Determination of lignosulfonates and humic components in humate fertilizers by ATR-FTIR-spectroscopy

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Currently, there is a problem of controlling the production of fertilizers based on humic substances (HSs) since the properties of the final product depend on the HS source and the characteristics of the technological process. The content of additional components, including lignosulfonates (LSs), is also not controlled. Since humus is formed during the lignin decomposition in nature, the structure and properties of HSs and LSs are similar, and, therefore, the methods for determining each component are the same. However, the functions of LSs and HSs in fertilizers differ; therefore, it is necessary to analyze and control each component individually in preparations based on HSs.

IR spectroscopy is already used to characterize the functional group composition of both HSs and LSs. However, since HSs are present in fertilizers in relatively high concentrations, their solutions have a high absorbance and strongly absorb radiation, thus complicating the use of IR spectroscopy in the transmission mode. FTIR spectroscopy allows the analysis of both solid objects and solutions. Therefore, this work aimed to develop a method for directly determining (1) LSs in individual solutions and (2) HSs and LSs in aqueous solutions of fertilizer preparations based on HS by ATR-IR spectroscopy with minimum sample preparation. Several commercial preparations of HS, LS, and fertilizers were investigated. A Bruker Vertex 70 spectrometer with a diamond-crystal ATR attachment was used.

Model mixtures show that without preliminary sample preparation, only qualitative determination of LS in a mixture with humate at the band of 1266 cm⁻¹ is possible, even with a 4-fold excess of humate. To quantify LS in a mixture with humate, it is necessary to first separate silicate impurities in humate by centrifuging the solutions for 30 min at a rate of 40,000 min⁻¹. Spectral information processing affects the error of determining LS in the mixture. The most accurate results are achieved by processing the band without using a baseline and taking into account the content of the second component in the mixture.

The most accurate determination of LS in the concentration range of 10–100 g/L is possible using 1093 and 1042 cm⁻¹ bands with an error of 0.5% in individual samples and up to 5% in humate mixtures. Determination of humate content in the concentration range of 10–150 g/L by 1570 and 1383 cm⁻¹ bands is possible with an error of up to 10% at HS : LS ratios of 2 : 1 or higher.

Thus, it is possible to simultaneously determine the content of lignosulfonate and humate in their mixtures.