B89 A Quantitative Comparison and Differentiation of Smokeless Powders

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Learning Overview: After attending this presentation, attendees will better understand the strengths and limitations of automated image analysis and GC/MS profiling for sample differentiation, comparison, and potential brand identification of commercially available smokeless powders.

Impact on the Forensic Science Community: This research will impact the forensic science community by demonstrating value of utilizing multiple techniques for the characterization and comparison of smokeless powders that can potentially be recovered from inefficient and/or undetonated improvised explosive devices.

Small arms propellants (SAP) are readily accessible and cost-effective materials that firearms enthusiasts can acquire for the legitimate assembly of ammunition. Unfortunately, the ease of access to and low cost of these materials is advantageous for their utilization in the construction of improvised explosive devices (IEDs). Typically, the SAP charge is loaded into a metal pipe (commonly steel) and sealed with screw-fit end caps. Two recent high profile domestic terrorist attacks using IEDs (Boston Marathon Bombing and NY/NJ attempted bombings) demonstrate their continued usage. Thus, there is a need to develop robust metrics for the characterization of propellants that are used as explosives as well as for comparisons between exemplar and recovered explosive residues.

The goals of the presented research are to investigate the utility of high-throughput, low cost quantitative automated image analysis and GC/MS additive of SAP for sample discrimination and potential brand identification. Ninety 1-pound canisters of SAP were purchased from local firearms stores. The samples are a wide selection of different distributors (n=8) and product brands (n=90). In addition, the sample set also has a diverse distribution of general particle morphologies (e.g., ball, disk, tube, flattened ball, etc). The first phase of the project was focused on method development for non-destructive automated image analysis. For this phase, individual smokeless powder particles were arranged on one-inch squares of clear mounting tape so that no particles were touching or overlapping. The sample preparations were then placed on a lightbox and photographed using a consumer-grade DSLR camera. Approximately 120-1600 grains/ sample were imaged. Linear calibration was performed using a NIST-certified image analysis micrometer. In addition, NIST-certified black polymer spheres of the target diameters: 100 µm, 500 µm, 1.5 mm and 3.00 mm were used as secondary standards to evaluate the linear calibration. Images were processed using FIJI, an open-source image analysis package. The following parameters were measured for each particle ($n \approx 34{,}000$ particles *in toto*): area, perimeter, major/ minor ellipse axis, roundness, circularity, and solidity.

The large matrix of morphometric data was analyzed using the open-source statistical package R. To classify the 90 brands, 80% of the data was randomly chosen and used as a "training" set for linear discriminant analysis (LDA). The remaining 20% of the data set was treated as unknowns and "matched" to the source (brand) with the smallest Mahalanobis distance.

The second phase of this research was to assess the utility of GC/MS additive profiling. Aliquots of 20 mg for each sample were extracted with 2 mL of 3:1 methanol: n-butanol, based on the method described by Reardon and MacCrehan.[1] This method was chosen because it preferentially extracts the additives, leaving behind the bulk nitrocellulose. Samples were run using an HP5890 series II gas chromatograph equipped with an HP 5972 MSD and an RTX-1 column (30m x 0.25mm ID x 0.25µm). The samples were run under the following conditions: injection port temperature: 175°C; flow: 1 mL/min He; 10:1 split ratio; GC oven: 100°C (hold 3 minutes); 10°C/min to 250°C; 250°C (hold 5 minutes); total run time was 23 minutes. The identified additives were primarily nitroglycerin (NG), ethyl centralite (EC), diphenylamine (DPA), and dibutyl phthalate (DBP). Of the 90 analyzed powders, 77% were double base (NG containing). All Winchester and Alliant powders in this study were double base. Seventy-seven% of the samples contained DPA, and 76% contained EC. All powders had at least two additives present at detectable levels. The preliminary results show that EC was found in products from IMR, Hodgdon, Winchester, Alliant, and Vihtvuori. 2,6-dinitrotolune was identified in products only from Hodgdon and IMR. It was noted that many samples gave the same GC/MS additive profiles but had different powder morphologies; demonstrating the need for the fusion of data from different analytical methods for more robust sample differentiation and comparison. A two-tiered method for combining both data sets (morphology and additive profiling) will be presented. The results from this study are very encouraging but will require more samples to obtain a more robust assessment.

Reference(s):

1. Reardon, M.; MacCrehan, W. Developing a quantitative extraction technique for determining the organic additives in smokeless handgun powder. *J Forensic Sci* 2001:46(4):802-807.

Smokeless Powder, Improvised Explosive Device, Image Analysis