



Non-destructive and quantitative analysis of inorganic ions in soil and sediments employing Diffuse reflectance Fourier transform infrared spectroscopy (DRS-FTIR)

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Soil are key links in the global carbon, nitrogen, phosphorus and sulphur cycles etc. These cycles involve soil chemical processes. Some of the processes include decomposition of organic matter, nitrification, denitrification, phosphorus fixation, sulphide oxidation etc. Thus, soil contains a large number of organic and inorganic components. Sulphate (SO_4^{2-}), nitrate(NO_3^-), phosphate(PO_4^{3-}), silicate(SiO_4^{2-}), carbonate(CO_3^{2-}), ammonium(NH_4^+) are the major inorganic ions in soil and sediments etc. These ions follow the important biogeochemical cycles in the environment for the sustainability of ecosystem. Some of these ions such as SO_4^{2-} and NO_3^- are also major precursors for acid-rain and soil acidification. Importantly therefore estimation of such changing soil ingredients decides the quality and composition of soil, biogeochemical processes in soil, environmental impacts on soils etc. Hence, quantification of all such ions in trace amounts in all environmental segments with very small sample size is of great concern to know their load, pathway, origin etc.

In the present work, DRS-FTIR has been employed as a non-destructive tool for quantitative determination of the important inorganic cationic species viz. NH_4^+ and anionic species viz. SO_4^{2-} , NO_3^- , PO_4^{3-} , SiO_4^{2-} and CO_3^{2-} with KBr matrix as background. The basis of determination of these ions is the selection of non-interfering quantitative vibrational peaks among the various observed peaks for the different symmetry types of the selected multiatomic ionic units. As an example, the peak at 617 cm^{-1} (bending vibration, ν_4) has been selected among the three observed peaks at 617, 983 and

1117 cm^{-1} for SO_4^{2-} determination and the peak at 1385 cm^{-1} (asymmetric stretching vibration, ν_3) has been selected among the four observed signals at 1385, 1050, 833, 719 cm^{-1} for the quantitative determination of NO_3^- due to the simple reason of strong and sharp signals.

The quantitative analysis has been done by preparing calibration curves by obtaining the peak areas for a wide range of concentration at selected peaks for respective ions. Analytical quality assurance (AQA) test has been performed to validate the method. Statistical parameters such as standard deviation, relative standard deviation, F- and t-test have also been done. The limit of detection (LOD) and the limit of quantification (LOQ) have also been calculated for the determination of these selected ions. The feasibility of this method has been tested to the determination in real environmental samples such as soil, sediment and aerosol samples. The method has relatively very high sample throughput value as compared to ion-chromatographic (IC) technique. The method is rapid, free of interferences and non-destructive.