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Salt Removal Using Solid Phase Extraction

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1. Introduction

Samples containing chelating compounds inherently have high salt concentrations in soil waste, and the salt concentrations are further increased when excess metal is added to form metal complexes. Salt peaks can interfere with the analysis by affecting the results, and can act as obstacles to chelating compound analysis by damaging the high performance liquid chromatography (HPLC) column, reducing analysis sensitivity, and causing negative impacts such as peak distortion, equipment contamination, and decreased performance of the mass spectrometer. To solve these problems, a pretreatment process to effectively remove high concentrations of salts prior to analysis is essential. In this study, we aimed to improve the reliability and efficiency of the HPLC analysis process by removing salts components using solid phase extraction (SPE).

2. Methods and Results

2.1 Sample Preparation

In order to study soil similar to real decontaminated soil, Jeju soil spiked with ethylenediaminetetraacetic acid (EDTA) was used. The chelate was extracted from the soil for analysis. The pH was then adjusted above 10 to precipitate pH-sensitive metals such as iron, and an excessive amount of nickel was added so that the chelate would form a complex with nickel. Then, the pH was adjusted to 6.5 and filtered using a 0.2 µm cellulose acetate filter.

2.2 Solid Phase Extraction

Salt peaks can affect the analysis results and act as interference in the analysis of chelating compounds. Therefore, a pretreatment process to remove salts prior to analysis is essential. SPE was used to remove the salts. Aminopropyl cartridges were used. During the conditioning step of injecting a mixed solution of acetonitrile (ACN) and HPLC grade water, the polar stationary phase interacts with the injection solution to become hydrophilic. During the loading step of injecting a mixed solution of ACN and soil supernatant, the stationary phase and nickel-chelate complex are adsorbed through hydrophilic interactions. Then, by injecting low-concentration ammonium hydrogen carbonite during the washing step, the weakly polar salts are removed with the washing solvent, and the highly polar nickel-chelate complex is left on the stationary phase. Finally, during the elution step by injecting high-concentration ammonium hydrogen carbonite, the interaction between the NH_2 cartridge and the nickel-chelate complex is weakened, so that the nickel chelate is eluted along with the mobile phase.

2.3 HPLC condition

Analyses were performed on the HPLC Shimadzu system. The Ni-EDTA was analyzed in gradient elution mode using hydrophilic interaction chromatography (HILIC). The mobile phase consisted of A: 0.1 mol L^{-1} ammonium buffer:ACN=1:9, B: 10 mmol L^{-1} ammonium buffer (pH 8.50 adjusted with ammonia solution). The A:B ratio was increased linearly from 100:0 to 60:40 over 10 minutes, maintained at 60:40 for 5 minutes, and then decreased linearly to 0:100 over 5 minutes. The temperature was maintained at 50℃, the injection volume was set to 10 µL, and the flow rate was 1.0 mL min^{-1}.

2.4 Results and discussion

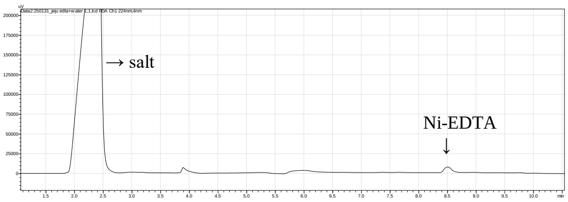

Fig. 1. HPLC measurement of jeju soil supernatant

After leaching the chelate and adding excess nickel, the HPLC measurement results showed a high salt peak at 2 minutes and an EDTA peak at 8.5 minutes (Figure 1).

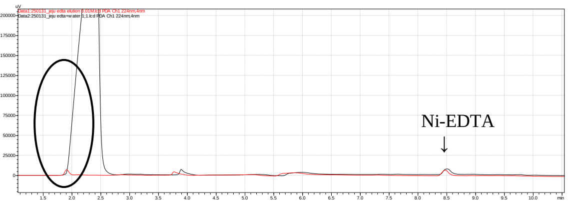

Fig. 2. Comparison of HPLC measurement of Jeju soil supernatant and HPLC measurement after solid phase extraction

Figure 2 shows a comparison of the measurement results of the Jeju soil supernatant and the solution after SPE. The red graph is the HPLC measurement result after SPE. After SPE, the salt peak almost disappears, and the EDTA peak remains.

3. Conclusions

SPE is a suitable technique for separating salt and chelate complexes from samples to increase the purity of the target compound. Through the process, salts were effectively removed, which is expected to extend the life of the analytical equipment, enhance the sensitivity of the results, and improve the peak resolution. These outcomes suggest that SPE can be an effective pretreatment method for chelating agent analysis in soil waste samples.

REFERENCES

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