UDK 543.054:543.421/424

ESTIMATION OF QUALITY OF CLEANING OF THE HUMUS MATTERS BY MEANS OF FTIR SPECTROSCOPY

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The quality of cleaning of humus specimens extracted from chernozem (black soil) and commercial preparation "Humifild" was studied by the method of the infrared spectroscopy.

During the preparation of humus substances to study their structure and properties, as well as factory production of humus preparations for their practical use in agriculture, the researchers faced a difficult question of purification of humus preparations from impurities, particularly clay minerals. Clay minerals are very small and they have strong adsorption properties, which leads to the formation of relatively stable organic-mineral colloidal systems with humus substances. There are a number of methods for purification of humus substances. Usually scientists change the terms of operations of cleaning and often combine them. It usually leads to receiving humus preparations that contain different additives [1]. Further investigation of the properties of these preparations showed, that their comparison is hampered because of the influence that impurities can cause. Commercial preparations may create additional difficulties due to the influence of impurities on the filter sprayers they are applied by.

The aim of this study is to compare the quality of cleaning of humus preparations using infrared (hereinafter - the IR) spectroscopy. IR spectroscopy was chosen due to the fact, that this method is well studied and successfully applied to the detection of clay minerals in soils and different functional groups of humus preparations.

Methodology of the research. Soil humus preparations were obtained from podzolized heavy loamy black soil after decalcification-sulphurous acid by common methods [2] without the use of coagulants and without division into humic and fulvat acids. The first variant was a drug that was cleared by centrifugation of suspension of humus substances at 8000 rpm during 10 min. Variant two - the preparation that is cleared by centrifugation at 8000 rpm during 10 min followed by passing the solution through cationite KU-2 and anionite AB-17-8. After this cleaning the solutions of soil and humus substances dried at 55 ˚ C. The third option was a commercial humus product "Humifild" of German production. Preparations were studied by Fourier infrared spectrometer "ALPHA" produced by "Bruker".

Results of the research. In humus substances of the 1st option, purified only by centrifugation, there are no absorption zones with maximum 410, 990 and 646 cm^{-1}, which belongs to quartz (Fig.). However, there are zones with maximum that belong to the montmorillonite, kaolinite and feldspar. In particular montmorillonite was detected behind a broad zone 2800-3700 cm^{-1} with maximum at 3400 cm^{-1} and maximum of 1635 cm^{-1} (connected with the OH-groups of the water molecules presented between reservoir areas of montmorillonite) [3-5]. Kaolinite - maximum 3695, 3620, and 908 cm [6]. Present carbonates - 1425 cm^{-1}. Feldspars are present - 410, 990 and 646 cm^{-1} [7]. There is a maximum of 615 cm^{-1}, which belongs to sulfates [8]. There is also a zone with a maximum at 1385 cm^{-1}, which belongs to the symmetric vibrations of -CH_3 groups [9]. It can be connected with high concentration of organic substance in humus preparations.

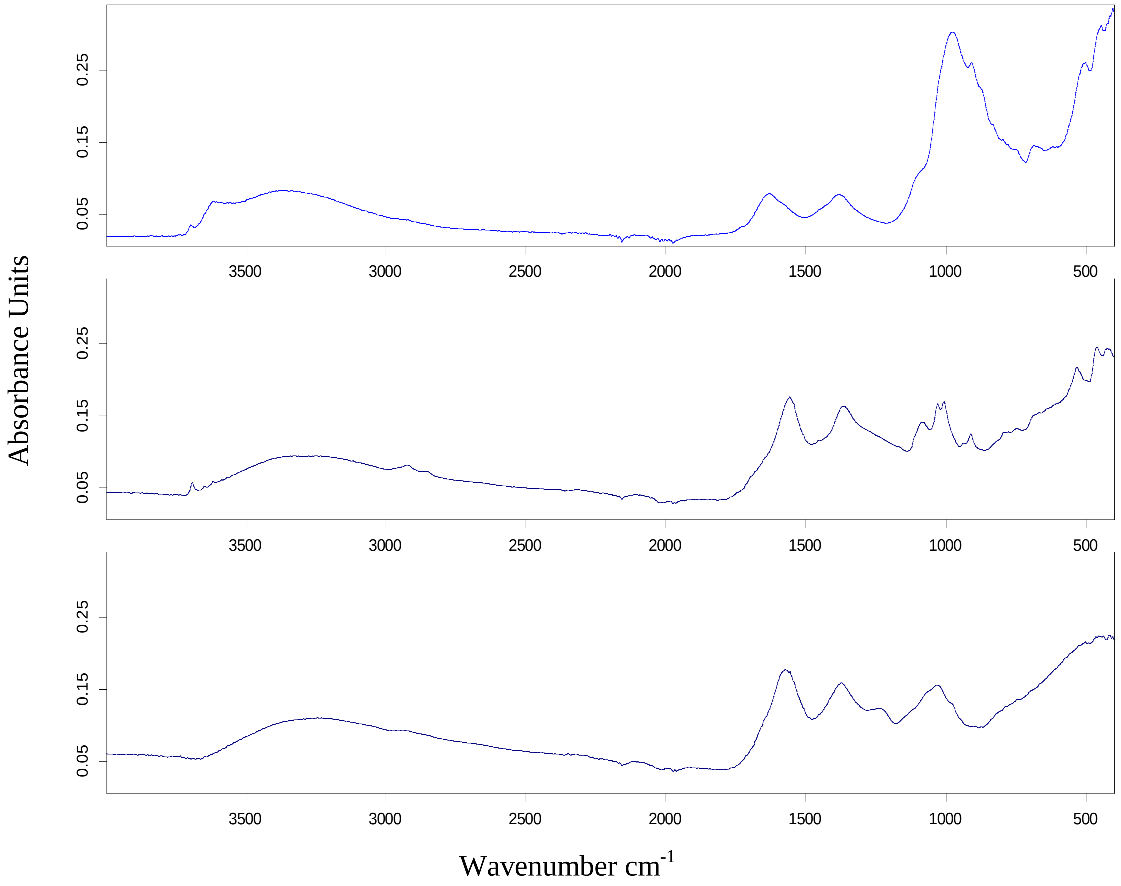

Fig. IR spectra of humus preparations: Variant 1 (top), variant 2 (bottom) and variant 3 (average).

In the spectrum of humus substances of the 2nd option there are no characteristic zones, which are attributed to kaolinite and montmorillonite. In the spectrum there is a broad intense zone in the interval 3300-3500 cm^{-1}, which must be included to fluctuations of groups -OH, connected with intermolecular hydrogen bonds, there is maximum at 1580 and 1385 cm^{-1}, usually absorption zones with maximum of 1590 - 1580 and 1400-1390 cm^{-1} attributed to the vibration of -COOgroup, while it is stated that within the zones 1625-1600 cm^{-1} there are vibration -C=C- group of amides I and hygroscopic water; there is a weak side at 1540 cm^{-1}, which belongs to II amides; there are polysaccharides at 1045-990 cm^{-1} [10-16]. In the range of 2nd option there was a maximum of 1223 cm^{-1}, some scholars refer the maximum in humus substances in the interval 1260-1200 cm^{-1} to amide III or groups C-O-C of aromatic esters [9, 17]. Zone with a maximum at 2924-2922 and 2855 cm^{-1} is also different, which belongs to the vibrations of aliphatic C-H bonds and there is a weak side at 1700 cm^{-1}. Similar spectrum in the interval 1690-1716 cm^{-1} are referred by scholars to the vibrations C = O bonds [10, 17]. There is a different weak side at 1506 cm^{-1}, similar maximum at 1510-1500 cm^{-1} of weak intensity attributed to fluctuations -C = C- group of aromatic compounds.

The range of 3rd variant in general is similar to the spectrum of 2nd variant. However, there are some differences. In the range of 3rd option the kaolinite with a maximum of 3695, 3620 and 908 cm^{-1} is present [6]. There are mild maximums of 795, 775 and 695 cm^{-1}, which belong to Si-O symmetric stretching vibration of quartz [3, 7]. There is a maximum at 531 cm^{-1}, which belongs to the Al-O-Si groups and a maximum of 467 cm^{-1} of Si-O-Si group [18]. More distinct is zone with a maximum at 2924-2922 and 2855 cm^{-1}, which belongs to the vibrations of aliphatic C-H bonds.

The whole range of 2nd option in the interval 400-950 cm^{-1} is more aligned comparing with the corresponding spectrum of 3rd option.

Range of research options sometimes contain a number of bands, including 838 cm^{-1}, attributing them to specific functional groups is problematic due to the lack of relevant data in the scientific sources.

Conclusion. Humus specimens cleared by centrifugation at the speed of 8000 rpm during 10 minutes, contain impurities of clay minerals. Humus substances, further purified by going through cationite and anionite, are released from clay minerals, sulfates, oxides of aluminum and silicon. Commercial preparation "Humifild" contains minor amounts of kaolinite, trace amounts of quartz, alumina and silicon.

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Одержано 16.05.13

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Определение качества очистки гумусовых препаратов при помощи ИК- спектроскопии

Методом ИК- спектроскопии исследовали качество очистки гумусовых препаратов от примесей. Установлено, что центрифугирование на скорости 8000 оборотов в минуту в течение 10 минут очищает раствор гумуса от кварца и уменьшается содержание глинистых минералов. Центрифугирование и использование ионного обмена очищает раствор гумуса от глинистых минералов, сульфатов, оксидов алюминия и кремния. Коммерческий препарат гумусовых веществ содержит незначительные количества каолинита, следовые количества кварца, оксиды алюминия и кремния.

Ключевые слова: *гумусовые вещества, глинистые минералы, инфракрасная спектроскопия.*

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Estimation of quality of cleaning of the humus matters by means of FTIR spectroscopy.

The purpose of this work was a study of influence of application of the most widespread and accessible methods of cleaning of humus matters from the admixtures of clay minerals. Small enough sizes and strong adsorption properties have clay minerals, which predetermine education durable organo-mineral colloid systems. Before research there was a task of cleaning of humus preparations from the admixtures of clay minerals. Scientists change the conditions of lead through such operations. As a result got humus preparations which differ in content of admixtures. Subsequent research of such preparations becomes complicated by influence of admixtures. FTIR spectroscopy is successfully used for the detection of content of clay minerals in soils and different functional groups in humus preparations. Three variants of cleaning of humus matters were probed. With the help of the method of FTIR spectroscopy was studied the quality of cleaning of humus matters from admixture by the methods of centrifugation, standing and ionic interchange. It is set that centrifugation on speed of 8000 turns in a minute within 10 minutes, purges humus solution from quartz and diminishes content of clay minerals, however in solution there are groups of Al–O–Si, Si–O–Si, Al–Al–OH and sulfates. Centrifugation and the use of the ionic interchange purge humus matters from clay minerals, sulfates, oxides of aluminum and silicon.

Key words: *humus matters, clay minerals, FTIR spectroscopy.*