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TECHNICAL NOTE

Bulk Explosive Investigation: Chemical Analyses of Fuel-Oxidizer Explosive Mixtures Commonly Used in Criminal Actions

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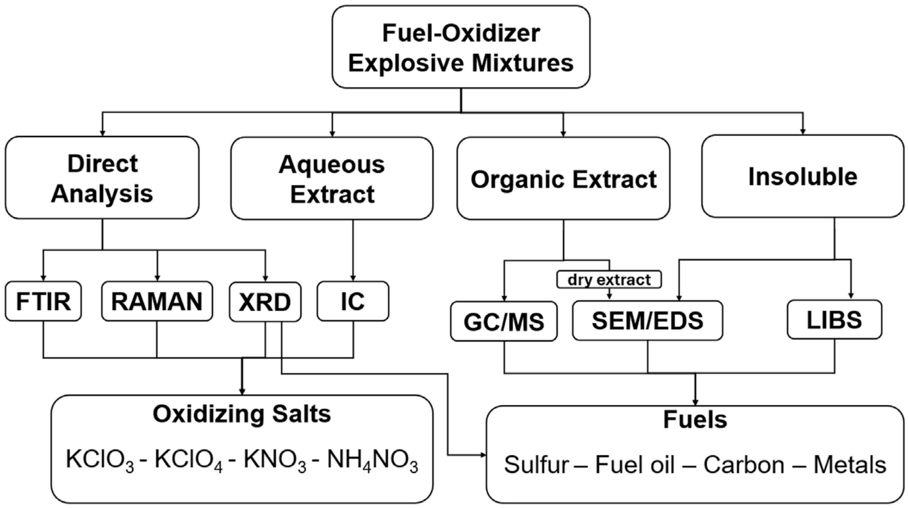

Explosives are widely utilized across various legal activities, including military operations, law enforcement, mining, and construction. Unfortunately, they are also involved in illegal acts such as terrorism, robbery and vandalism. The analysis of explosives and post-explosion residues plays a crucial role in forensic chemistry, aiding investigations into explosive-related crimes. This analysis aims to identify the explosive involved in criminal action. It aids in determining the cause of the explosion, assessing potential clandestine facilities, establishing authorship, and identifying trends for criminal purposes. One type of explosive widely used, not only in Brazil but also worldwide, is fuel-oxidizer mixtures, such as black powder, explosive emulsions, and mixtures based on chlorate and/or perchlorate salts. These explosives are composed of substances with significantly different chemical natures. As a result, their complete identification generally requires the application of various analytical techniques. For this purpose, in addition to direct analyses of the mixtures, they often involve procedures for separating their components and analyzing each of them using appropriate analytical techniques. This separation is typically achieved through simple solvent extractions, often using water and organic solvents. The aqueous and organic extracts, along with any insoluble fractions, are then analyzed, as outlined in this study. This work presents a simple workflow for sample preparation and analysis of bulk fuel-oxidizer explosive mixtures, which are frequently encountered in criminal activities globally, including in Brazil. The workflow employs various analytical techniques, including Gas Chromatography-Mass Spectrometry (GC-MS), Fourier Transform Infrared Spectroscopy (FTIR), Raman Spectroscopy, Ion Chromatography (IC), Laser-Induced Breakdown Spectroscopy (LIBS), Scanning Electron Microscopy with

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Energy-Dispersive X-ray Spectroscopy (SEM/EDS), and X-ray Diffraction (XRD). This procedure provides a practical guide for forensic laboratories, enhancing their ability to analyze commonly encountered explosive samples with precision and reliability.

Keywords: explosives, FTIR, RAMAN, GC-MS, Ion Chromatography, LIBS, SEM/EDS, XRD

INTRODUCTION

Explosives are legally used in various activities, including but not limited to military and police operations, mining, oil exploration, demolitions, highway and railway construction, fireworks displays, industrial applications (such as in the aeronautical and automotive sectors), welding, and more. Conversely, the unlawful use of explosives is a prevalent issue, with examples including terrorism, extortion, vandalism, illegal fishing, break-ins, and robberies (such as bank heists or thefts involving the transport of valuables).[1]

In forensic chemistry, the examination of explosives and post-explosion residues is highly important and assists in investigating numerous crimes involving explosives. The main objective of such analysis is the identification of the explosive involved in the crime. Depending on the case, this information can help clarify the cause of the explosion, identify the origin of the explosive, indicate whether clandestine production or storage of explosives is involved, and determine authorship. Furthermore, by gathering information from multiple cases over time, it is possible to identify the most commonly used explosives for specific criminal activities and detect emerging trends.[2,3]

Explosives can be classified in various ways, including their chemical nature, reaction speed, sensitivity, and applications. One particularly interesting differentiation, based on the chemical nature, is the division into two groups: i) individual explosive molecules, typically composed of organic substances such as nitroesters, nitramines, nitroaromatics, nitroaliphatics, and peroxides; and ii) fuel-oxidizer explosive mixtures, which consist of inorganic salts (such as nitrates, chlorates, and perchlorates), serving as oxidants, combined with different fuels (such as fuel oil, vaseline, sulfur, carbon, powdered metals (Al, Mg), sugars, etc.).[2–5] This classification is particularly relevant for chemical analysis purposes, as the chosen methodology for conducting the analysis largely depends on it.[5]

According to a previous study conducted in Brazil, it was revealed that most crimes involving the use of explosives in Brazil involve Improvised Explosive Devices (IEDs) based on fuel-oxidizer mixtures, such as black powder, mixtures containing chlorate and/or perchlorate salts, and explosive emulsions.[3] As fuel-oxidizer explosives consist of mixtures composed of substances with significantly different chemical natures, they often require the application of various analytical techniques to achieve their complete identification. For this purpose, in addition to direct analysis of the mixtures, they often involve procedures for separating their components and analyzing each of them using appropriate analytical techniques. This separation is typically performed using straightforward solvent extractions, commonly involving water and organic solvents. The resulting aqueous and organic phases, along with any insoluble fractions, are then analyzed.[2]

Among the various analytical techniques used for this purpose, Gas Chromatography-Mass Spectrometry (GC-MS),[6–10] Fourier Transform Infrared Spectroscopy (FTIR),[6,11–14] Raman spectroscopy,[14–23] Ion Chromatography (IC),[10,24–31] Laser-Induced Breakdown Spectroscopy (LIBS),[32,33] Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (SEM/EDS),[14,34] and X-ray Diffraction (XRD)[35–38] stand out. These instrumental techniques are based on different chemical principles. GC-MS separates and identifies volatile compounds, including hydrocarbons and a wide range of other substances. FTIR and Raman spectroscopy provide structural insights by analyzing molecular vibrations, facilitating the identification of functional groups in organic and inorganic compounds. Ion chromatography (IC) is a powerful technique for detecting ionic species, including oxidizing salts, in aqueous solutions. LIBS and SEM-EDS are techniques for elemental analysis: LIBS employs laser-induced plasma to detect metals and other elements in solid samples, while SEM-EDS combines electron microscopy with X-ray spectroscopy to identify elements. Lastly, XRD analyzes the crystalline structure of materials, aiding in the identification of crystalline compounds, such as oxidizing salts, within mixtures.

In post-explosion scenarios, where bulk particles are generally absent, common challenges such as limited sample quantities and low analyte concentrations make chemical analyses more complicated due to heightened concerns about potential interferences from various sources, including Explosive Ordnance Disposal (EOD) operations,[2,39] matrices,[40–42] environmental factors,[2] and sampling, collection, and processing materials.[30,43] Moreover, the analytical techniques applicable in these cases are often limited to chromatographic methods due to the characteristics of the samples. Conversely, in cases involving bulk explosive mixtures, sample quantity and analyte concentration are typically not a concern, enabling the use of a wide range of analytical techniques to identify all components of interest. In fact, it is almost always necessary to use more than one technique, as a single method is generally insufficient to identify all components of a fuel-oxidizer mixture due to the specific detection limitations of each, as previously mentioned. Combining multiple techniques, therefore, ensures a more robust and comprehensive analysis.

Even though explosive analysis has been carried out for many years in various forensic chemistry laboratories, particularly in Brazil, its non-routine nature in several forensic units still raises questions among many professionals who occasionally need to handle such cases. Therefore, the objective of this study is to provide a workflow for sample preparation and analysis of bulk fuel-oxidizer explosive mixtures using different analytical techniques, including GC-MS, FTIR, Raman spectroscopy, IC, LIBS, SEM/EDS, and XRD.

MATERIALS AND METHODS

Reagents and materials

Five fuel-oxidizer explosive mixtures were obtained from Improvised Explosive Devices (IEDs) seized by the Brazilian Federal Police. These mixtures included flash powder, explosive emulsion, black powder, $KClO_3$/sulfur, and $KClO_3$/sulfur/starch combinations. The GC-MS analyses were performed using extractions prepared with n-hexane (99%) and dichloromethane (P.A.), both sourced from Sciavicco® Comércio Indústria LTDA. The results were compared with a n-alkanes (C_7-C_{40}) reference standard, at a concentration of 1000 mg L^{-1}, obtained from Sigma-Aldrich®. Ultrapure water (18.2 MΩ cm at 25 °C) was supplied by a Millipore Direct-Q5 purification system, while Grade 5 helium was provided by White Martins Gases Industriais Ltd. For sample filtration, a hydrophilic PTFE syringe filter (25 mm, 0.45 µm) from Nova Analítica Imp. Exp. Ltda. was used for aqueous extractions, and a Nylon filter (25 mm, 0.45 µm) from Agilent Tech. Brasil LTDA. was employed for organic solvent extractions.

Sample preparation

For the FTIR, Raman, and XRD analyses, the samples were analyzed directly without any preparation. For the analysis using the other techniques, the preparation steps for each of the explosive mixtures are detailed below.

Flash powder

A 100 mg sample of the flash powder was subjected to extraction in 5 mL of water by vortexing for 1 min, followed by centrifugation at 4000 rpm for 5 min. This process was repeated three times, with each extraction followed by centrifugation. The first extract was reserved for IC analysis, while the remaining two extracts were discarded. The fraction reserved for IC analysis was diluted (1:1000) in the same solvent (water), filtered through a 0.45 µm filter, and transferred into an appropriate IC vial. The insoluble fraction obtained after this procedure was then reserved for analysis by LIBS and SEM/EDS.

Black powder

A 100 mg sample of the black powder was subjected to extraction in 5 mL of dichloromethane by vortexing for 1 min, followed by centrifugation at 4000 rpm for 5 min. The extract was then filtered through a 0.45 µm filter and dispensed into a suitable GC vial. The resulting insoluble material underwent the same extraction procedure three times, this time using water as the solvent. The first aqueous extract was further diluted (1:1000) in the same solvent (water), while the remaining two extracts were discarded. This diluted extract was filtered through a 0.45 μm filter and dispensed into a suitable IC vial. The insoluble fraction obtained after this procedure, along with the dichloromethane extract dried at room temperature, was then set aside for analysis by LIBS and SEM/EDS.

Explosive emulsion

A 100 mg sample of the explosive emulsion was subjected to extraction in 5 mL of n-hexane by vortexing for 1 min, followed by centrifugation at 4000 rpm for 5 min. The extract was then filtered through a 0.45 μm filter and dispensed into a suitable GC vial. The resulting insoluble material underwent the same extraction procedure, this time using water as the solvent. The aqueous extract was further diluted (1:1000) in the same solvent (water), filtered through a 0.45 μm filter, and dispensed into a suitable IC vial.

$KClO_3$/sulfur and $KClO_3$/sulfur/starch

A 100 mg sample of each mixture ($KClO_3$/sulfur and $KClO_3$/sulfur/starch) was subjected to extraction in 5 mL of dichloromethane by vortexing for 1 min, followed by centrifugation at 4000 rpm for 5 min. The extracts were then filtered through a 0.45 μm filter and dispensed into suitable GC vials. The resulting insoluble materials underwent the same procedure using water as the solvent. The aqueous extracts were further diluted (1:1000) in the same solvent (water), filtered through a 0.45 μm filter, and dispensed into suitable IC vials.

Flowchart

Figure 1 presents a flowchart providing a general overview of the sample preparation procedures and the subsequent analytical techniques used.

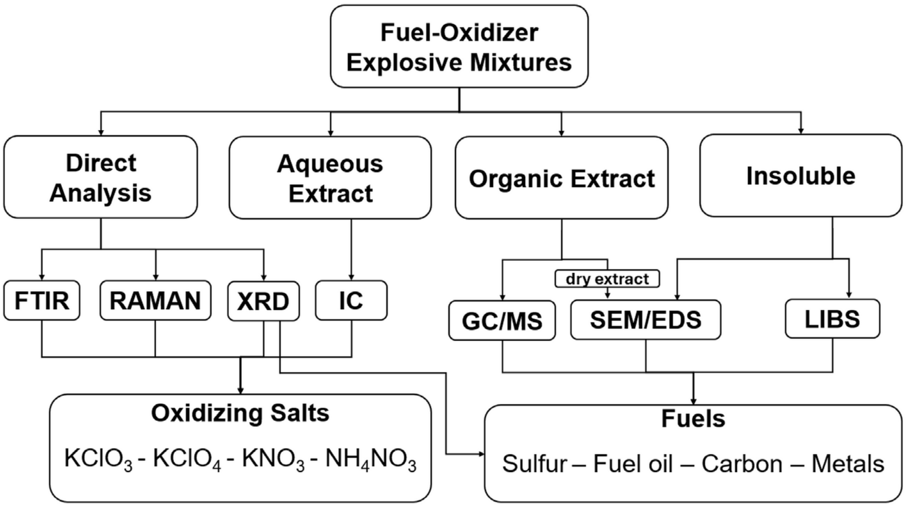

Figure 1. Flowchart illustrating the sample preparation procedures and the chemical analysis techniques employed.

Instrumentation

Gas chromatography-mass spectrometry (GC-MS)

The chromatographic analysis was carried out on an Agilent 6890N gas chromatograph using an Agilent 5973 mass spectrometer detector and Agilent 7683B autosampler. The conditions were as follows: injection volume: 0.2 μL; Split Ratio: 50:1; column: RXi-5MS methyl siloxane, 30 m × 250 μm (i.d.) × 0.25 μm film thickness; oven temperature program: initial: 150 °C, 40 °C min^{-1} to 315 °C, 315 °C for 4.87 min (sulfur analysis) and 14.87 min (fuel oil analysis); injection port temperature: 280 °C; carrier: 0.8 mL min^{-1}

(helium). Data was processed using Agilent GC-MSD ChemStation software (version 17). The mass spectra of all analytes were compared with their correspondence spectra of the NIST 17 MS Database (Agilent Technologies), using MS Search Program version 2.3.

Fourier transform infrared (FTIR)

FTIR analyses were conducted using a Thermo Scientific Nicolet iS10 FTIR Spectrometer, equipped with a diamond ATR (attenuated total reflectance) accessory with one or multiple reflections, and a DTGS (Deuterated TriGlycine Sulfate) detector. The instrument was operated at room temperature with a spectral range from 4000 to 400 cm^{-1}, a resolution of 4 cm^{-1}, and a scan number of 16 scans per sample. Spectra acquisition and analyses were carried out using the OMNIC 8.1.0.10 software (Thermo Fisher Scientific, Waltham, MA, USA).

Ion chromatography-conductivity detector (IC-CD)

The chromatographic analysis was performed using a Thermo ICS-5000 system with a self-regenerating electrolytic suppression and autosampler accessory. Cation separation was carried out using a Dionex Ion Pac™ CS12A column (2x250 mm) using isocratic conditions with 20 mM methane sulfonic acid (MSA) eluent, automatic generated from reagent-grade water applying an MSA cartridge. Anion separation was achieved with a Dionex Ion Pac™ AS19 column (2x250 mm) and an eluent generator (EGC) set with a potassium hydroxide (KOH) cartridge. The equipment operated under a multi-step gradient, beginning at 10 mM and increasing to 45 mM by 40 min. Both methods utilized a 10 µL injection loop and a constant flow rate of 0.25 mL/min, while for the cation and anion separations the suppressor current was set to 15 and 28 mA, respectively. The column temperature was kept at 30 °C, and the detector operated at 25 °C for both methods. Instrument control and data collection were managed through Chromeleon® software.

Raman spectroscopy

Raman analyses were conducted using a HORIBA XploRATM Plus confocal Raman microscope, with a laser excitation wavelength of 785 nm and a spectral range of 200-3500 cm^{-1}. The system used a 20X objective lens (NA = 0.4) and the analyses were performed with a laser power of 15 mW. The data were processed and analyzed using LabSpec 6 software (HORIBA Scientific, Edison, NJ, USA).

Laser-induced breakdown spectroscopy (LIBS)

LIBS analyses were performed using a Leica DM6 LIBS system, equipped with a Leica 20X magnification objective and a 337 nm nitrogen laser. The laser pulse duration was approximately 5 ns, and the energy per pulse was set to 15 mJ. The analyses were conducted at room temperature, with a laser repetition rate of 10 Hz. The data were processed with AtomAnalyzer software (LEICA Microsystems, Wetzlar, Germany).

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS)

Analyses were conducted using a scanning electron microscope from FEI, model QUANTA 200 3D (FEI Company, Hillsboro, OR, USA), equipped with an Energy Dispersive Spectrometer (EDS) for X-ray analyses. The microscope was operated at 15 kV with a working distance of 10 mm, and the EDS analysis was conducted with an energy resolution of 127 eV. The data were processed with INCA energy software (Oxford Instruments, Abingdon, UK).

X-ray diffractometry (XRD)

X-ray diffraction experiments were performed using a Bruker AXS D8 Discover X-ray diffractometer (Bruker Corporation, Billerica, MA, USA) with CuKα radiation (40 kV, 40 mA). The diffractometer was equipped with a General Area Detector Diffraction System (GADDS) and a step size of 0.02° 2θ, with a scan rate of 0.5°/min. The data was processed using EVA software (Bruker Corporation, Billerica, MA, USA).

Additional safety comments

It is important to highlight that the analysis of bulk explosives requires additional safety measures beyond the standard laboratory protocols followed by chemical professionals. This is due to the fact that certain explosives exhibit high sensitivity to shock, friction, and temperature. Therefore, analysts involved in this field should ideally possess training not only in chemistry but also in explosives, such as through EOD (Explosive Ordnance Disposal) courses. This specialized training enables them to gain a deeper understanding of different types of explosives, their mechanisms, and common EOD procedures. By having this comprehensive knowledge, analysts can effectively handle and analyze explosives while ensuring the highest level of safety throughout the process.

Some examples of basic precautions when working with materials suspected to be explosive include: i) always work with minimal amounts of material; ii) avoid using tools that could generate sparks when handling materials; iii) exercise increased caution with analytical techniques that involve heating, pressure, and/or friction of the sample. For instance, techniques like FTIR-ATR and XRD often involve pressure during sample preparation, while RAMAN spectroscopy can initiate certain types of fuel-oxidizers through heating.

In the specific case of Raman analysis, which generally involves higher risks of activation, particularly for dark-colored explosives, some equipment provides additional precautions to prevent accidents, such as utilizing the “Delay” function, reducing laser power, using samples in uncapped vials, and maintaining a safe distance when activating the laser.

Another interesting precaution, when feasible, is to commence the analysis with less risky analytical techniques to determine the explosive being dealt with, and only subsequently employ techniques that may pose greater risks.

The procedures outlined in this work aim not only to assist analysts in identifying various fuel-oxidizer mixtures but also to enhance safety during sample preparation and analysis. The isolated components that constitute each mixture do not pose explosion risks during preparation and analysis, unlike the direct analysis of these materials. By facilitating the separation of these components, the procedures described in this work enable safer analyses. Isolating the solid fractions mitigates risks associated with friction, shock, pressure, and temperature, while the extracts prepared for analysis present no risks of explosion. These measures contribute to a safer working environment, ensuring both the accuracy and efficiency of fuel-oxidizer mixture analysis.

RESULTS AND DISCUSSION

The primary objective of each procedure described in the “Sample Preparation” section, as well as the subsequent analyses discussed in the following subsections, is to identify the constituents of the explosive samples used in this study. These procedures follow protocols established by the Forensic Chemistry Laboratory of the National Institute of Criminalistics of the Brazilian Federal Police, which holds ISO 17025 certification, encompassing explosives and post-explosion residue analysis within its scope.

Table I provides information on the main constituents of those fuel-oxidizer explosive mixtures, namely flash powder, black powder, explosive emulsion, $KClO_3$/sulfur, and $KClO_3$/sulfur/starch.

Table I. Fuel-oxidizer explosive mixtures used in this work and their composition

Fuel-oxidizer explosive mixtures		
Oxidizer	Fuel	Generic Name
Potassium perchlorate	Aluminum	Flash Powder
Potassium nitrate	Sulfur/Carbon	Black Powder
Ammonium nitrate	Fuel oil	Explosive Emulsion

(continued on next page)

Table I. Fuel-oxidizer explosive mixtures used in this work and their composition (continuation)

Fuel-oxidizer explosive mixtures		
Oxidizer	Fuel	Generic Name
Potassium chlorate	Sulfur	------
Potassium chlorate	Starch/Sulfur	------

The results obtained for each of the fuel-oxidizer explosive mixtures studied through each of the analytical techniques are discussed separately below.

Flash powder

Regarding flash powder, the primary analytes/components are potassium chlorate and metallic aluminum. Regarding the identification of the oxidizing salt $KClO_4$, the main recommended techniques are FTIR and IC. Figures 2A and 2B show the results obtained for the samples selected for this study. Concerning metallic aluminum, the fuel component of flash powder, SEM/EDS, and LIBS are two techniques that allow for a conclusive analysis. Figures 2C and 2D show some characteristic results.

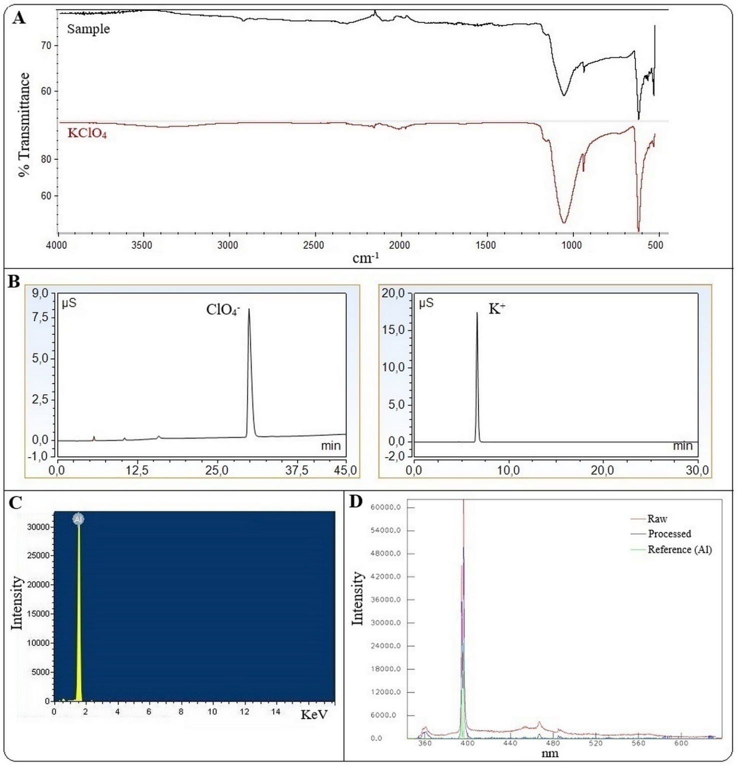

Figure 2. Results of the flash powder analysis. Identification of $KClO_4$ by FTIR (A) and IC (B), in addition to the identification of aluminum by SEM/EDS (C) and LIBS (D).

As seen in Figure 2A, the infrared spectrum of the sample is relatively simple, with few characteristic bands for the analyte, which is also common for several other oxidizing salts used in fuel-oxidizer explosive mixtures. However, since the second components in flash powders are metals, such as aluminum and magnesium in powder form, the identification of the salt remains possible even in direct mixture analyses.

This is because these metals do not generate signals in FTIR, making the technique highly effective. FTIR is an excellent first-choice method: it is safe, simple, quick, and provides valuable insights that can guide subsequent analyses.

The main bands of the analyzed flash powder sample are primarily associated with $KClO_4$, observed at 567 cm^{-1}, 940 cm^{-1}, and 1058 cm^{-1}. The confirmation of $KClO_4$ identity is achieved through an independent analysis by IC, with the identification of the corresponding ions K^+ and ClO_4^- (Figure 2B). The identification of aluminum by SEM/EDS and LIBS can be observed in Figures 2C and 2D, with more prominent signals, respectively, at 1.486 KeV and 394 nm. In this study, SEM/EDS and LIBS were used for elemental analysis; however, other techniques such as FAAS, ICP-OES, and ICP-MS can also be employed for this purpose. The selected techniques offer several advantages, including the ability to analyze solid samples directly without the need for dissolution, high spatial resolution, rapid multi-elemental detection, and a less invasive approach. These features make SEM/EDS and LIBS particularly suitable for analyzing fuel-oxidizer mixtures containing powdered metals and/or nonmetals like sulfur and carbon in their compositions.

Black powder

In the context of confirming the identity of black powder, the following components must be present: potassium nitrate, sulfur, and carbon powder. Therefore, for the identification of the oxidizing salt KNO_3, FTIR and IC techniques are again the primary choices, as illustrated in the results depicted in Figures 3A and 3B. Regarding the fuel components, sulfur, and carbon powder, the techniques predominantly employed are GC-MS and SEM/EDS (Figures 3C and 3D).

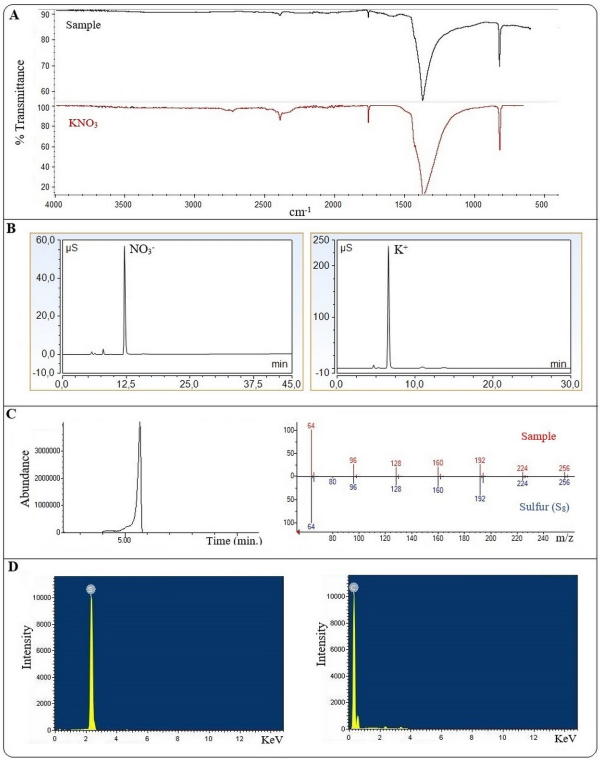

Figure 3. Results of the black powder analysis. Identification of KNO_3 by FTIR (A) and IC (B), in addition to the identification of sulfur by GC-MS (C), and sulfur and carbon by SEM/EDS (D).

As depicted in Figure 3A, once again, the infrared spectrum is simple, not presenting many bands. However, it remains a highly useful technique for guiding subsequent analyses. Since both carbon and sulfur do not generate significant signals via FTIR, the main bands of the analyzed black powder sample are primarily associated with KNO_3, observed at 824 cm^{-1}, 1373 cm^{-1}, 1762 cm^{-1}, and 2395 cm^{-1}. The identification of KNO_3 was confirmed by IC, which identified the corresponding ions K^+ and NO_3^- (Figure 3B). In GC-MS, the *m/z* signals at 256, 224, 192, 160, 128, 96, and 64 from the main GC peak are indicative of elemental sulfur (Figure 3C). The identification of the fuel components, sulfur, and carbon, by EDS can be observed in Figure 3D, with more prominent signals, respectively, at 2.307 KeV and 0.277 KeV.

Explosive emulsion

In the case of explosive emulsion, the relevant constituents for identification are ammonium nitrate and fuel oil. Hence, to discern the oxidizing salt NH_4NO_3, FTIR, RAMAN, and IC techniques can offer results for a conclusive decision, as showcased in the results portrayed in Figures 4A, 4B, and 4C. As for the fuel elements, predominantly comprised of a mixture of n-alkanes (C_{20} to C_{34}) forming fuel oil, GC-MS techniques were employed (Figure 4D).

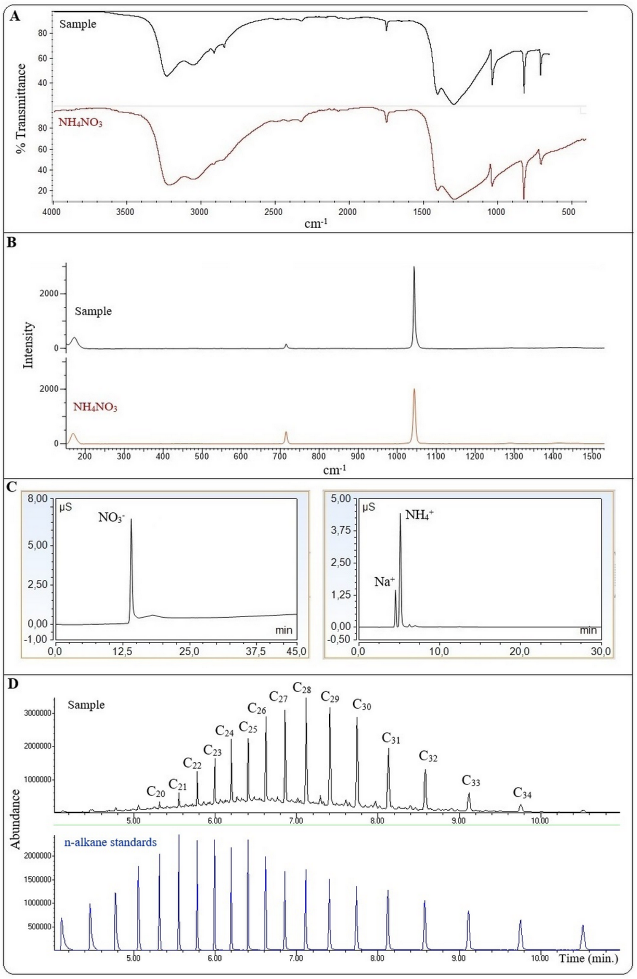

Figure 4. Results of the emulsion explosive analysis. Identification of NH_4NO_3 by FTIR (A), RAMAN (B) and IC (C), in addition to the identification of mixture of n-alkanes (C_{20} to C_{34}) forming fuel oil by GC-MS (D).

As depicted in Figure 4A, once again, the infrared spectrum essentially contributes to the identification of the oxidizing salt, NH_4NO_3, with the main bands observed at 714 cm^{-1}, 826 cm^{-1}, 1041 cm^{-1}, 1301 cm^{-1}, 1409 cm^{-1}, 1754 cm^{-1}, 3061 cm^{-1}, and 3234 cm^{-1}. Additionally, some less intense bands related to the organic phase of fuel oil can also be observed at 2849 cm^{-1} and 2917 cm^{-1}, which is consistent with fuel oil concentrations generally ranging from 3% to 6%. The identification of NH_4NO_3 was confirmed by Raman with the characteristic bands at 1044 cm^{-1} and 712 cm^{-1} (Figure 4B) and by IC, which identified the corresponding ions NH_4^+ and NO_3^- (Figure 4C). The analysis of the organic fraction containing the fuel component is depicted in Figure 4D, where predominantly n-alkanes peaks (C_{20} to C_{34}) can be observed, confirmed by mass spectrum and comparison with n-alkane standards. The fuel profile presented here is the most commonly observed based on cases analyzed in crimes occurring in Brazil; however, other fuels could theoretically be used as well.

$KClO_3$/sulfur

In the context of the $KClO_3$/sulfur mixture, the target components are obviously potassium chlorate and sulfur. Thus, for the identification of the oxidizing component $KClO_3$, FTIR and IC techniques were once again employed, as illustrated in the results depicted in Figures 5A and 5B. Concerning the fuel component, sulfur, the techniques utilized were once more GC-MS (Figure 5C).

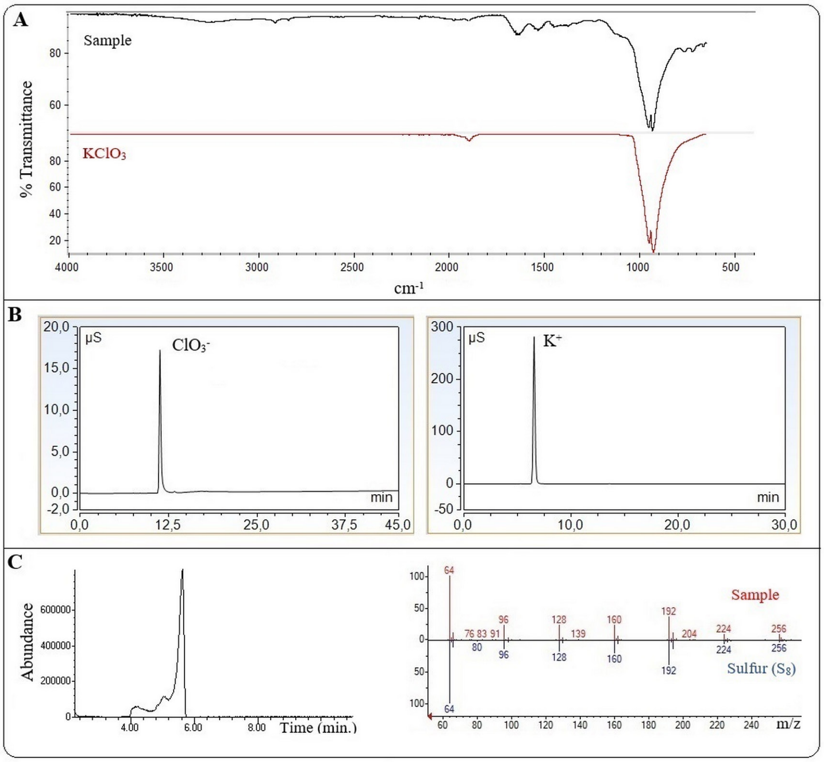

Figure 5. Results of the $KClO_3$/sulfur mixture analysis. Identification of $KClO_3$ by FTIR (A) and IC (B), in addition to the identification of sulfur by GC-MS (C).

As depicted in Figure 5A, the FTIR spectrum is again relatively simple, with only a few characteristic bands, and since sulfur does not generate significant signals, the few bands essentially correspond to $KClO_3$, with the two main bands at 937 cm^{-1} and 956 cm^{-1}. The identification of $KClO_3$ was confirmed by IC, which identified the corresponding ions K^+ and ClO_3^- (Figure 5B). The identification of the fuel component (Figure 5C), sulfur, is essentially the same as shown for black powder.

$KClO_3$/sulfur/starch

A second case involving a different type of $KClO_3$/sulfur mixture is presented below, this time with an additional fuel component: starch. As mentioned earlier, there is a wide variety of fuel components that can be used in the manufacture of explosives. Various carbohydrates can serve this purpose, with starch being one example, and it has the advantage of being readily available in any supermarket. The identification of $KClO_3$ by IC and the identification of sulfur by GC-MS will not be shown here, as they are essentially the same as in the previous case. In this instance, the presented results were only those related to the direct analysis by FTIR, in addition to the iodine-starch test and XRD (Figure 6A-B).

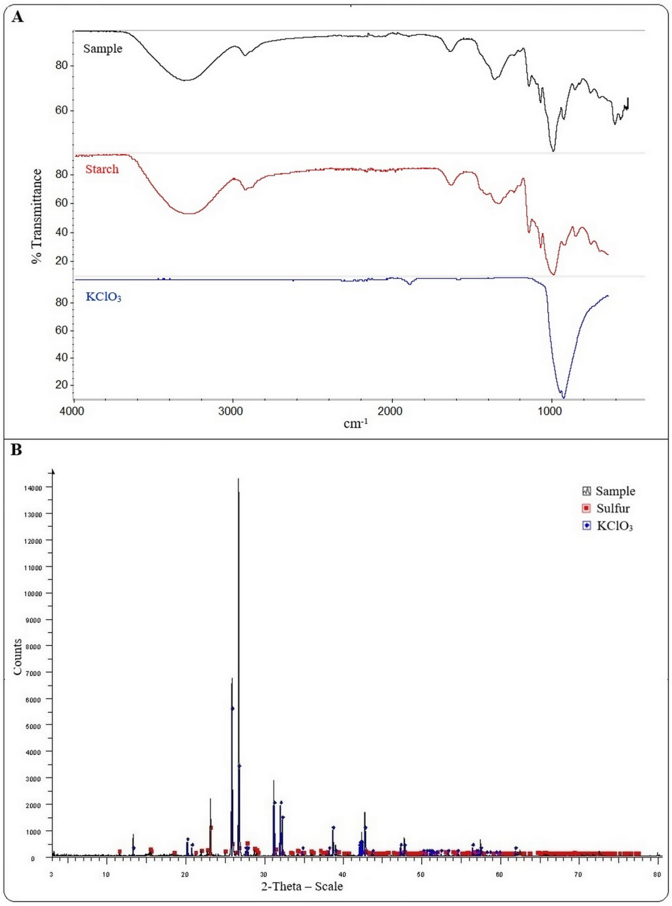

Figure 6. Results of the analysis of an explosive mixture based on potassium chlorate, sulfur, and starch. Comparison of FTIR spectra among the sample, starch standard, and $KClO_3$ standard (A), and identification of $KClO_3$ and sulfur by XRD (B).

Note that the FTIR analysis (Figure 6A) identifies only one component of the mixture, which is starch, with the main bands observed at 861 cm^{-1}, 930 cm^{-1}, 996 cm^{-1}, 1076 cm^{-1}, 1149 cm^{-1}, 1366 cm^{-1}, 1644 cm^{-1}, 2929 cm^{-1}, and 3312 cm^{-1}. The signals of starch are prominent compared to the signals of potassium chlorate, causing the latter to be masked. It's important to note that sulfur, which is another component, does not exhibit significant signals in FTIR analysis and therefore cannot be identified using this technique. The presence of starch was confirmed via the iodine-starch test. This result underscores the importance of using confirmatory techniques in specific cases. The composition of these mixtures can vary greatly, and relying solely on a single analytical technique may not suffice to accurately identify all the components. Additional techniques are required to ensure comprehensive identification and analysis of the mixture.

In this instance, XRD analysis was employed, and its results were compared with available database diffractograms, confirming the presence of $KClO_3$ and sulfur (Figure 6B).

Table II provides an overview of the potential analytes that can be identified using the analytical techniques applied to the types of explosive mixtures analyzed in this study. It is important to note that some techniques may be employed redundantly for confirmation purposes. However, it should be emphasized that the use of all techniques is not always necessary to reach a robust and reliable conclusion, as this often depends on the analytical infrastructure available in each laboratory. In fact, in this study, which is based on real cases, only a subset of the techniques was applied to each case. The summary of the results, presented and discussed in this technical note, is highlighted in italics in Table II.

Table II. Summary of potential analytes identified by the analytical techniques discussed in this study (results presented and discussed in this technical note are highlighted in italics)

Explosives	**Analytical technique**						
	FTIR	**Raman**	**GC-MS**	**LIBS**	**SEM-EDS**	**XRD**	**IC**
Flash Powder	$\mathit{KClO_4}$	$\mathrm{KClO_4}$	------	*Al*	*Al*	$\mathrm{KClO_4}$	$\mathit{K^+, ClO_4^-}$
Black Powder	$\mathit{KNO_3}$	$\mathrm{KNO_3}$	*sulfur*	sulfur	*sulfur, carbon*	$\mathrm{KNO_3}$/sulfur	$\mathit{K^+, NO_3^-}$
Explosive Emulsion	$\mathit{NH_4NO_3}$	$\mathit{NH_4NO_3}$	*n-alkanes*	------	------	$\mathrm{NH_4NO_3}$	$\mathit{NH_4^+, NO_3^-}$
$\mathrm{KClO_3}$/Sulfur	$\mathit{KClO_3}$	$\mathrm{KClO_3}$	*sulfur*	sulfur	sulfur	$\mathrm{KClO_3}$/sulfur	$\mathit{K^+, ClO_3^-}$
$\mathrm{KClO_3}$/ Starch/ Sulfur	$\mathrm{KClO_3}$*/starch*	$\mathrm{KClO_3}$	*sulfur*	sulfur	sulfur	$\mathit{KClO_3}$*/sulfur/*starch	$\mathrm{K^+, ClO_3^-}$

Additionally, regarding the solvents used in the extractions, it is important to clarify certain points. Water was the obvious choice for extracting oxidizing salts. For the extraction of elemental sulfur, dichloromethane was employed; however, other solvents have also been reported in the literature for various applications, including cyclohexane, carbon disulfide, and toluene, among others.[44–49] For extracting the fuel fraction from the explosive emulsion, n-hexane was utilized, though alternative solvents such as diethyl ether and dichloromethane are also viable alternatives.[2,10,43,50]

Although this work specifically addresses five fuel-oxidizer mixtures, the procedures presented are applicable to a wider variety of explosive mixtures since oxidizing salts and fuels are generally interchangeable, allowing for many other fuel-oxidizer compositions. Additionally, they can also be used for mixtures with other metals, such as magnesium, as well as other hydrocarbons, like paraffin, kerosene, and motor oil.[51–53] Table III shows some other examples of mixtures, to which the procedures presented here can be applied.

Table III. Some other examples of fuel-oxidizer explosive mixtures

Fuel-oxidizer explosive mixtures		
Oxidizer	**Fuel**	**Generic name**
Ammonium nitrate	Aluminum	ANAL
Ammonium nitrate	Carbon powder	------
Ammonium nitrate	Fuel oil	ANFO
Ammonium nitrate	Magnesium powder	------

(continued on next page)

Table III. Some other examples of fuel-oxidizer explosive mixtures (continuation)

Fuel-oxidizer explosive mixtures		
Oxidizer	**Fuel**	**Generic name**
Potassium chlorate	Aluminum	Flash powder
Potassium chlorate or perchlorate	Aluminum/Sulfur	Flash powder
Potassium chlorate or perchlorate	Paraffin	------
Potassium chlorate or perchlorate	Petroleum jelly	------

It's worth noting that the sets of techniques used in the examples presented in this work are applicable to pre-explosion analysis cases. They are also applicable for post-explosion analysis cases with the presence of bulk particles, which is less common but occurs in some cases in post-explosions of fuel-oxidizer mixtures. In cases of post-explosion without the presence of bulk particles, the techniques are more limited, generally to chromatography and capillary electrophoresis techniques, as these techniques allow the separation of the ions of interest and their identification with low detection limits.[42,54] Therefore, post-explosion residue analyses present some additional analytical challenges due to several factors, such as i) the scarcity of material; ii) the various possible matrices; and iii) the presence of environmental interference.[2,3,30,42]

CONCLUSIONS

In this study, we present a comprehensive workflow for sample preparation and analysis of bulk fuel-oxidizer explosive mixtures using various analytical techniques, including GC-MS, FTIR, Raman spectroscopy, IC, LIBS, SEM/EDS, and XRD. We provide the results of analyses from five fuel-oxidizer explosive mixtures obtained from real-world IED cases, commonly encountered in criminal activities both in Brazil and globally. Through these examples, we illustrate the application of different analytical techniques for each explosive mixture, enabling the identification of their individual components.

The study demonstrates simple yet effective procedures for separating the components of these mixtures, allowing for the unambiguous identification of each substance. Although the techniques presented are not exhaustive, they offer a practical approach for sample preparation and analysis in forensic investigations of fuel-oxidizer mixtures. The research emphasizes the potential of using straightforward solvent extractions to isolate components before applying the appropriate analytical methods. The outlined procedures offer a streamlined workflow for forensic laboratories, helping them identify and assess explosive mixtures with greater efficiency. This study contributes to the forensic science field by providing an accessible and reliable guide for analyzing commonly encountered explosive mixtures used in illicit activities.

Conflicts of interest

The authors declare no conflicts of interest.

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