B131 The Development of Magnetic Carbon Nanotubes (Mag-CNT) for Dispersive Solid Phase Extraction (dSPE) of Cyanide Metabolite (2-Aminothiazoline-4-Carboxylic Acid) in Biological Samples

Sun Yi Li, BSc, Sam Houston State University, Huntsville, TX 77341; Jorn Chi-Chung Yu, PhD, Sam Houston State University, Huntsville, TX 77341*

Learning Overview: The purpose of this presentation is to familiarize the audience with the novel method of analyzing cyanide metabolite from biological samples using Magnetic Carbon Nanotubes facilitated Dispersive Solid Phase Extraction (Mag-CNT/d-SPE) coupled with Gas Chromatography/Mass Spectrometry (GC/MS) analysis.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by demonstrating the application of the magnetic carbon Magnetic Carbon Nanotubes facilitated Dispersive Solid Phase Extraction (Mag-CNT/d-SPE) for the extraction of the cyanide metabolite, 2-aminothiazoline-4-carboxylic acid (ATCA), from biological samples. The use of this new method may help to confirm cyanide exposure in forensic analysis.

Approximately 8% of the cyanide exposure cases in 2016 were due to intentional poisoning. The lack of consistent and conclusive autopsy findings increases the difficulty in confirming cyanide exposure. Other than that, due to the reactivity and instability of cyanide, improper storage conditions of the biological samples further increase the unreliability of confirmation cyanide exposure by direct detection of cyanide. An alternative approach to confirm cyanide exposure is to detect its minor metabolite—ATCA. Current studies published to extract ATCA from biological samples mainly focused on the conventional solid phase extraction (SPE) or liquid-liquid extraction (LLE). In this work, the capability of Mag-CNT to extract ATCA from biological samples was investigated.

Magnetic carbon nanotubes (Mag-CNT) were first synthetized in-house. The capability of Mag-CNT to extract ATCA was first tested in water samples spiked with ATCA standard. In a microcentrifuge tube, 2 mg of Mag-CNT was added in 100 µL of deionized water (D.I. water) and 1000 ng/mL of ATCA in triplicates. The samples were acidified with formic acid, vortex, and sonicated to extract for 10 min. The Mag-CNT were isolated with aid of a strong magnet and the supernatants were transferred in separate tubes. Back-extraction was performed on the Mag-CNT with 150 µL D.I. water/ 5% ammonium hydroxide. The Mag-CNT were isolated again, and the back-extract were transferred to separate tubes. An isotopic compound, ATCA - ^{13}C, ^{15}N, was used as the internal standard. The three portions (Mag-CNT, supernatant, back-extract) were dried under vacuum at 65°C and derivatized with N-methyl-N-(trimethylsilyl) trifluoroacetamide (MSTFA) and subjected to gas chromatography/mass spectrometry (GC/MS) analysis. Optimization of the extraction parameters, including extraction time, amount of Mag-CNT, and types of Mag-CNT, was performed in synthetic urine and bovine blood.

The Mag-CNT is found to be capable of extracting ATCA from both biological samples. Average recoveries of ATCA were 97.28% and 99.19% for synthetic urine and bovine blood respectively. The new approach not only has a satisfactory bias and precision within ± 20% at the low, medium, and high concentration levels, but also has a quantitation limit of 30 – 1000 ng/mL that can detect endogenous ATCA level in human urine and blood. The novel methodology in extracting ATCA from biological samples has a potential of forensic application to assist in the confirmation of cyanide exposure and might serve as an alternative method to overcome some limitations associated with the conventional SPE and LLE methods.

Mag-CNT/dSPE, Cyanide Metabolite, ATCA