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Studying the Effect of Agriculturally Exploited Soils on the Transformation of Mica Minerals using Infrared Spectroscopy (IR)

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Five soil sites were selected in the Middle Euphrates region, represented by the governorates (Diwaniyah, Najaf, Babel, and Muthanna), for soils of different agricultural exploitation (rice, vegetables, wheat, and palm trees), in addition to an unexploited agricultural soil. The soils were distinguished by having the same topography and climatic conditions and similar texture in order to study the weathering of mica minerals by using the infrared technique. The appearance of the absorption spectrum at the frequency of 3550 cm^{-1}, with medium broadening in the unexploited agriculture soil (Muthanna), indicated a gradual transformation of mica towards di-octahedra minerals, and reflected the increasing substitution of Al^{+3} in tetrahedral sheet in these soils. The results of the examination of clay particles in soils exploited for cultivation of rice (Diwaniyah) showed that the absorption spectrum at the wavelength of 1642 cm^{-1} represents the absorption of zeolite water, while the presence of absorption spectra at the frequencies of 796, 1036, 3418, and 3616 cm^{-1} indicated the presence of mica minerals in all rice soils, also the presence of the broad absorption spectrum band at the frequency of 3418 cm^{-1}, which was accompanied by the presence of the spectrum band at the frequency of 1036 cm^{-1}.

Key words: Infrared spectroscopy; clay mineral; discriminant analysis

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Interactions between soil clay and soil organic materials and mineralogical soil play key roles in soil chemistry, which will influence soil quality and soil fertility. Chemical analysis of soil clay and soil organic materials is time-consuming, furthermore chemical analysis is difficult to be used in in situ monitoring, and soil extraction may destroy soil structure [1]. Infrared spectroscopy is an alternative technique for qualifying the interactive effect of soil clay and soil organic materials, and is thus widely used in soil analysis [2]. Recently, infrared photoacoustic spectroscopy (FTIR-PAS) was applied in soil science, which shows a great potential in soil analysis [3]. Usually, infrared transmittance and reflectance spectroscopy is used to characterize soil clay minerals, however, the pellet preparation is time-consuming for transmittance spectra, and the signal is not stable enough due to varied sample particle size for reflectance spectra [4]. The infrared (IR) technical is widely used in the study and analysis of inorganic crystalline materials as well as organic materials in soil, and it is a confirmatory study of results obtained from studying and examining these materials using X-ray, or differential thermal analysis tests. DTA, or TEM scanning electron microscope. The importance of using infrared rays in the study of clay minerals increases through its contribution to the diagnosis of amorphous bands of the minerals by measuring the wavelengths of the rays, in addition to the wavenumber or what is called the frequency field, in which the absorption of rays occurs. The current study aimed to study the transformations of mica minerals and compare them with the effect of different agricultural utilization of the soil, by using the infrared (IR) technical.

MATERIALS AND METHODS

Five soil sites were chosen from the Middle Euphrates region, represented by governorates (Najaf, Babel, Diwaniyah, and Muthanna), in addition to an unexploited soil. The soils were exploited by planted with different crops (wheat, rice, vegetables, palm trees and others for the unexploited agricultural soil). The chosen soils were characterized by having the similar texture, parent material, flat topography, and climatic conditions in order to avoid the effect of the variation in these characters on chemical and mineralogical properties of the studied soils. After the samples were collected, physical and chemical analyses were performed, as shown in **Table 1.** and clay fraction was separated by following steps

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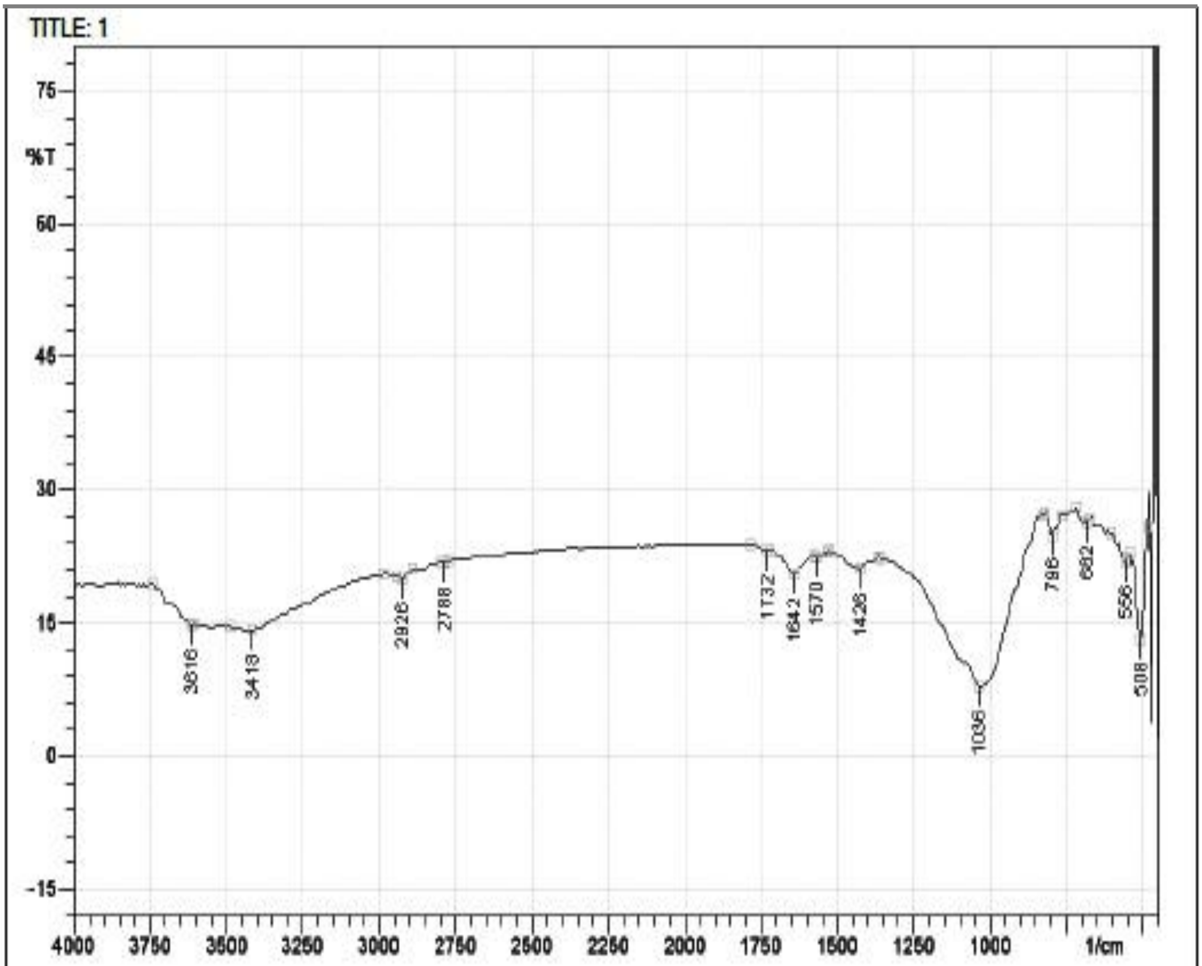
Table 1. Physical and chemical properties

Governorate and type of agricultural exploitation	Volumetric distribution of soil separators				$CaCo_3$	O.M	CEC Cmol.Kg^{-1}	pH	Ec ds.m^{-1}
	Sand	Silt	Clay	Texture	g. Kg^{-1}				
Diwaniyah / Rice	185	435	407	Silt clay	253.17	11.92	22.15	7.7	4.40
Najaf / Vegetables	195	405	400	Silt clay	252.08	2.00	21.28	7.5	2.85
Babylon / Wheat	98	450	452	Silt clay	213.72	15.5	26.10	7.8	3.80
Muthanna / Nakheel	75	465	460	Silt clay	2.46.60	11.00	20.10	7.8	2.21
Muthanna / Unexploited	188	450	362	Silt clay loam	216.99	2.10	19.21	7.8	26.50

represented by dissolved salts, carbonate minerals, and free oxides according to the methods described in [5], [6] and [7], respectively. Sand particles were separated using a 50 microns sieve and clay from silt by sedimentation method according to the Stock law, as stated in [8]. The IR technical was used to examine the mineral properties of the soils using Infrared Spectro photo, SHIMADZU IR AFFinity^{-1}.

RESULTS AND DISCUSSION

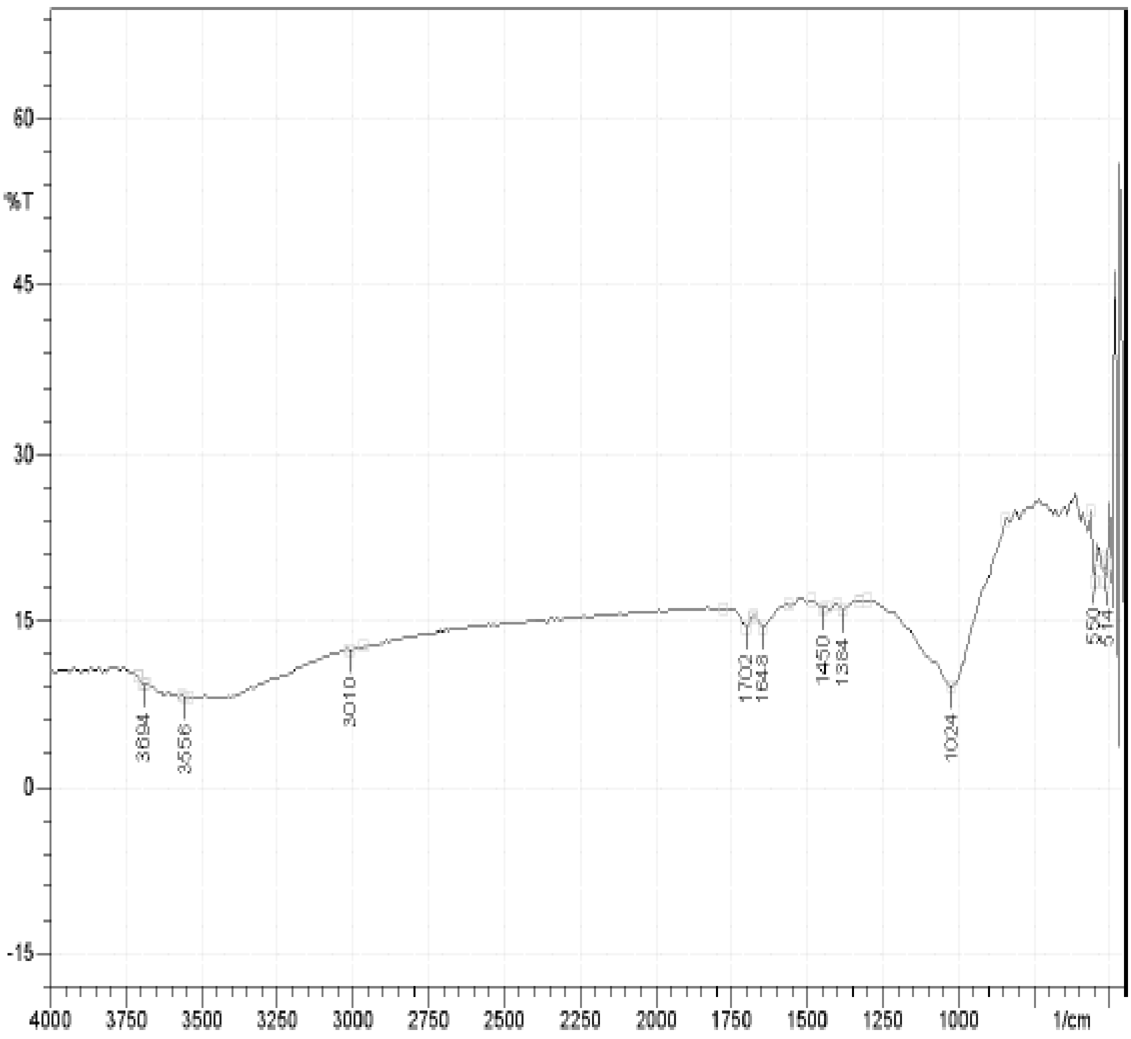
The infrared (IR) technical was used in the study and determination of the types of minerals present in the clay fraction of the studied soils. The infrared spectrum bands of selected samples were examined (**Figures 1-5**). **Figure 1** shows the results of the clay samples of the Al Diwaniyah soil site, which is exploited for the cultivation of rice. The emergence of broad spectrum bands at the frequencies of 3616 and 3418 cm^{-1} confirmed the presence of the Al-OH hydroxide bond, and that the presence of these broad bands was accompanied by the presence of the spectrum band at the frequency of 1642 cm^{-1}, which indicated the presence of water molecules on the surfaces of clay minerals in those soils, which is called zeolite water. These results were identical to the results obtained by [9-10] while studying the nature of the effect of the source of sedimentation on the mineral properties of some Iraqi soils, where the results showed the presence of a spectrum band at 1635 cm^{-1}, which indicated the hydrated nature of the clay minerals carried within the sediments of the Tigris River. Therefore, the appearance of the spectrum band at the frequency of 1642 cm^{-1} in the clay samples of the rice soils in the current study confirmed the hydrated nature of the clay minerals in those soils, which is attributed to the waterlogged conditions that the soils are exposed to during the cultivation season of rice. Numerous studies conducted on rice fields and the effect of their conditions on the properties of clay minerals have shown that the prevailing waterlogged conditions in rice fields affect the structural composition of clay minerals, transforming them into hydrated minerals. The results showed the emergence of absorption spectrum bands at the frequencies of 796, 1036, 3418, and 3616 cm^{-1}, which indicated the presence of mica minerals in the clay samples of the soils, and that the variation of these bands in their intensity and values indicated the diversity of the presence and appearance of mica minerals in more types of minerals. Therefore, the appearance of the absorption spectrum band at the frequency of 3616 cm^{-1} represented the hydroxylated $^{-}$OH bond, the band at 1036 cm^{-}1 represented the Si-o bond, and the band at 796 cm^{-1} represented the Al-OH and Al-o-Al bonds, as well as the absorption spectrum band at the frequency of 682 cm^{-1}, which represented the Si-o-Si, Si-o-Si bonds. The results confirmed the presence of the biotite mineral in the sample, as [15] showed that the absorption spectrum bands of the biotite mineral appear at the frequencies of 766, 1039, 3410, and 3622 cm^{-1}, while [10] showed that the absorption spectrum bands of the biotite mineral within the frequencies of 675, 1072, 3481, and 3618 cm^{-1}. The results also showed the presence of a wide absorption spectrum band at the frequency of 3418 cm^{-1}, accompanied by the presence of the spectrum band at the frequency of 1036 cm^{-1}, which confirmed the absorption of water molecules by the mica minerals. The results were consistent with [16] that showed the presence of broad bands in the absorption spectrum at the frequency of 3320 - 3400 cm^{-1}, accompanied by the spectrum band at the frequency of 1639 cm^{-1}, thus confirms the presence of hydrated mica (Illite mineral) in the studied samples. These were confirmed by the results of [15] that the presence of the broad spectrum band at the frequency of 3410 cm^{-1} represents the absorption of water molecules by mica minerals, which was supported by the presence of the absorption spectrum band at the frequency of 1624-1639 cm^{-1}. All these results confirm what the current study obtained with regard to the hydrated nature of the clay minerals in the soils.The results of the clay samples of the Diwaniyah site **(Figure 1**),



Peak table of 4

PeakNumber	Begin [1/cm]	Maximum [1/cm]	End [1/cm]	Width [1/cm]	Height [%T]	Absolute Height [%T]	Peak Area	Peak Area (Abs. Sum)	Peak Area (Algebr. Sum)
1	484	508	544	60	-12.51	13.22	-387.83	395.69	-395.69
2	550	556	604	54	-3.62	21.83	-106.42	116.11	-116.11
3	676	682	718	42	-0.65	26.29	-25.81	25.82	-25.81
4	766	796	820	54	-2.75	24.92	-79.95	82.06	-82.06
5	826	1036	1360	534	-18.84	7.84	-5177.82	5188.06	-5188.06
6	1366	1426	1528	162	-4.03	21.11	-496.83	510.78	-510.78
7	1534	1570	1576	42	-2.15	22.46	-83.73	95.02	-95.02
8	1576	1642	1720	144	-3.72	20.62	-348.23	356.84	-356.84
9	1720	1732	1786	66	-0.87	23.14	-31.51	34.02	-34.02
10	2776	2788	2800	24	-0.12	21.85	-1.2	1.2	-1.2
11	2890	2926	2944	54	-0.56	20.01	-12.7	12.7	-12.7
12	2986	3418	3490	504	-5.73	14.17	-1462.84	1478.32	-1478.32
13	3610	3616	3742	132	-4.86	14.74	-343.76	357.73	-357.73

Figure 1. Infrared rays of the clay separator in Al Diwaniyah soil cultivated with rice.



Peak table of 8

PeakNumber	Begin [1/cm]	Maximum [1/cm]	End [1/cm]	Width [1/cm]	Height [%T]	Absolute Height [%T]	Peak Area	Peak Area (Abs. Sum)	Peak Area (Algebr. Sum)
1	508	514	532	24	-2.19	18.46	-34.1	38.53	-38.53
2	532	550	562	30	-5.2	18.56	-68.96	73.4	-73.4
3	844	1024	1300	456	-13.26	9.1	-3310.07	3318.85	-3318.85
4	1330	1384	1402	72	-3.23	15.92	-207.06	223	-223
5	1438	1450	1486	48	-2.44	16.17	-98.71	110.46	-110.46
6	1564	1648	1678	114	-2.61	14.49	-204.73	212.77	-212.77
7	1684	1702	1780	96	-2.32	14.38	-96.47	100.87	-100.87
8	2968	3010	3016	48	-0.13	12.32	-3.33	3.33	-3.33
9	3544	3556	3568	24	-0.55	8.05	-11.96	14.63	-14.63
10	3688	3694	3712	24	-0.22	9.31	-3.13	3.13	-3.13

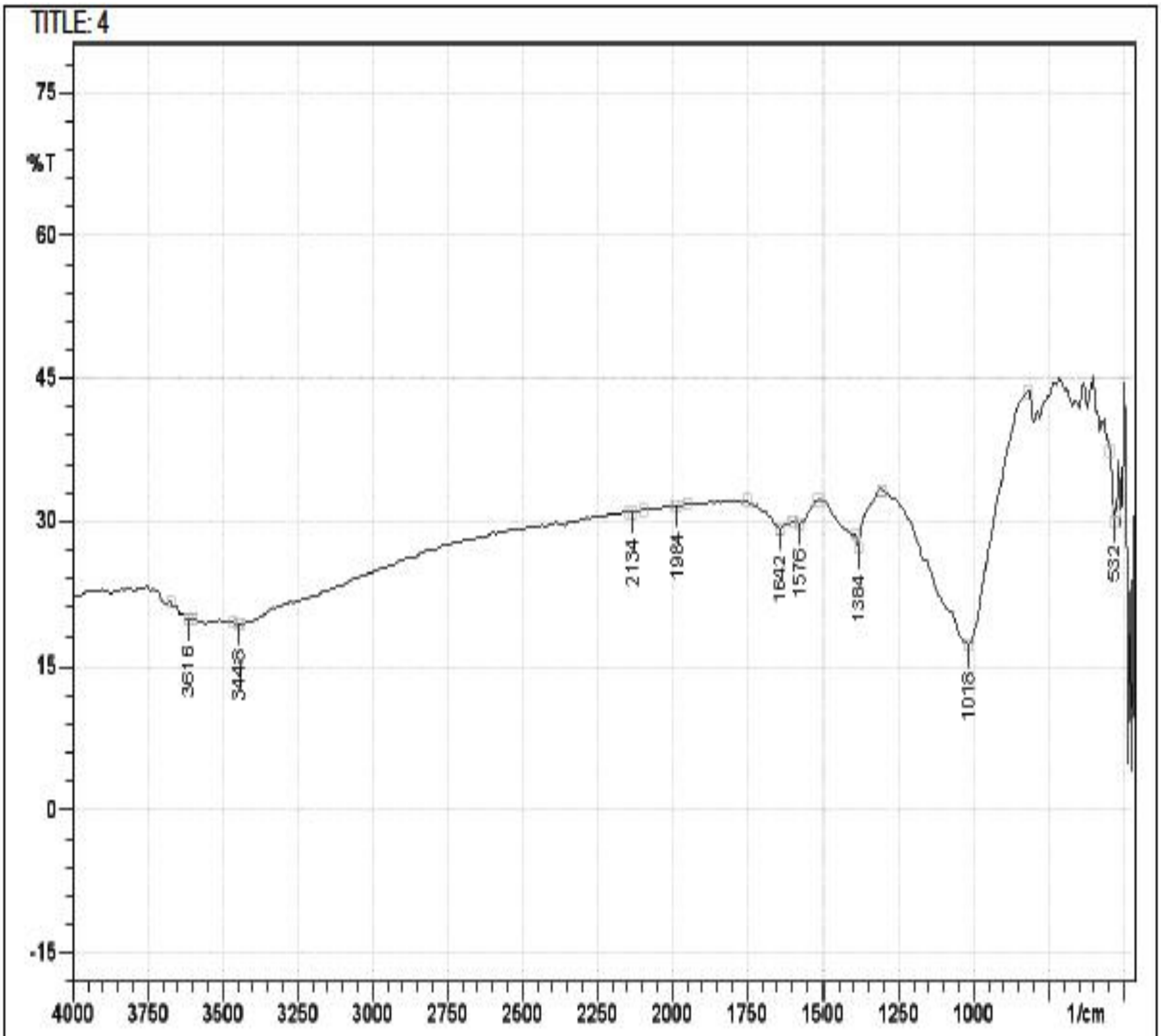
Figure 2. Infrared rays of the clay separator in Babylon soil cultivated with wheat.

which is exploited for the cultivation of rice, showed the presence of absorption spectrum bands at the frequencies of 682, 1036, 1426, 1642, and 3616 cm^{-1}, which confirmed their relevance to the group of smectite minerals in the samples. [17] showed that the spectrum bands of smectite minerals appeared at the frequencies of 3405 – 3616, 843, 915, 1040, 1450, and 1630 cm^{-1}. Also, the results of the examination **(Figure 1**) showed that no absorption bands appeared at the frequencies of 915 - 935 cm^{-1} and 820 - 843 cm^{-1}, which represent the hydroxylated bonds of the minerals (Al Al-OH) and nitronate (Fe Fe-OH) [16-17]. As the bands represent the two minerals as individuals of the group of smectite minerals, their absence in the examined samples suggests that the smectite minerals present in those soils belong to the montmorillonite-mineral. This assumption was confirmed by the presence of the band at the frequency of 682 cm^{-1}, belonging to the Mg^3-$(OH)_6$ group, which indicated that the octahedral sites of the mineral were occupied by Mg^{2+} ions. This variation in behavior led us to believe that the existing montmorillonite mineral was trioctahedral smectite, and the appearance of the absorption band at the frequency of 1426 cm^{-1} was due to the presence of calcium in some octahedral sites of the montmorillonite mineral, according to what has been shown by several studies [16, 17, 18]. It was shown that the montmorillonite present in Iraqi soils is Ca-Montmorillonite and attributed that to the calcareous nature of Iraqi soils. In general, these results confirm that the mineral montmorillonite in those soils is inherited from the mica of minerals, and the evidence for this is that both the minerals of mica and montmorillonite present in those soils are the tri-octahedra. [19] showed that all minerals in mica weathering series follow in their crystal structures the same composition as the mica of minerals, meaning that the smectite of minerals group (montmorillonite, bidlite and nontronite) inherited from the tri-octahedra mica minerals will have their tri-octahedra crystal structures as well.

The results of the examination (**Figure 1**) showed the appearance of the absorption spectrum bands at the frequency ranges of 3418-3616 cm^{-1} and 682-796 cm^{-1}, which confirmed the presence of chlorite in the clay samples of the Al Diwaniyah site, and these results were consistent with the findings of [15], and the appearance of the band at the frequency of 682 cm^{-1}, belonging to the Mg^3 - $(OH)_6$ group, indicateed that the hydroxyl interlayer in the chlorite mineral is in the form of a Brucite trio-octahedra [10-20]. The emergence of absorption spectrum bands at the frequencies of 796, 565, and 508 cm^{-1} confirmed the presence of the interstratified mineral (Illite-smectite) in the clay samples of these soils, and that these results were identical to that obtained by [21], which showed that the absorption spectrum bands of the interstratified mineral (illite - smectite) fall within the range of 750 and 530 cm^{-1}, while [22] showed that the absorption spectrum bands of the interstratified mineral (Illite-montmorillonite) can be diagnosed within the range of 500-700 cm^{-1} when using infrared technical to examine the clay particles, and according to the results obtained which confirmed the presence of the interstratified mineral (illite-Smectite) in the clay samples of the Al Diwaniyah site.

Figure 2 shows the results of the examination of the clay fractions in the soil of the Babylonian site exploited for the cultivation of wheat. **Figure 2** shows the absorption spectrum bands at the frequency ranges of 3010-3556 cm^{-1} and 1386-1450 cm^{-1}, which confirmed the presence of smectite minerals in the sample. It was also observed from the results that the examination curve was divided into three main parts. The first part included the frequencies of 3010, 3556, and 3694 cm^{-1}, which represented the Mg - OH - Mg group. [23] showed that the Mg - OH - Mg group falls within the frequency range of 3583 -3585 cm^{-1}, which confirmed the presence of tri-octahedra (biotite) mica minerals. The second part of the curve included the frequencies of 1384, 1450, 1648, and 1702 cm^{-1}, which represented the Si-O group, as [23] showed that the Si-o belongs to mica minerals within the frequencies of 1012, 1090, and 1112 cm^{-1}. The third part included the frequency range of 514 – 550 cm^{-1}, which confirmed the presence of tri-octahedra (biotite) mica minerals in the sample and confirmed the presence of tri-octahedra mica minerals in the clay sample of the Babylonian soil. Several studies [22, 24, 25] had shown that dioctahedral mica minerals (muscovite) exhibit sharp absorption spectrum bands in the frequency range of 400-600 cm^{-1}, which represent the aggregates Si-o and Si-o-Si. The studies confirmed that these frequencies shift with increasing Mg^{2+} content in the octahedra layer to fall within the range of 509-535 cm^{-1}, which confirms the presence of the tri-octahedra mica (biotite) in the examined sample, and the results showed the presence of the absorption spectrum band in the frequency range of 3556 - 3694 cm^{-1} belonging to the OH bond, and the appearance of the absorption spectrum band in the frequency range confirmed the presence of the mineral chlorite in the examination sample, as [26] showed that the hydroxyl groups in the octahedra layer of minerals. The clay 2:1 gave the absorption spectrum band at the frequency of 3617 cm^{-1}, and another bundle of the absorption spectrum bands may be given in the clay samples taken from the horizons of soils, in which the phenomenon of chloritization occurs at the frequency of 3570 cm^{-1}, since the frequency can increase to reach 3580 cm^{-1} as a result of the contribution of magnesium ions in increasing the filling of the inner hydroxyl layer in these minerals, transforming it in the direction of swollen chlorite, and this is confirmed by [27] that the hydroxyl groups of the inner hydroxyl layer in the clay minerals shifted towards the chlorite mineral. The absorption spectrum band appeared in the frequency range of 3400-3580 cm^{-1}. The emergence of the absorption spectrum band in the field range of 3556-3694 cm^{-1} in the clay samples of the soil of the Babylonian site confirmed the presence of the mineral chlorite in the examined sample, which was identical to the results obtained by [28] during a study of the phenomenon of chloritization in Iraqi soils.

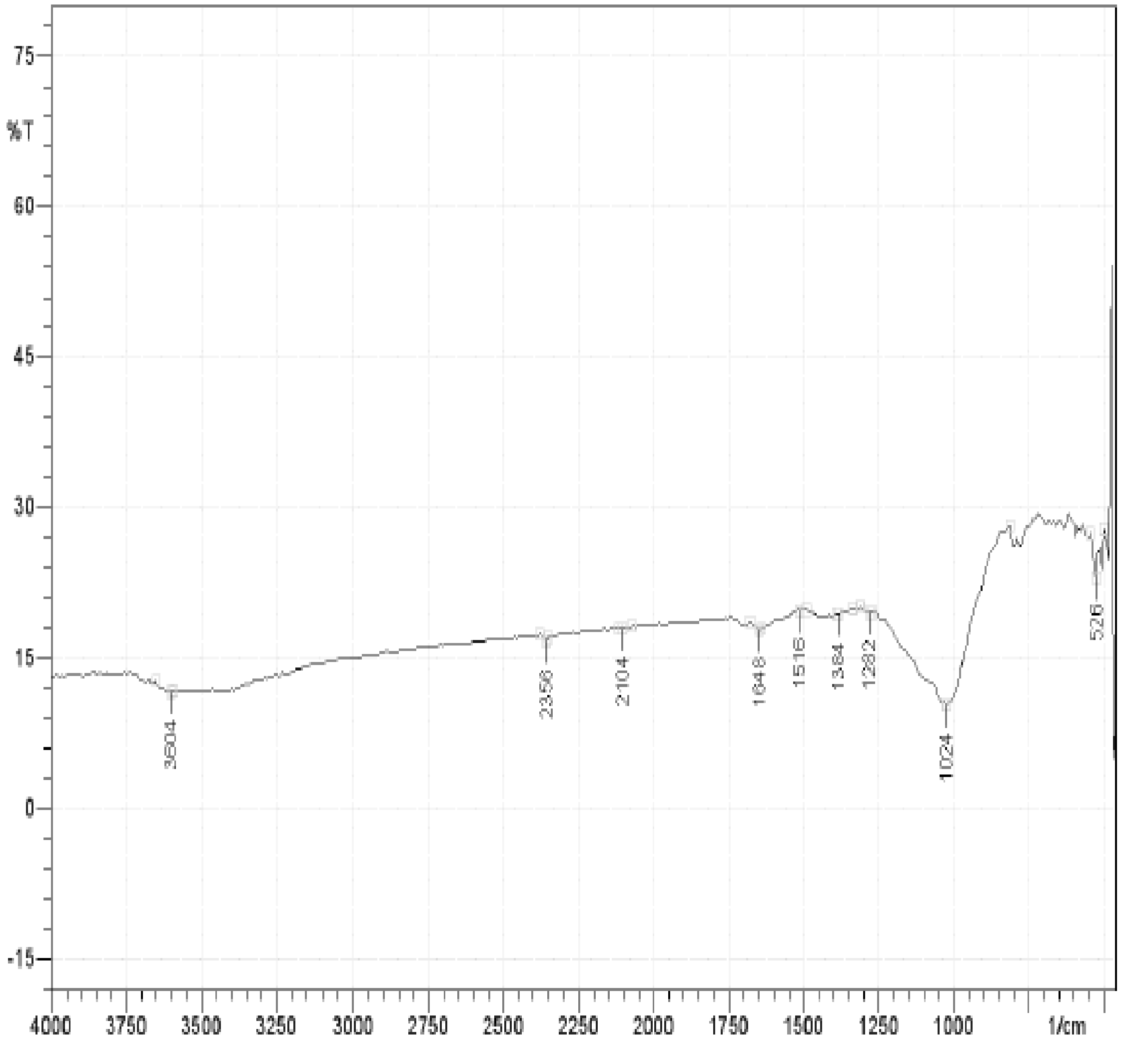
The results of the examination of the soil samples of the Najaf site (**Figure 3**), exploited by cultivating vegetables, showed the presence of absorption spectrum bands at the frequencies of 1018, 3448, and 3616 cm^{-1}, which indicated the presence of mica minerals in the samples. The presence of the band at 532 cm^{-1} confirmed the presence of the tri-octahedra (biotite) mica minerals in the samples [23]. The emergence of absorption spectrum bands at the frequencies of 1018, 1576, 1642, and 3616 cm^{-1} confirmed the presence of smectite minerals in the clay samples of the soil. The absence of absorption spectrum bands in the frequeny ranges of 915 - 935 cm^{-1} and 820 - 843 cm^{-1}, which represent the hydroxylated bonds of the two minerals (Al Al-OH) and nitronate (Fe Fe-OH), confirmed that the smectite minerals present in these soils belong to the mineral montmorillonite [16-17].



Peak table of2

PeakNumber	Begin [1/cm]	Maximum [1/cm]	End [1/cm]	Width [1/cm]	Height [%T]	Absolute Height [%T]	Peak Area	Peak Area (Abs. Sum)	Peak Area (Algebr. Sum)
1	520	532	544	24	-4.01	30.13	-35.46	35.46	-35.46
2	820	1018	1306	486	-23.71	17.18	-5908.43	5920.36	-5920.36
3	1306	1384	1396	90	-9.13	27.18	-509.02	543.48	-543.48
4	1516	1576	1594	78	-4.36	29.76	-286.77	306.17	-306.17
5	1600	1642	1750	150	-4.13	29.27	-372.78	384.12	-384.12
6	1948	1984	1990	42	-0.09	31.61	-3.03	3.03	-3.03
7	2098	2134	2146	48	-0.06	30.95	-2.71	2.71	-2.71
8	3442	3448	3466	24	-0.08	19.25	-1.5	1.5	-1.5
9	3604	3616	3676	72	-1.3	19.66	-68.96	73.26	-73.26

Figure 3. Infrared rays of separated clay in Najaf soil cultivated with vegetables

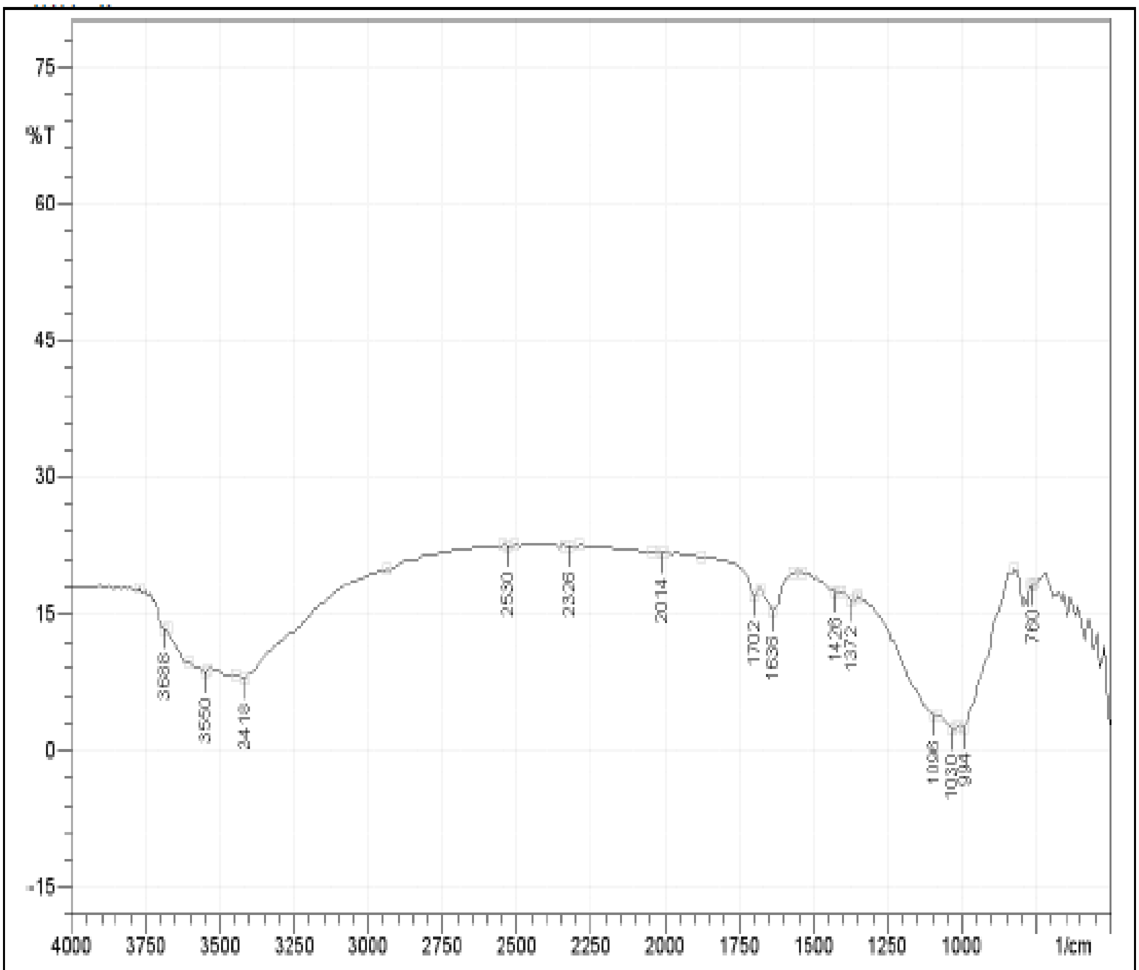


Peak table of20

PeakNumber	Begin [1/cm]	Maximum [1/cm]	End [1/cm]	Width [1/cm]	Height [%T]	Absolute Height [%T]	Peak Area	Peak Area (Abs. Sum)	Peak Area (Algebr. Sum)
1	502	526	544	42	-4.63	23.05	-101.32	101.32	-101.32
2	814	1024	1270	456	-15.52	10.36	-3734.27	3745.83	-3745.83
3	1270	1282	1312	42	-3.78	19.58	-148.87	169.49	-169.49
4	1336	1384	1390	54	-3.08	19.35	-158.75	176.36	-176.36
5	1492	1516	1648	156	-1.63	19.66	-278.36	290.2	-290.2
6	1642	1648	1678	36	-2.32	17.88	-68.84	79.67	-79.67
7	2074	2104	2116	42	-0.13	17.95	-2.48	2.49	-2.48
8	2350	2356	2380	30	-0.74	16.69	-9.03	10.24	-10.24
9	3592	3604	3652	60	-0.56	11.7	-29.52	30.62	-30.62

Figure 4 Infrared rays of the clay joint in Muthanna soil planted with palm trees.

The results of the examination in **Figure 4** related to the clay samples of the soil of the Muthanna site exploited for the cultivation of palm trees showed the emergence of absorption spectrum bands at the frequencies of 526, 648, and 3604 cm^{-1}, which confirmed the presence of mica minerals in the samples, and that the appearance of the band at 3604 cm^{-1} in width, which was accompanied by the band at 1648 cm^{-1} confirmed that the present mica minerals were hydrated mica (Illite mineral) [15-16]. Also, the results showed the presence of absorption spectrum bands at the frequencies of 1024, 1384, 1516, 1648, and 3604 cm^{-1} were related to smectite. [17] showed that bands of the spectrum of smectite minerals appeared at the frequencies of 1450, 1630, 3616, 3604, and 1040 cm^{-1}. The emergence of the absorption spectrum band at the frequency of 526 cm^{-1} in the clay samples of the Muthanna site suggested the presence of the interstratified mineral (illite - smectite) in the sample, since [21]one of the interstratified mineral bundles (Illite - smectite). It appeared at the frequency of 530 cm^{-1}, and was shared by both [22] as they showed that the absorption spectrum band of the interstratified mineral (Illite-Montmorillonite) appeared at the frequency in 500 cm^{-1} and took a wide range to reach range 700 cm^{-1}.



Peak table of 12:

PeakNumber	Begin [1/cm]	Maximum [1/cm]	End [1/cm]	Width [1/cm]	Height [%T]	Absolute Height [%T]	Peak Area	Peak Area (Abs. Sum)	Peak Area (Algebr. Sum)
1	754	760	766	12	-0.26	18.1	-1.54	1.54	-1.54
2	826	994	1012	186	-17.85	2.41	-1785.96	1839.16	-1839.16
3	1018	1030	1078	60	-17.96	2.35	-1039.88	1142.43	-1142.43
4	1078	1096	1348	270	-16.59	3.81	-2736.64	2797.69	-2797.69
5	1354	1372	1408	54	-4.36	16.44	-213.08	235.45	-235.45
6	1414	1426	1540	126	-3.49	17.38	-303.77	318.47	-318.47
7	1570	1636	1678	108	-5.82	15.36	-423.07	438.21	-438.21
8	1678	1702	1876	198	-4.25	17.04	-274.39	285.63	-285.63
9	2002	2014	2044	42	-0.12	21.65	-4.1	4.1	-4.1
10	2290	2326	2338	48	-0.12	22.38	-3.02	3.02	-3.02
11	2506	2530	2548	42	-0.13	22.41	-2.9	2.9	-2.9
12	2938	3418	3448	510	-11.15	7.96	-2750.44	2786.02	-2786.02
13	3538	3550	3604	66	-9.96	8.64	-624.47	679.81	-679.81

Figure 5. Infrared rays of the clay separator in unexploited Muthanna soil.

The results of the examination in **Figure 5** for the clay samples of the unexploited Muthanna soil showed the presence of absorption spectrum bands at the frequencies of 1636, 3418, and 3550 cm^{-1} that confirmed the presence of mica minerals in the samples, and that the appearance of the band at 3550 cm^{-1} with an average amplitude indicated a gradual shift. The gradual occurrence of mica minerals in the direction of di-octahedra mica an increase in aluminum ion substitution in the tetrahydra layer, and that the presence of the band at 3418 cm^{-1} next to it confirmed the presence of tri-octahedra mica in the sample as well [23]. Therefore, the two types of mica minerals (muscovite and biotite) could be present in the examined samples, which confirmed the presence of biotite in these soils with the emergence of the absorption spectrum band at the frequency of 760 cm^{-1} [15]. The presence of absorption spectrum bands at the frequencies of 994, 1030, 1426, 1636, and 3418 cm^{-1} in the clay samples of the soil of the Al-Muthanna site may be due to the minerals of(montmorillonite and bid lite since the frequencies were 1030, 1426, 1636, and 3418 cm^{-1}. For the hydroxylated bond Mg_3-OH, which confirmed the presence of the montmorillonite mineral in the samples, and the appearance of the absorption band at the frequency of 1426 cm^{-1} due to the presence of calcium in some octahedra sites of the montmorillonite mineral [10-17], as well as the appearance of the 994 cm^{-1} band confirmed the presence of bid lite mineral in those soils. [16-29] showed the presence of the beidellite mineral in Iraqi soils and indicated that the presence of the beidellite mineral in these soils is due to the parent material of sedimentary soils transported by the Tigris and Euphrates.

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