J1 A Fast Examination of Counterfeit Pharmaceutical Packaging Through Laser-Induced Breakdown Spectroscopy (LIBS) and Attenuated Total Reflectance/Fourier Transform Infrared (ATR/FTIR) Spectroscopy

Emily A. Haase, BA, BS, West Virginia University, Morgantown, WV 26506; Mandy Ho, BS, West Virginia University, Oglebay Hall, Morgantown, WV 26506; Tatiana Trejos, PhD, West Virginia University, Morgantown, WV 26506; Luis E. Arroyo, PhD, West Virginia University, Morgantown, WV 26506-6121*

Learning Overview: The goal of this presentation is to inform attendees about the advantages, feasibility, and preliminary classification ability of LIBS and ATR/FTIR for the purposes of counterfeit pharmaceutical packaging examination.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by introducing a fast, portable, non-invasive, sensitive technique for the examination of counterfeit pharmaceutical packaging. In analyzing counterfeit and authentic pharmaceutical packaging, this technique has shown correct classification rates of over 90% using quadratic discriminant analysis for a variety of ink colors and paperboard substrates. These rates indicate that this technique is an encouraging possibility for the analysis of counterfeit pharmaceutical packaging as well as pre-established visual examination methods.

The global production and sale of counterfeit pharmaceuticals present a substantial health and economic risk, affecting thousands of people and costing billions of dollars every year. While covert and overt anti-counterfeit technologies (such as Radio Frequency Identification [RFID], holograms, and security ink) exist to prevent the accurate replication of authentic pharmaceuticals, their cost considerably prevents widespread implementation. As sophisticated counterfeit pharmaceuticals become more difficult to discriminate from their authentic counterparts by simple visual inspection, there is a need for alternative spectrochemical methods for the identification of counterfeit pharmaceuticals and the associated packaging. These spectrochemical methods should ideally have a fast, accurate, and mobile instrument available for rapid on-site detection. LIBS and ATR/FTIR are quick, sensitive, portable techniques to identify the chemical composition of the packaging and packaging ink. LIBS is an inorganic, quasi-non-destructive technique, where only a very small portion of the sample is ablated from the surface, while ATR/FTIR is entirely non-destructive and detects the organic compounds within the sample. Current methods for the examination of suspected counterfeit pharmaceuticals involve the analysis of the Active Pharmaceutical Ingredient (API) and of the pharmaceutical packaging. The investigation of the packaging itself, as opposed to the pharmaceutical, is particularly important in cases of bulk suspected counterfeit pharmaceuticals in which the pharmaceuticals are missing, or when only the packaging has been altered.

In this study, over 150 counterfeit and authentic pharmaceutical packaging samples were analyzed using LIBS and ATR/FTIR. Seven different colors of ink (brown, n=21; pink, n=45; green, n=31; red, n=46; yellow, n=74; blue, n=92; and black, n=122) and the paperboard material of the packaging (n=147) were analyzed. Each sample was run with eight replicates in different locations to account for variation within the sample; duplicate control samples analyzed on different days were used to assess inter-day and intra-day variations; and samples from the same lot or printed with the same ink/paper/printer system were measured to assess the intra-lot variability. The study included the analysis of packaging from common pharmaceutical products, including hormones, antibiotics, and analgesics. Six machine learning and statistical methods (k-nearest neighbors, neural network, naïve Bayes, random forest, quadratic discriminant analysis, and linear discriminant analysis) were used to evaluate the methods' capabilities to differentiate between the authentic and counterfeit sample sources and to correctly classify the samples. Fifty percent of the samples were used to train the machine learning classification algorithm; the other half were used to test the classification algorithm. For combined LIBS and ATR/FTIR, the successful classification rate within same-color datasets was over 70% for all techniques and over 90% for the quadratic discriminant analysis technique. These correct classification rates indicate that a combined LIBS and ATR/FTIR technique is auspicious for the non-invasive, rapid (few minutes), on-site examination of pharmaceutical packaging.

Counterfeit Pharmaceuticals, LIBS, ATR/FTIR