in

rhizospheric activities. Soils are the common reservoir for carbon, store three times as much Carbon as plant biomass above the earth's surface, and considered as a storage for both organic and mineral Carbon, as soil organic matter and pedogenic carbonates. The generation within the soil solution of dissolved Carbon came from organisms and determination of its precipitation as calcite. However, it expressed the potential value of this case for synthesized-middle CO₂ trapping within soils (Schlesinger, 1995; Van Hees et al., 2003).

The ability of trees such as eucalyptus to produce organic acids as root exudates is crucial, and releases (70 mol/kg) of secreted Carbon, equivalent to the plant's weight. This is still an order of magnitude less than measured Carbon production (Moulton et al., 2000), as a single source of Carbon. Hence, there is apparently no deficient of available soluble Carbon, as HCO₃ in solution, and so the genesis of pedogenic carbonates will be depended on the concentration of Calcium in solution. Which came from chemical weathering, to the primarily of silicate minerals (natural plagioclase feldspars and pyroxenes, (Zamanian et al., 2016).

Accordingly, this research is aimed to shed light on the precipitation of calcium carbonate on the surfaces of eucalyptus trees root and how to distinguish between it and pedogenic or inherited calcium carbonate by using X-ray techniques.

Materials and methods:

This research was conducted in Eucalyptus orchard founded on calcareous soil in the city of Diwanya, 80 km south of Baghdad, Iraq, longitude 243. 4 59 32N latitude 684 . 39 47 44E, In order to investigate the origin and nature of in situ calcium carbonate formation on the surface of the eucalyptus roots, after digging and preparing soil pedon in contact with the root system of Eucalyptus tree which called exploited soil pedon, Two types of soil samples were collected from this pedon which include accumulated calcium carbonate samples on the surface of plant roots, and other sample were taken from the bulk of the second pedon which called non-exploited soil pedon, besides soil samples from each horizon were taken and kept in plastic bags for chemical and physical and mineralogical analysis according to Page et al., (1982) were particle size distribution was determined by using pipette method, particle density by Picnometer, bulk density by using paraffin wax methods, porosity calculated from the mathematical relationship between particle and bulk density, pH and Ec are determined in the 1:1 soil:water extract, cation exchange capacity (CEC) by using ammonium acetate saturation method, calcium carbonate by calcimeter method, gypsum by using acetone method, organic matter by using wet digestion by dichromate method according to Walkley-Black and potassium and sodium by using flame photometer, calcium and magnesium ions by titration with Na₂EDTA, Chloride by titration with silver nitrate, carbonate and bicarbonate by titration with sulphuric acid, sulphate by using turbidity method after precipitation with barium chloride. The grains and powder of collected calcium carbonate were also kept in plastic bags for mineralogical analysis by x-ray diffraction techniques (Jackson, 1958).

Results and discussion:

Results in Table (1) show some physical properties of studied soil pedons which indicate that the exploited soil is silty clay loam with fractions percentage ranged between (182.40-171.70), (299.45-392.62) and (389.1-427.89) g.kg⁻¹ for clay, silt and sand respectively and loam for or non-exploited soil with fractions percentage ranged between (192.24-335.70), (332.43-408.19) and (325.46-403.57) g.kg⁻¹ for clay, silt and sand respectively, in the other hand bulk density ranged between (1.10-1.32)

M.gm.m⁻³ and particle density (3.36-2.03) M.gm.m⁻³ while non-exploited bulk density ranged between (1.10-1.32) M.gm.m⁻³ and particle density (3.36-2.03) M.gm.m⁻³, the results of physical properties suggest that the studied soils characterize by dominance of fine fractions which reflect on the values of bulk and particle density.

Table (2) showed some chemical properties of studied soils where the values of pH ranged between (7.41-7.82) and (7.21-7.45) for exploited and non-exploited soils respectively and EC (Electrical Conductivity) values ranged between (3.79-4.38) and (3.00-4.01) Ds.m⁻¹ for exploited and non-exploited soils respectively. Also, CEC values ranged between (21.85-26.08),(18.29-21.04) Cmolc.kg⁻¹ for exploited and non-exploited soils respectively. The values of Gypsum ranged between (23-45),(29-42) gm.kg⁻¹ for exploited and non-exploited soils respectively, organic matter values ranged between(11.66-14.6),(17.24-21.05) gm.kg⁻¹ for exploited and non-exploited soils respectively while the values of calcium carbonate ranged between (295-338),(236-286) gm.kg⁻¹ for exploited and non-exploited soils respectively .

According to the results of Table (2) the studied soils are moderately alkaline and slightly saline soils . The other chemical properties namely (CEC, OM, Gypsum and calcium carbonates are within the range of arid and semi arid levels which characterized by high base saturation ratio due to the contribution of calcium carbonate in the increment of calcium ion in soil solution which causes high alkalinity in the solution , this conditions consider as an ideal media for the precipitation of calcium carbonate crystals between soil fractions (Hillel , 2005).

Table (1) some physical properties of studied soils at different soil depths.

location	horizon	Depth	clay	Silt	sand	Texture	Bulk density	Particle density
		cm		g.kg ⁻¹				M.gm.m ⁻³
Diwanya Exploited	A _p	0-45	182.4	352.6	426.6	Silty clay loam	1.31	3.35
	C _{k1}	45-70	171.7	354.2	427.9	Silty clay loam	1.32	3.36
	C _{k2}	70-90	262.8	299.5	389.1	Silty clay loam	1.12	2.03
	C _{k3}	90-130	247.4	392.6	345.7	Silty clay loam	1.13	2.10
	C _{k4}	+130	234.5	302.9	399.9	Silty clay loam	1.10	2.10
Diwanya Non-Exploited	A	0-25	192.2	408.2	403.6	Silty clay loam	1.20	2.20
	C _{k1}	25-45	335.7	346.1	325.5	loam	1.11	2.30
	C _{k2}	45-57	329.6	332.4	338.9	loam	1.12	2.40
	C _{k3}	57-85	321.8	338.2	340.4	loam	1.19	2.40
	C _{k4}	+85	320.2	339.1	395.7	loam	1.13	2.40

Table (2) some chemical properties of studied soils at different soil depths.

location	horizon	Depth	CaCO ₃	O.M	Gypsum	EC	pH	CEC
		cm	g.kg ⁻¹	g.kg ⁻¹	g.kg ⁻¹	Ds.m ⁻¹		Cmolc.kg ⁻¹
Diwanya Exploited	A _p	0-45	332	14.6	45	4.14	7.82	26.08
	C _{k1}	45-70	338	12.2	34	4.33	7.79	21.85
	C _{k2}	70-90	335	11.95	41	4.38	7.39	22.15
	C _{k3}	90-130	295	12.65	37	3.95	7.46	23.21
	C _{k4}	+130	324	11.66	23	3.79	7.41	22.34
Diwanya Non-Exploited	A	0-25	286	21.05	36	4.01	7.21	21.04
	C _{k1}	25-45	256	19.63	42	3.27	7.45	19.62
	C _{k2}	45-57	265	18.12	38	3.23	7.38	18.23
	C _{k3}	57-85	236	17.24	32	3.00	7.33	18.87
	C _{k4}	+85	248	18.02	29	3.09	7.41	18.29

Mineral composition of studied soils:

In order to illustrate the mineralogical composition in the studied soil environment, a series of x-ray diffraction tests were done, including bulk soil samples and clay samples as further as the calcite powder taken from the surface of the root of eucalyptus trees , The results of the X-ray studied soils showed that the exploited soil in Figure (1) the appearance of Smectite mineral which was diagnosed by the peak at 14.21 A° in air dry magnesium-saturated treatment which expanded to 16.28 A° when saturated with ethylene glycol, where clarity of peak was increased, on the other hand, the appearance of the peak 14.21 A° in magnesium saturation treatment, as a shoulder on the peak of the mica mineral (10.14 A°) in the 550 C° heating treatment confirms the presence of chlorite mineral knowing that the chlorite mineral in this soil is swelling chlorite Many studies explained the main source of this mineral in arid and semi-arid soils was the substance of origin of this soil. The presence of the chlorite mineral confirmed the appearance of its second peak at 7.09 A° , which remained constant in all treatments.

Dixon et al. (1990) also stated that the non-real chlorite mineral (swelling) maintains its base distance of 14.14 A° within air dry magnesium saturation and ethylene glycol saturation, and then 550 C° heating treatment lead to its disappearance (break it down) or its appearance as a shoulder on the diffraction on the peak (10 A°) of mica mineral. It was shared in this many studies carried out on Iraqi soil. The smectite mineral diagnosed in this soil through peak at 16.28 A° In the case of magnesium saturation and ethylene glycol treatment, the percentage of this mineral in this soil, which is due to the weathering process and is in its initial stages through the process of agricultural exploitation, was observed to decrease despite the availability of conditions for its composition of calcium ions and magnesium in addition to the degree of interaction. Ismail (1969, 1970) has explained the possibility of this mineral being a product of biotite weathering in arid and semi-arid conditions. Increasing the percentage of this mineral by increasing the salinity of the soil is also due to increasing the concentration of calcium and magnesium in exchange sites.

X-ray curves in Figure (1) showed the presence of d-spacing in the range ($16.73 - 14.21\text{ A}^\circ$) in the sample saturated with magnesium and air-dried where between the mineral properties of the interstratified smectite- chlorite mineral ,This indicates that there are shifts of smectite minerals towards chlorite as a result of the precipitation of the brucite layer $[\text{Mg}_3(\text{OH})_6]$ between the interlayers of (2:1) minerals(Yousefifard et al, 2015 .)

X-ray curves in Figure (1) of exploited soil showed the presence of d-spacing in the range ($10.00\text{ A}^\circ - 10.62\text{ A}^\circ$) in the magnesium-saturated sample and air dry treatment, where it showed the mineral properties of the interstratified mineral (palicorskite-illite) The existence of this mineral is yet another indication of the prevalence of weathering and transformation of the mica minerals towards the palicorskite mineral as a result of agricultural exploitation, continuous irrigation and organic residues that reinforces the transformation process.

The presence of mica in this soil was demonstrated by X-ray diffraction curves through the d-spacing at 10.10 A° which remained constant in all treatments with increased intensity in potassium saturation and 550 C° heating treatments and the presence of this mineral means that the reservoir of this soil from potassium element is increased.

The results also showed the existence of the peaks (3.33 and 4.75) \AA° combinations and their survival in all treatments, confirming the existence of weatherproof calcite and quartz minerals respectively.

The results of X-ray examinations of Figure (2) showed the same mineralogical composition of the exploited soil with less peak intensity, especially expansive 2:1 (Montmorellonite) minerals, which appeared in the clearly and high intensity in exploited soil, which support the theory of the transformation of Mica minerals into expansive 2:1 minerals. The results also showed a lower intensity of Mica minerals in exploited soils compared to non-exploited soils that reinforce the theory of transformation of chlorite into expansive 2:1 minerals (Ivanić et al,2020; Zafar et al,2022).



Figure (1) calcite precipitation on the root surface of eucalyptus in studied area

Calcite mineralogical properties:

Calcite is a type of calcium carbonate mineral that can form in a variety of ways. One way that calcite can form is on the surface of plant roots. This can happen when calcium ions in the soil solution are taken up by the roots and then deposited on the surface of the root in the form of calcite crystals (Casas et al ,2020) . This process can be facilitated by the presence of certain types of bacteria on the root surface, which can help to catalyze the precipitation of the calcite crystals (Lambers et al ,2009). Additionally, the presence of organic molecules on the root surface can also help to promote the formation of calcite crystal as shown in Figure (1) for studied soil.

Calcite is a type of calcium carbonate mineral that can be found in soil. The morphological features of calcite in soil are determined by the way in which the calcite crystals have formed and grown. Some common morphological features of calcite in soil include the size and shape of the crystals, the degree of crystal perfection, and the overall arrangement of the crystals within the soil (Huguet et al, 2021).

The values of the X-ray diffraction peaks of calcite minerals depend on a number of factors, including the specific type of calcite mineral, the crystal structure of the calcite, and the conditions under which the calcite was formed. In general, calcite minerals have characteristic diffraction peaks at the following: 29.5, 31.7, 34.5, 35.7, 47.3, 56.4, and 63.4 degrees. However, these values can vary somewhat depending on the specific sample being studied (Stevenson et al, 2005; Gocke et al , 2011).

Biogenic calcite is calcite that is in the soil profile, and it is not directly associated with the activity of living organism. formed through the action of living organisms, such as bacteria or algae. This type of calcite is typically found on the surface of plant roots, and it can form through a variety of processes. In contrast, inherited calcite is calcite that is present in the soil as a result of previous geological processes, such as the weathering of rocks or the deposition of calcite-rich sediment. Inherited calcite is typically found in deeper horizons, (Arnott and Pautard, 1970 ; Gunal and Ransom, 2006 , Jones et al , 2003).

Calcite has a hexagonal crystal structure, which means that its crystals have a six-sided shape. The specific shape of calcite crystals can vary depending on the conditions under which the calcite formed and the amount of impurities that were present in the original solution. In general, calcite crystals tend to be hexagonal in shape, but they can also have a variety of other shapes, including rhombohedral, scalenohedral, and prismatic (Khalaf et al , 2000 ; Toyoda,etal , 2021)

The results of X-ray diffraction showed that the existence of calcite peaks in broad and sharp shape and this peak revealed the difference in its intensity between the sample from the surface of eucalyptus root and the sample in bulk soil were in the root surface the value of peak were 3.1° and in the bulk soil were 3.2 and 3.6° , This variation in the intensity and shape of the peak is due to the difference in the conditions of formation which reflect on the degree of crystallinity and the crystal dimensions , In more specific details this variation by the effect of organisms and its exudates such as organic acids and some organic molecules that change soil solution conditions (Phillips et al , 1987,Majchrzyk et al , 2021; Manning , 2018) .

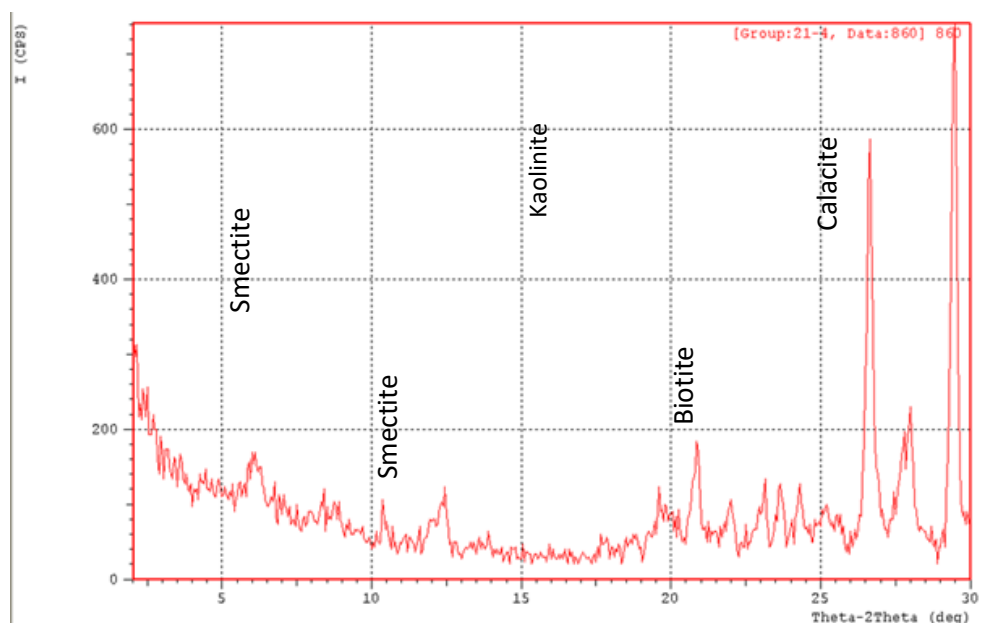


figure (2) X-ray diffraction of clay fraction for studied soil sample

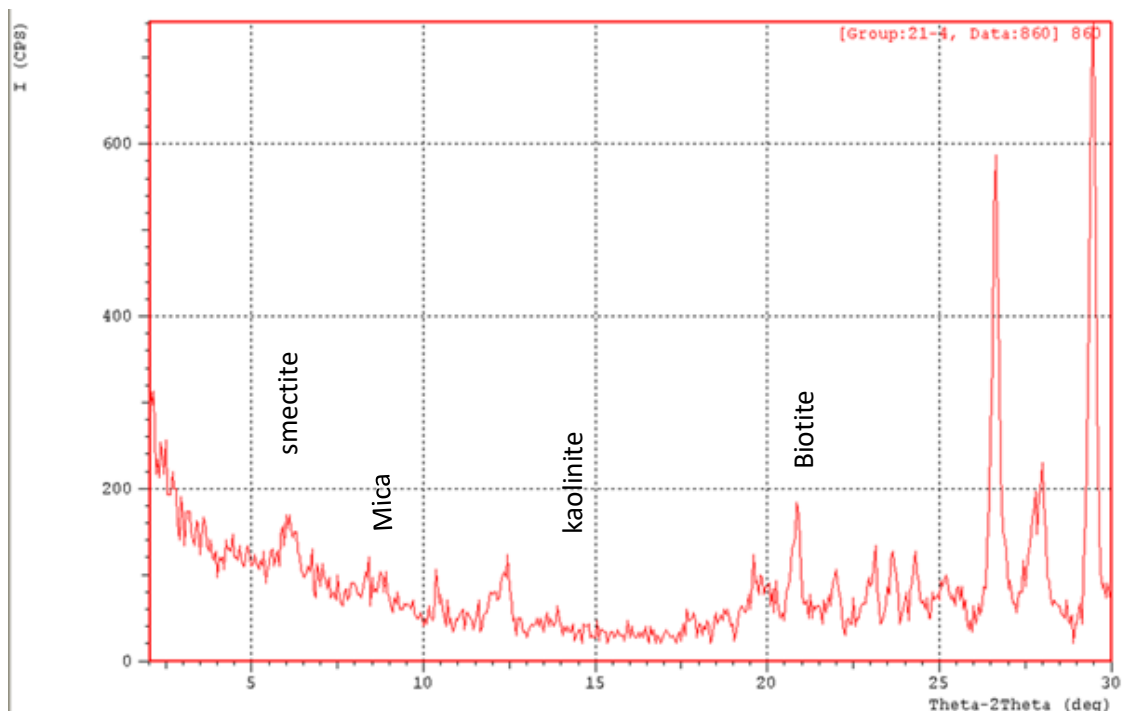


Figure (3) X-ray powder diffraction of calcite powder from the bulk soil sample

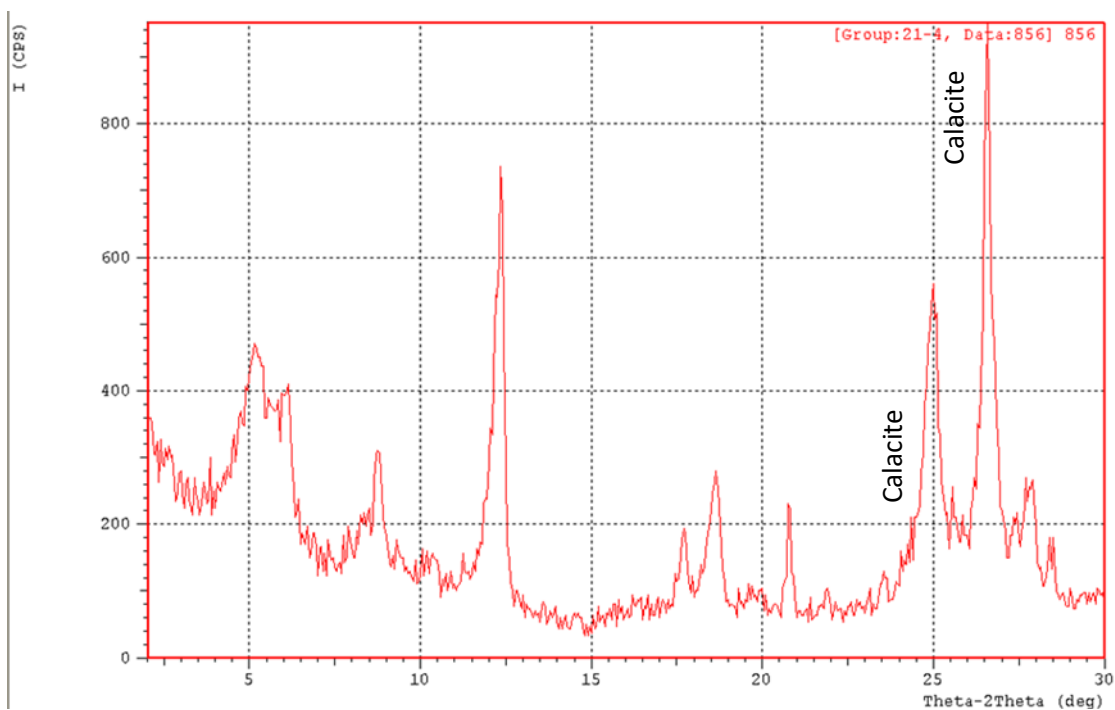


Figure (4) X-ray powder diffraction of calcite powder on the root surface of eucalyptus.

Conclusions:

Calcium carbonate in arid and semi-arid conditions may appear in more than one form, where most of calcium carbonate is inherited from parent rocks and minerals, in geological processes. nevertheless, the other form is biogenic calcium carbonate which can synthesize by the activity of different soil exudates on the root surfaces, however, eucalyptus root accumulate calcium carbonate in a form of white crystal. X-ray diffraction techniques can easily be used to distinguish between two types of calcite according to peak intensity values, shapes and d-spacing.

Roots could have an important role in calcite genesis including excreting organic compounds exudates including organic acids. Various plant species such as trees, shrubs, grasses and weeds have different roots density, depths and adaptation to higher pH due to high calcite content. Various plant types contribute calcite crystallization mechanisms at different calcium carbonate content and the specific concentration of some alkaline cations such as (Ca) are still under investigation and research.

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دراسة مقارنة بين كاربونات الكالسيوم الموروثة و المتكوتة بيولوجيا على سطوح جذور أشجار اليوكالبتوز باستخدام تقنية الاشعة السينية الحادة والمظاهر الحقلية

هاشم حنين كريم¹ كهرمان حسين حبيب² ليث سليم سلمان الشحماني²

1: جامعة ميسان /كلية التربية الأساسية/ قسم العلوم العامة

2: جامعة واسط /كلية الزراعة/ قسم التربة والموارد المائية

<https://orcid.org/0000-0002-2720-7381>: Orcid

hashim.hanin@uomisan.edu.iq

المستخلص

هدفت هذه الدراسة إلى توضيح آلية تكوين الكالسييت وتسليط الضوء على بلورة الكربونات المتكونة بيوكيميائيا على اسطح الجذور لكون هذه الآلية غير واضحة نوعا ما ومثيرة للتساؤلات. وعموماً أن الكالسييت تشكل في الأصل من الصخور الأم وليس في التربة وهو أصل الكالسييت في التربة الكلسية في مدينة الديوانية، تم اختيار عينتين من التربة، إحداها من منطقة التلاصق مع سطح الجذر والأخرى من التربة السائبة ومن نظام الجذر، أظهر حيود الأشعة السينية أن الكالسييت له نوعان، الأول يتبلور جيداً ويرتبط مع انحرافات طفيفة فقط في معلمات وحدة الخلايا الشبكية بالنسبة إلى الكالسييت النموذجي. يتم إثراء الكالسييت بحجم الطين (الكالسييت النشط) في آفاق A و C بالنسبة إلى الكالسييت الحيوي، ويوجد هذا المعدن بحجم أكبر، ويظهر بوضوح على شكل بلورات بيضاء حول أسطح جذر الأوكالبتوس كما هو موضح في الرسوم البيانية. أظهرت نتائج الأشعة السينية أن ظهور المعدن الصغير الذي تم تشخيصه بالذروة 14.21 درجة في العلاج المشبع بالمغنيسيوم الجاف في الهواء والذي توسع إلى 16.28 درجة عند تشبعه بالإيثيلين جليكول، كما أظهرت النتائج أن ظهور الذروة 14.21 درجة في الهواء المشبع بالمغنيسيوم قد جف، ككتف على قمة الميكا (10.14 درجة أ). بشكل عام، معادن الكالسييت لديها قمم حيود الأشعة السينية المميزة في الزوايا التالية: 29.5، 31.7، 34.5، 35.7، 47.3، 56.4، و 63.4 درجة.

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