

PROFILING OF INORGANIC IONS IN SELECTED POST-BLAST
PYROTECHNIC RESIDUES

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To my beloved father and mother

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ABSTRACT

Pyrotechnic homemade explosives have emerged as a new threat to society. Analysis of explosive residues is useful in identification of explosives and establishing link to the perpetrators. The objectives of this study were to detect the selected inorganic ions in firework samples followed by the determination of chemical profiles. Six different types of firework samples were analyzed in this study. Inorganic ions of pre-blast and post-blast samples were analyzed using ion chromatography with conductivity detection. All targeted anions (F^- , Cl^- , NO_2^- , NO_3^- , ClO_3^- , SO_4^{2-} , PO_4^{3-} and SCN^-) were successfully separated within 42 minutes using Metrosep A SUPP 5 column and $NaHCO_3$ - Na_2CO_3 as the eluent. Calibration graph of targeted anions with good linearity ($r^2 > 0.9796$) were obtained with detection limits within range from 16 to 120 $\mu g/L$. Besides that, all targeted cations (Li^+ , Na^+ , NH_4^+ , K^+ , Ca^{2+} , Mg^{2+}) were well separated within 24 minutes using Metrosep C 4 employing nitric acid-dipicolinic acid as the eluent. Good linearity ($r^2 > 0.9948$) of calibration graph was obtained and detection limits was 31-171 $\mu g/L$. Anions (Cl^- , NO_2^- , NO_3^- , ClO_3^- and SO_4^{2-}) and cations (Na^+ , NH_4^+ , K^+ , Ca^{2+} , Mg^{2+}) were detected in pre-blast firework samples. Concentration of NO_3^- , ClO_3^- and K^+ were the highest among detected ions in pre-blast samples. Magnesium ion which is a common element in fireworks was detected in all samples. In post-blast residues, targeted ions presence in pre-blast samples can still be detected but in lower concentration. There was significant reduction in NO_3^- and ClO_3^- concentration in post-blast residues. Concentrations of SO_4^{2-} are relatively higher compared to other anions detected in post-blast residues. Identification of fireworks cannot be based solely on chemical profile of post-blast residues as it is not fully accurate. However, presence of Ca^{2+} and Mg^{2+} in post-blast residues strongly suggest the used of pyrotechnic fireworks.

ABSTRAK

Bahan letupan piroteknik buatan sendiri telah timbul sebagai ancaman yang baru kepada masyarakat. Analisis sisa-sisa letupan adalah berguna dalam mengenalpastian jenis bahan letupan serta menentukan suspek yang terlibat. Tujuan kajian ini adalah untuk mengesan kehadiran anion dan kation tertentu yang terdapat dalam sampel bunga api diikuti dengan penentuan profil kimianya. Sebanyak enam jenis bunga api telah dianalisis dalam kajian ini. Ion-ion tak organik dalam sampel sebelum dan selepas letupan telah dianalisis dengan menggunakan ion kromatografi dengan pengesanan konduktiviti. Semua anion yang terpilih (F^- , Cl^- , NO_2^- , NO_3^- , ClO_3^- , SO_4^{2-} , PO_4^{3-} dan SCN^-) telah berjaya diasingkan dalam masa 42 minit dengan menggunakan turus Metrosep A SUPP 5. Eluen yang digunakan ialah campuran $NaHCO_3$ dan Na_2CO_3 . Graf penentukuran setiap ion mencapai garis lurus yang bagus ($r^2 > 0.9796$) dan had pengesanan adalah dalam lingkungan 16-120 $\mu g/L$. Selain itu, kation (Na^+ , NH_4^+ , K^+ , Ca^{2+} , Mg^{2+}) telah diasingkan dalam masa 24 minit dengan menggunakan Metrosep C 4, eluen nitric asid-dipikolinik asid. Garis lurus setiap ion dalam graf penentukuran mencapai $r^2 > 0.9948$ dan had pengesanan ialah 31-171 $\mu g/L$. Anion (Cl^- , NO_2^- , NO_3^- , ClO_3^- and SO_4^{2-}) dan kation (Na^+ , NH_4^+ , K^+ , Ca^{2+} , Mg^{2+}) telah dikesan dalam sampel sebelum letupan. Kepekatan ion NO_3^- , ClO_3^- , and K^+ adalah yang tertinggi di antara ion-ion yang dikesan dalam sampel bunga api sebelum letupan. Ion Mg^{2+} yang biasa terdapat dalam bunga api telah dikesan dalam semua sampel. Dalam analisis sisa-sisa letupan, ion-ion yang dikesan adalah sama seperti sampel sebelum letupan tetapi dengan kuantiti yang kurang dari asal. Kepekatan NO_3^- dan ClO_3^- berkurang dengan ketara dalam sisa letupan. Kepekatan relatif SO_4^{2-} adalah lebih tinggi berbanding dengan anion yang lain. Pengenalpastian jenis bahan letupan berdasarkan profil kimia sisa-sisa letupan sahaja adalah tidak tepat. Bagaimanapun, kehadiran Ca^{2+} dan Mg^{2+} dalam sisa letupan telah mencadangkan penggunaan piroteknik bunga api.

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LIST OF ABBREVIATIONS

ATM	-	Automated Teller Machine
DDW	-	Distilled deionised water
CE	-	Capillary Electrophoresis
C-4	-	Composition-4
FRDM	-	Fire and Rescue Department of Malaysia
GC	-	Gas chromatography
HPLC	-	High performance liquid chromatography
HMX	-	High-velocity military explosive
IC	-	Ion chromatography
IMS	-	Ion Mobility Spectroscopy
ND	-	Not detected
MS	-	Mass spectrometer
MYR	-	Malaysia Ringgit
PDA	-	Photodiode array
PETN	-	Pentaerythritol tetranitrate
RDX	-	Research Department Explosive
RMP	-	Royal Malaysia Police

RSD	-	Relative standard deviation
SD	-	Standard deviation
SEM-EDA	-	Scanning electron microscope-Energy dispersive x-ray analyzer
TATP	-	Triacetone triperoxide
TEA	-	Thermal energy analyzer
TLC	-	Thin layer chromatography
TNT	-	Trinitrotoluene
UV	-	Ultraviolet
VAAR	-	Vinyl acetate alcohol resin

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Pyrotechnics mixed explosives are often used in making homemade explosives. It emerges as a potential threat to public security as pyrotechnics such as fireworks can be easily purchased either legally or illegally. In recent years, improvised explosives mixed with pyrotechnic have been used by terrorists to increase the damage of explosion. Increasing cases involving pyrotechnic mixed homemade explosives in international and local have raised the awareness of law enforcers to study pyrotechnics components in depth (Ahmad *et al.*, 2011). According to Law of Malaysia Explosive Act 1958, explosive substance include any materials for making any explosive substance and any bomb, grenade, apparatus, machine, implement, or material used or intended to be used or adapted for causing or aiding in causing any explosion in or with any explosive substance and any part of such bomb, grenade, apparatus, machine or implement.

On 2nd of May 2010, a car bomb was found near the Times Square in New York. The car bomb was loaded with fireworks, fuel and fertilizer. The bomb was successfully defused and later a Pakistani suspect was arrested for investigation (The Star, 2010).

On 17th of April 2011, a bomb was detonated at a Nigeria hotel and wounded eight people during the presidential election. The bomb was later identified containing celebratory fireworks (The Star, 2011).

Homemade explosives cases have increased in Malaysia in recent years. In 2008, at Shah Alam and Puchong, three cases where explosives made from fireworks were used to blow up Automated Teller Machines (ATMs). Although the explosion caused damage to the machines, the thieves failed to get the cash. Among three attempts, only one successfully got away with MYR 30000 (The Star, 2008).

On 29th August 2010, a deadly firework explosive packed in a parcel was placed in front of an apartment unit in Desa Tun Razak, Cheras. When a woman opened the parcel, it detonated. The woman was killed and another co-worker was badly injured. The explosive was identified as mixture of cannonball fireworks with kerosene (The Star, 2010).

Another case that happened on 9th January 2012, involved three homemade explosives that exploded in front of Jalan Duta court complex during an assembly by Datuk Seri Anwar Ibrahim's supporters. Four members of the public were injured while vehicles parked nearby suffered minor damage. The explosive was suspected of containing pyrotechnics and black powder (The Star, 2012).

1.2 Statement of Problem

Pyrotechnic composition is often used in homemade explosives. The lack of an explosive database hinders Royal Malaysian Police (RMP) when investigating explosion cases. RMP often faced difficulties when it comes to linking the explosives to its origin. Therefore, a research to develop explosive database is essential in order to fill the missing link for RMP investigation.

1.3 Objectives

This study embarks in the following objectives;

- i. To detect the presence of anions and cations in pre and post blast pyrotechnic fireworks samples.
- ii. To determine the chemical profile of pyrotechnic fireworks samples.

1.4 Scope of Study

This study focuses on the analysis of the constituent of pyrotechnic from pre blast and post blast source. Profiling of the pyrotechnic samples is based on the detecting of anions (F^- , Cl^- , NO_2^- , NO_3^- , ClO_3^- , SO_4^{2-} , PO_4^{3-} , SCN^-) and cations (Li^+ , Na^+ , NH_4^+ , K^+ , Ca^{2+} , Mg^{2+}) studied using ion chromatography. Common inorganic explosives such as nitrate, chlorate, sulfate, ammonium, and potassium are part of the study.

1.5 Significance of Study

This study can be used to help Royal Malaysia Police (RMP) and Malaysian Fire and Rescue Department (FRDM) in determining the type of explosives used in a bombing attack. Analysis of the explosive residues will provide valuable information regarding the substances that been used in making the explosives. This will eventually establish a link between the explosive and its perpetrators.

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