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Intercation Between Some Micro-Elements And Humic Substances in Some Forest Soils Northern Iraq

ABSTRACT

Four pedons were selected for different soils in the forest cover, two in each of Mankeesh and Atrush areas in Dohuk governorate, under pine and oak trees, in order to study the distribution of the main groups of humic compounds (humic and fulvic acids) and their association with iron and manganese ions. The results showed that the variation in the nature of the forest cover and the speed of decomposition of its remains led to a difference in the distribution of the content of organic matter, the ratios of Humic acid / fulvic acid (FA / HA) and the amount of humic substances produced in each soil, regarding the optical density of the extracted humic compounds, which is expressed as E4/E6, this ratio depends on the chemical composition of these Humic compounds. It is noted from the results that this ratio between E4: E6 ranged between (0.43 - 1.79), low values reflect the presence of aromatic Humic compounds with relatively low molecular weights, while high values indicate the presence of aliphatic humic compounds, the study also showed a difference in the values of iron and manganese associated with humic and fulvic acid as organic complexes, which is consistent with the difference in the degree of decomposition of forest tree residues and plant residues, and in turn is reflected on the quantity and type of humic compounds resulting from the decomposition. The Atrush soil under pine forests excelled in its composition of chelated iron with fulvic acid, Humic acid and manganese chelated with fulvic acid, while the Atrush soil under oak forests excelled in the composition of chelated manganese with humic acid on the rest of the studied soils.

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INTRODUCTION

Soil organic matter (SOM) has an important role in influencing the chemistry of transitional elements in soil through their complex interactions with various soil components, including elements and the formation of metal complexes (Han et al., 2019), and that the association of the active organic groups found in humic compounds with other elements arises from the formation of complexes, and that the chelates of these elements possess the property of dissolving and moving in the soil solution and this reduces the activity and effectiveness of these elements, so it can be said that humic compounds work to bind iron and manganese with them strongly forming complexes and their organic chelates (Tan, 2011; Chirwa and Yerokun, 2012), as the plant residues go through different decomposition stages, resulting in the formation of new complex humic compounds (humus) as a result of the humification process formed as a result of biochemical reactions and chemical transformations that occur to plant residues in the presence of microorganisms that contribute to the formation of these compounds, which represents the last stage of their decomposition. However, this humus in the soil is subjected to disintegration, breakdown, and new re-formation by the action of microbes with the loss of part of the organic carbon in the formation of new microbial tissues, meaning there is a permanent compensation for carbon from another source (Tan, 2003 ; Al-Khafagi, 2021). Humic compounds tend to differ in their chemical

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composition depending on the different origin and type of these compounds that are affected by plant residues, whether they are tree residues (leaves, branches, fruits) or plant residues of various kinds, according to (Banach-Szott et al., 2019), the chemical composition is affected or subject for : different soils, changing compounds over time, different techniques and separation of these compounds, therefore, the mechanism by which these materials are formed was not clear to many researchers, but there are some hypotheses or assumptions that differ in the origin or source of these materials, but they agree that these compounds are the source of the remains of plants of different types (Hedges, 1988; Weber, 2020). Therefore, several attempts were made to clarify the importance of the degree of humification depending on the optical density and absorption values of the humic extracts using a chromatography spectrophotometer. This is achieved by measuring the optical density of the extract of humic compounds using a Spectrophotometer at wavelengths 465 and 665 nanometers (nm), expressed in the color ratio, which represents E4 / E6 shown in the following equation (Kononova, 1966):

$$E4 / E6 = \log \text{ absorbance at } 465 / \log \text{ absorbance } 665 \quad\ldots\ldots(1)$$

The spectral absorption of humic and fulvic acid is characterized in terms of the logarithm of absorption as a linear function versus the wavelength of visible light with a slope used as an indicator to distinguish between humic compounds, fulvic acids have a greater intensity compared to humic acids (Kumada, 1985 ; Tan, 2000). The high values of the chromaticity ratio at 7-8 or more indicate the presence of more severe curves related to fulvic acid, and the ratio between 3-5 indicates the presence of less severe curves related to humic acid, while the humification index is in terms of the change in the logarithm of absorbance (EΔ). It is calculated according to the following equation:

$$\Delta \log E = \log E_{465} - \log E_{665} \quad\ldots\ldots(2)$$

The value of $\Delta \log E$ can change from 1: 1 or more for compost materials to 0.6 or less in humic acids (Tan, 2003; Even et al., 2014). Humic compounds are characterized by the instability of their molecular weight as a result of the substitution and exchange that occurs between hydrogen (H^+) organic groups and other elements, which makes these compounds are broken down into humic acids depending on the principle of difference in solubility at different pH conditions and their behavior towards solvents, whether acids, bases and water. Accordingly, the carbon ratio of C_{HA} / C_{FA} is estimated through the ratio of HA / FA based on (Martin et al., 1998; Mladkova et al., 2006). These acids play a role in determining the quality of organic matter depending on the way the carbon atoms are arranged in the above acids, either in the form of a chain consisting of what is known as aliphatic chains, or in the form of a network or ring consisting of what is known as aromatic nets, or through the ratio of humic acid / Fulvic acid in addition to the absorption property of the humic extract at wavelengths of 465 and 665 nanometers (nm) represented by the ratio between them E4 / E6 as mentioned previously (Stevenson, 1994; Mladkova et al., 2006). These acids also affect the regulatory capacity of the soil and the degree of its interaction because humic acid and fulvic acid have the ability to separate H^+ from organic groups, often the carboxyl group (-COOH) and the phenol group (-OH), which results in the emergence of different negative charges due mainly to the cation exchange capacity (Weber et al., 2018). The aim of this research is to identify the chemical behavior of micro elements (Fe and Mn) and characterize humic compounds in some forest soils in northern Iraq.

MATERIALS AND METHODS

Selection of study sites:

After reconnaissance, field observation, and review of maps and satellite visuals, two areas were chosen within the Dohuk governorate in northern Iraq (the Mankeesh area with two sites (pine and oak), and the Atrush area also with two sites (pine and oak) as indicated in Figure (1).
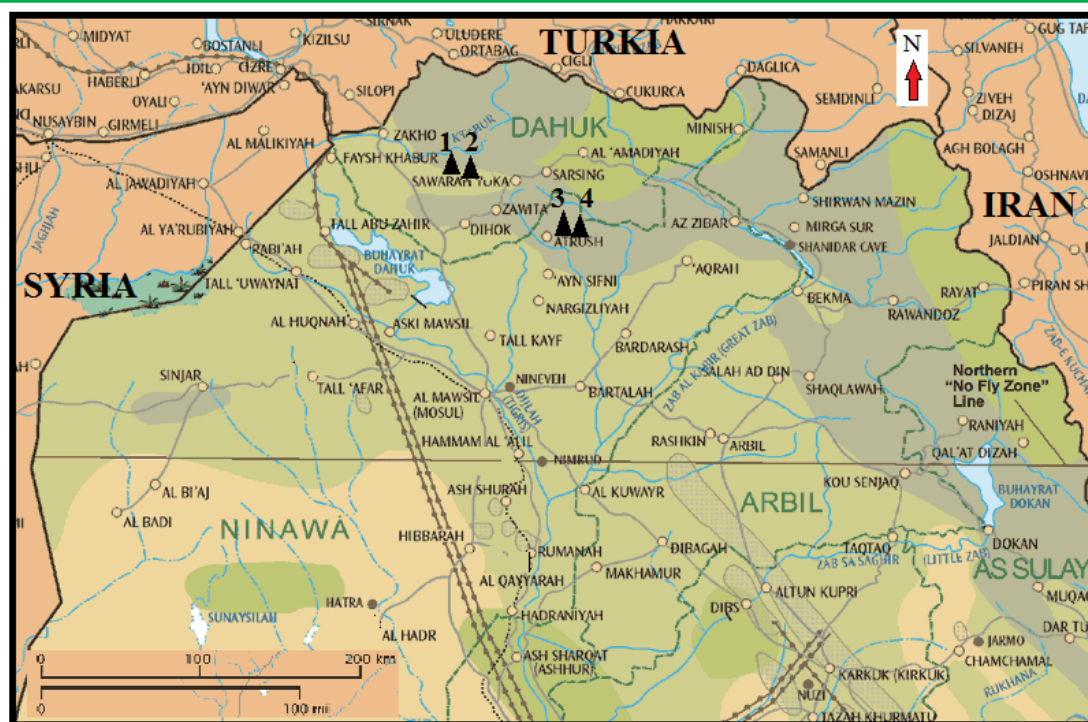
1: Mangeesh-Pine   2: Mangeesh-Oak   3: Atrush-Pine   4: Atrush-Oak

Figure (1): A map of Dohuk showing the study sites

Collection of soil samples

One pedon was dug in each of the study sites with a depth of 100 cm and a width of 80 cm, taking into account that the pedon faced the sun, the pedon was divided into three horizons, soil samples were collected from each horizon and for all pedons, and samples were collected from bottom to top with the numbering of each sample, soil samples were dried pneumatically, then transferred to the laboratory, crushed and sieved with a sieve with a diameter of 2 mm, so that they were ready to some physical and chemical analyzes. The soil particles distribution was estimated, then the soil texture was determined according to the method of (Gee and Bauder 1986), the electrical conductivity (Ec) and (pH) were measured with the soil extract (1:1), as stated in (Page et al., 1982), and calcium carbonate was estimated according to what was mentioned by (Ryan et al., 1996), the organic matter (OM) was estimated according to what was stated (Tandon, 1999) and as in Table (1).

Extraction and separation of humic compounds from soil

The humic compounds were extracted by taking 10 gm of soil sample and adding to it 100 ml of dilute hydrochloric acid (0.1 N) to remove calcium carbonate and using centrifugation to separate the soil from the leachate. Then the soil was washed twice with distilled water and centrifuged to get rid of the leachate. After that, added to the washed soil 100 ml of a mixture of sodium hydroxide (0.1M) and sodium pyrophosphate (0.1M) with pH 12, then the above mixture is shaken for two hours and left for 24 hours, after which the soil is separated from the filtrate in a centrifuge (3000 rpm for 5 minutes) that represents the extract of humic acids (humic and fulvic acid). Humic acid is separated from fulvic acid by acidifying the humic extract by adding gradual amounts of HCl (6 N) acid, as humic acid (HA) coagulates, and then this humic acid is dissolved by NaOH according to the mentioned method (Page et al., 1982), while for the non-coagulated fraction, it represents flavic acid (FA) (figure 2), after that, the organic carbon is estimated in both humic and fulvic acid by oxidation method, Then iron and manganese were measured in both humic acid and fulvic acid by an atomic absorption.

Table (1): Some properties of the studied soils in different locations

| Location	Depth	pH	EC	CaCO₃	Texture
	cm		dS.m⁻¹	gm.kg⁻¹	
Mangeesh - Pine	0 - 20	7.97	0.555	360	Silt Clay Loam
	20 - 50	8.11	0.605	410	Clay Loam
	50 - 90	7.76	0.740	425	Clay Loam
Atrush- Pine	0 - 20	7.39	0.745	395	Clay Loam
	20 - 40	7.42	0.650	450	Loam
	40 - 55	7.47	0.500	465	Silty Clay
Mangeesh - Oak	0 - 20	7.86	0.965	230	Clay Loam
	20- 55	8.45	0.785	425	Sandy Clay Loam
	55 - 90	8.07	0.790	425	Sandy Loam
Atrush- Oak	0 - 20	7.46	0.765	195	Clay Loam
	20 - 45	7.30	0.760	380	Clay
	45 - 60	7.38	0.790	295	Clay Loam
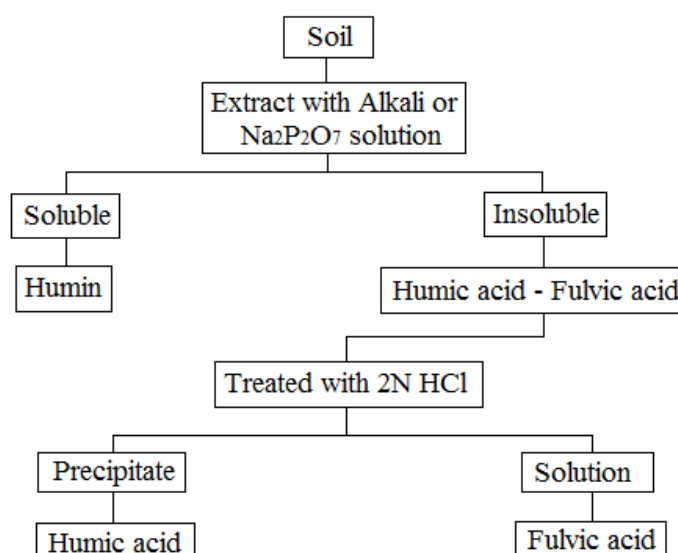
Figure (2): The process of fractionation the organic matter according to the (Schnitzer, 1982)

Determination of the optical density of humic compounds

The humic extract that was extracted from the soil before separating the humic acids from each other was measured by a spectrophotometer at light wavelengths of 465 and 665 μm expressed as E4 / E6 as described by it (Kononova, 1966), where taken 2 ml of the humic extract and placed in a glass test tube, and added it 4 ml of evidence (sulfuric acid - potassium dichromate), which was prepared from dissolving 1.27 g of potassium dichromate in 200 ml of concentrated sulfuric acid, then shake the test tube and leave it for 30 minutes for the purpose of reading it by the spectrophotometer at the wavelengths mentioned above, and use sucrose as a chemical containing carbon to prepare the original standard solution, then, standard solutions of different concentrations are prepared from it and read by the spectrophotometer to draw the standard curve, from which E4 and E6 are calculated (Stevenson, 1994).

RESULTS AND DISCUSSION

1- Soil Organic matter (SOM)

Humic compounds form part of the organic matter that has reached the stage of decomposition and complete decomposition during the humification process, the importance of organic matter is that it has a significant impact on improving the chemical, physical and biological properties of the soil and thus increasing its productivity (Margesin and Schinner, 2005). Table (2)

shows the variation in the organic matter content in the study soils, which ranged from 3.44 gm.kg^{-1} in the third depth of Mankeesh soil - oak to 32.68 gm.kg^{-1} in the first depth of Atrush soil - oak. This variation in the organic matter content of the study soil is due to the difference in the type of forest vegetation, temperature, humidity, activity and effectiveness of microorganisms in the soil and their role in the process of decomposition of tree and plant residues, and thus increasing the organic matter of the soil (Al-Dhahi, 2009). The difference in the rate of decomposition of the remains of trees and plants had an effect on the difference in the content of organic matter for the study soil, and it was noted that there was a fluctuation in the amount of organic matter between the depths of one pedon and between different pedons, which is associated with the degrees of decomposition to which trees and plants are exposed and then transformed into colloids organic represented by humic compounds that facilitate their movement between different soil depths (Al-Khafagi, 2021).

2- Humic compounds:

2-1 Humic acid

Humic acid is characterized as being one of the humic compounds with an unstable and variable molecular weight because it contains active organic groups that ionize to separate hydrogen from the organic group radical and be replaced by another element, and so on, for this reason, its molecular weight is variable. This behavior is reflected on the distribution of humic acid in the studied soils, as it is noted from Table (2) that there is a discrepancy in the values of humic acid between soil sites, as its values ranged between (0.50 - 8.16) gm.kg^{-1} soil, and that the highest value was found in the first depth of the Mankeesh soil - oak, and the lowest value was in the third depth of the Mankeesh soil - pine. Strobel (2001) showed that the difference in forest and plant covers caused a discrepancy in the amount of organic matter between soil sites, where the accumulation of plant residues (tree residues) under forests is more than agricultural soils, which is accompanied by fluctuations in the process of decomposition under these two covers, as for the vertical distribution of humic acid in the soil, it was in line with the distribution of organic matter, as its content is high in the surface depths and decreases with increasing soil depth. This is reflected in the distribution and quantity of humic acids because they are affected by the nature and quantity of organic matter and factors that help decomposition (Certini et al., 2000).

2-2 Fulvic acid

Fulvic acid is one of the most important components of the breakdown products and decomposition of organic matter, as it is a more effective component due to the increase in the active organic groups compared to humic acid because this acid arranges organic groups in the form of an aromatic ring that is more present in this acid (Ali and Mindari, 2016). Table (2) showed that the amount of fulvic acid in the study soils was less compared with humic acid, which ranged from 0.24 gm.kg^{-1} soil in the third depth of the Atrush soil (oak and pine) to 10.20 gm.kg^{-1}soil in the first depth of the Atrush soil - Pine. This difference could be related to the degree of decomposition of plant residues and its effect on the main components of the organic matter and its relationship to the activity of microorganisms in the soil (Hees et al., 1999). In this regard, Hongve (1999) explained that the superiority of forest trees, whether they are deciduous or evergreen, is accompanied by a discrepancy in the amount of nutrients because the leaves of trees differ in terms of being broad or bushy and therefore the chemical composition of these leaves varies by virtue of the environmental conditions ready to decompose and disintegrate these leaves over time (Schroth et al., 2007).

Table (2): The total content of the organic matter and its fractions and the optical density of the pedons of the studied soils

Depth	Fractionation of Organic Matter (gm.kg⁻¹)					E4 : E6 Ratio
cm	SOM	Humic Acid	Fulvic Acid	Humin	HA : FA Ratio	
Mangeesh –Pine						
0 – 20	15.16	2.05	3.08	10.03	0.66	1.79
20 – 50	13.78	2.05	5.05	6.68	0.40	1.28
50 – 90	13.09	0.50	0.24	12.35	2.08	1.03
Atrush-Pine						
0 – 20	28.20	3.05	10.20	14.95	0.29	1.56
20 – 40	17.54	3.07	0.50	13.97	6.14	1.58
40 – 55	6.19	1.04	0.24	4.91	4.33	0.43
Mangeesh –Oak						
0 – 20	30.61	8.16	1.01	21.44	8.07	1.23
20- 55	7.22	4.04	0.51	2.67	7.92	1.26
55 – 90	3.44	2.75	0.52	0.17	5.28	0.92
Atrush-Oak						
0 – 20	32.68	5.01	2.03	25.64	2.46	1.50
20 – 45	19.60	3.98	0.50	15.12	7.96	1.60
45 – 60	15.82	1.02	0.24	14.56	4.25	1.70

2-3 Humin

It is a complex mixture of humic and fulvic acids that cannot be extracted with acidic and basic solutions because it is precipitated in both media. Therefore, its values are higher than humic and fulvic acid, as showed from Table (2), where the results showed that the humin content in the study soil ranged between 0.17 gm.kg⁻¹ in the third depth of Mankeesh-oak soil to 25.64 gm.kg⁻¹ in the first depth for atrush soil - oak, it is noted from the results of the table that the humin decreases with the increase in the depth of the soil, and the highest values were in the surface depths of the different soil sites, except for the Mankeesh soil –pine were the highest value was in the third depth. The results showed that the ratio between HA/FA when it is less than 1 indicates the dominance of fulvic acid over humic acid and this appeared in the Atrush soil - pine, Mankeesh soil – pine. When the ratio between them is greater than 1 it means that humic acid exceeds fulvic acid as it is in the rest of the other soil sites, and it was possible to divide the soil according to HA/FA according to (Kononova, 1966) to the following groups (less than 1, 1 – 3, 3 – 6, 6 - 10) depending on the results obtained (Table 2), this division clearly indicates that there is a difference in the composition of humus in the humic compounds (humic and fulvic acid) according to the different environmental conditions of each soil and the type of forest and vegetation cover present (Oktaba et al., 2018). Regarding the optical density of the extracted humic compounds, which is expressed as E4/E6, as described by it (Kononova, 1966; Reddy et al., 2014). This ratio depends on the chemical composition of these humic compounds, as it is noted from Table (2) that this ratio between E4: E6 ranged between (0.43 - 1.79), this means that there are low values less than 1 and values between 1 - 2, where low values indicate the presence of humic compounds in the form of an aromatic network with relatively low molecular weights, while high values indicate the presence of humic compounds in the form of a aliphatic chain. Therefore, it is noticeable and according to the above results, we find the superiority of humic acid over fulvic acid in the study soils (Schnitzer et al., 1991; Moniruzzaman et al., 2019).

3- Organic complexes of iron and manganese

The association of the active organic groups present in the humic compounds with the minor elements results in the formation of complexes and chelates of these elements possessing the property of dissolution and movement in the soil solution, and this reduces the activity and effectiveness of these elements. Therefore, it can be said that humic compounds bind iron and manganese with them strongly, forming complexes and organic chelates for them (Tan, 2011;

Chirwa and Yerokun, 2012). In order to apply this concept to iron and manganese and the formation of organic complexes in the study soils, Table (3) presents the binding values of Fe and Mn with humic and fulvic acid, from which it is noted that the humic compounds represented by HA and FA vary in their ability to chelate iron and manganese in different soil locations according to the type of forest cover, the ability of humic acid to chelate iron and manganese was greater than fulvic acid in the formation of chelates for these two elements, as evidenced by the fact that the values of chelated iron (HA-Fe) ranged from 18.82 mg.kg^{-1} in the third depth of the Atrush soil -pine to 87.01 mg.kg^{-1} at The first depth of the Atrush soil – Pine. Likewise for chelated manganese (HA-Mn), its values ranged between 2.15 mg.kg^{-1} in the third depth of Mankeesh soil pine to 7.71 mg.kg^{-1} in Mankeesh soil - oak. The same behavior was observed with fulvic acid, the results showed that the values of chelated iron with fulvic acid (FA-Fe) were higher than the values of chelated manganese (FA-Mn), whose values ranged from 15.64 mg.kg^{-1} in the first depth of the Mankeesh soil - oak to 33.80 mg.kg^{-1} in the third depth of the Atrush soil - oak, the chelated manganese with fulvic acid gave values ranging between 0.35 - 15.02 mg.kg^{-1} in Atrush soil - pine and Atrush soil - oak.

Table (3): Organic complexes of iron and manganese with humic and fulvic acids in studied soils

Location	Depth	Fulvic acid		Humic acid	
		Fe-FA	Mn-FA	Fe-HA	Mn-HA
	cm	mg.kg^{-1}		mg.kg^{-1}	
Mangeesh – Pine	0 - 20	22.68	0.59	21.36	3.57
	20 - 50	18.39	0.68	65.46	4.24
	50 - 90	15.70	0.70	45.95	2.15
Atrush- Pine	0 - 20	27.77	15.02	87.01	6.50
	20 - 40	20.91	7.81	20.95	6.45
	40 - 55	33.80	1.79	18.82	3.82
Mangeesh – Oak	0 - 20	15.64	3.25	31.97	3.93
	20- 55	20.74	2.76	21.31	2.98
	55 - 90	24.05	1.27	19.70	7.71
Atrush- Oak	0 - 20	21.32	1.53	29.18	7.45
	20 - 45	24.49	1.19	23.14	5.47
	45 – 60	20.70	0.35	33.26	7.09

The results also showed that the soil of Atrush - pine excelled in the formation of chelated iron with humic acid and fulvic acid and manganese chelated with fulvic acid, while soil of Atrush - oak excelled in the formation of chelated manganese with humic acid over the rest of the soils of the other sites. This superiority of humic acid over fulvic acid in the formation of chelates and complexes is due to the active organic groups that are linked to each other in the form of an aliphatic chain and thus there is a greater chance for the hydrogen bonded with organic groups to be separated and replaced by iron or manganese, while in fulvic acid the organic groups are arranged in a ring shape aromatic, and in order for these rings to separate from each other, they need energy to break the bonds of these rings first, and then the hydrogen to separate from the organic groups secondly (Ali and Mindari, 2016; Chefetz et al., 2002). In light of these results, it was observed that there is a difference in the values of iron and manganese associated with humic and fulvic acid as organic complexes, which is consistent with the difference in the degree of decomposition of forest tree residues and plant residues, and in turn is reflected in the quantity and type of humic compounds resulting from the decomposition (Kumada, 1987).

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التداخل بين بعض العناصر الصغرى والمركبات الدبالية في بعض ترب الغابات شمالي العراق

قحطان درويش عيسى الخفاجي

قسم علوم التربة والموارد المائية / كلية الزراعة والغابات / جامعة الموصل

الخلاصة

الكلمات المفتاحية: المركبات الدبالية , الحوامض العضوية , الكثافة الضوئية

اختيرت اربعة بيدونات لترب مختلفة في الغطاء الغابي اثنان في كل من منطقتي مانكيش وأتروش في محافظة دهوك تحت أشجار الصنوبر والبلوط, بهدف دراسة توزيع المجاميع الرئيسية للمركبات الدبالية (حامضي الهيوميك والفولفيك) وارتباطهما مع أيوني الحديد والمنغنيز. فقد أوضحت النتائج بأن التباين في طبيعة الغطاء الغابي وسرعة تحلل بقاياه أدى الى اختلاف كل من توزيع محتوى المادة العضوية ونسب حامض الهيوميك/ حامض الفولفيك (FA/HA) وكمية المواد الدبالية الناتجة في كل تربة, وفيما يخص الكثافة الضوئية للمركبات الدبالية المستخلصة والمعبر عنها بـ E4/E6 فأن هذه النسبة تعتمد على التركيب الكيميائي لهذه المركبات الدبالية, اذ يلاحظ من النتائج أن هذه النسبة بين E4: E6 تراوحت بين (0.43 – 1.79), فالقيم المنخفضة تعبر عن وجود مركبات دبالية اروماتيكية ذات أوزان جزيئية واطئة نسبياً أما القيم العالية فانها تدل على وجود مركبات دبالية اليفاتيكية. كما بينت الدراسة وجود تباين واختلاف في قيم الحديد والمنغنيز المرتبطين مع حامض الهيوميك والفولفيك كمعقدات عضوية والذي يتماشى مع اختلاف درجة تفسخ وتحلل مخلفات اشجار الغابات ومخلفات بقايا النباتات وبدوره ينعكس على كمية ونوع المركبات الدبالية الناتجة من التحلل, اذ تفوقت تربة اتروش تحت غابات الصنوبر في تكوينها للحديد المخلبي مع كل من حامض الفولفيك وحامض الهيوميك والمنغنيز المخلبي مع حامض الفولفيك في حين تفوقت تربة أتروش تحت غابات البلوط في تكوين المنغنيز المخلبي مع حامض الهيوميك على بقية الترب المدروسة.