Simultaneous Separation of Residual EDTA, DTPA, and NTA from Metal Surfaces

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1. Introduction

Ethylenediaminetetraacetic acid (EDTA), diethylenetriaminepentaacetic acid (DTPA), and nitrilotriacetic acid (NTA) are amino carboxylate ligands widely used in radioactive metal decontamination due to their strong metal-binding properties.

Because these ligands readily form stable complexes with transition metals, accurate characterization of residual chelating agents in treated metal waste is essential for assessing the chemical stability of the waste matrix under disposal conditions.

In this study, we developed and comparatively evaluated an high-performance liquid chromatography (HPLC) method for the simultaneous separation of EDTA, DTPA, and NTA under simulated decontamination conditions.

2. Methods and Results

2.1. Sample Preparation

To simulate realistic decontamination conditions, metal samples were treated with EDTA, DTPA, and NTA solutions. The chelating agent solutions were applied to the metal surfaces and dried under an infrared lamp. Two experimental approaches were employed: a metal leaching method and a swipe method.

In the metal leaching method, the dried metal samples were immersed in distilled water to extract the chelating agents. Sodium hydroxide (NaOH) was added to the leachate to adjust the pH, allowing interfering metals such as iron to precipitate. Subsequently, an excess amount of nickel was added to ensure the formation of Ni–chelate complexes.

In the swipe method, the dried metal surfaces were wiped using filter paper, and the used filter paper was then extracted with distilled water. The extract was treated in the same manner as in the metal leaching method: the pH was adjusted to precipitate interfering metals, followed by the addition of excess nickel to form Ni–chelate complexes. The resulting solution was analyzed by HPLC.

2.2. HPLC conditions

HPLC analysis was performed using a Shimadzu HPLC system. Ni–chelate complexes were analyzed under hydrophilic interaction liquid chromatography (HILIC) conditions in gradient elution mode.

Mobile phase A consisted of ammonium buffer and acetonitrile (ACN), adjusted to pH 8.50 with HCl. Mobile phase B consisted of ammonium buffer adjusted to pH 8.50 with aqueous ammonia.

A gradient program was applied by varying the ratio of mobile phases A and B to achieve effective separation of the target Ni–chelate complexes within a 30 min run time.

The temperature was maintained at 50°C. The injection volume was 10 µL, and the flow rate was set at 1.0 mL min^{-1}. Detection was carried out at a wavelength of 224 nm.

2.3. Results and discussion

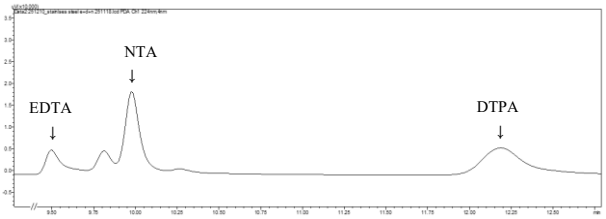

Fig. 1. HPLC chromatogram of Ni-chelate complexes obtained using the metal leaching method.

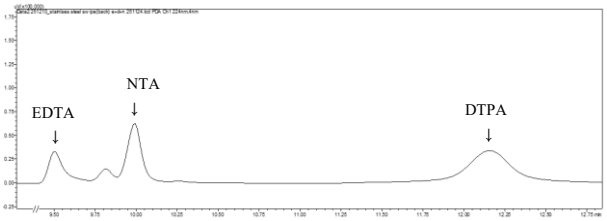

Fig. 2. HPLC chromatogram of Ni-chelate complexes obtained using the swipe method.

The HPLC chromatograms obtained from both the metal leaching and swipe methods showed clear separation of three Ni-chelate complexes corresponding to EDTA, DTPA, and NTA. Distinct and well-resolved peaks were observed under the established HILIC gradient conditions, demonstrating the feasibility of simultaneous separation in metal waste samples.

Although both methods enabled successful separation, differences in peak intensity were observed between the two preparation approaches, suggesting variations in extraction efficiency.

3. Conclusions

An HPLC method was successfully established for the simultaneous separation of EDTA, DTPA, and NTA in metal waste under simulated decontamination conditions. The use of Ni complexation combined with HILIC gradient elution enabled clear and well-resolved separation of structurally similar chelating agents. These findings highlight the effectiveness of the proposed approach for recovery and separation of chelating agents from contaminated metal surfaces.

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