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Research Article

Determination of elemental ratios correlating to the projectiles of conventional and non-toxic ammunitions

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Abstract
'Non-toxic' ammunitions are unveiled to achieve comparable ballistic performance to conventional ammunition while simultaneously mitigating the risk of hazardous heavy metals. Previous studies greatly emphasized the analysis of gunshot residues released by these ammunitions; however, the elemental profiles of their respective projectile cores were less explored. Therefore, this study aimed to determine the elemental ratios correlating to the projectiles of both conventional and non-toxic ammunitions. In this study, three projectiles from eight ammunition types were collected, acid-digested, and analysed to detect antimony, arsenic, bismuth, cadmium, copper, iron, lead, silver, tin, and zinc through inductively coupled plasma-mass spectrometry. Projectiles from Geco 9 mm and ARX 9 mm ammunitions were detected with low levels of lead but higher copper content, differentiating them from those conventional projectiles where lead was the major composition. The varying copper-zinc ratios also suggested the jacketed projectiles. Subsequently, the principal component analysis applied to the relative elemental abundance successfully formed six clusters, allowing for discrimination. To conclude, elemental ratios correlated to the type of projectiles can be useful when compared with the projectiles found on the scene, perhaps to link to the residues on an impact mark made by any projectile.

Keywords: Projectile, ammunition, ICP-MS, element, discrimination

Introduction

Gun violence is frequently associated with violent crimes worldwide, particularly robbery and murder. In Malaysia, according to the Department of Statistics, a total of 30 cases of gang robbery with firearms were reported in 2023, which increased by nearly 100% compared to 2022. Armed robbery also demonstrated a similar trend, recording an increment from 7 to 13 cases since 2022. Apart from that, murder cases, although the involvement of firearms was not disclosed, were reported at high prevalence, with 258 cases in one year [1]. The statistics revealed the seriousness of firearm-related cases, and therefore, the greatest investigative efforts are required to recover and examine every potentially analysable trace in a firing scene.

Based on the respective external and functional characteristics, various types of ammunitions are available in the markets. Their weight, diameter, length, number of plain or knurled cannelures, nose type (such as full jacketed or hollow point), and core type (hard or softcore) have been used for classification, while the primer composition is more common. However, there is no classification based on the compositional makeup of the projectile [2,3]. In recent times, firearm manufacturers have unveiled new types of ammunition, often labelled as 'non-toxic,' 'eco-friendly,' 'heavy-metal-free,' or 'lead-free.' These non-toxic ammunitions employ various 'non-toxic' chemical compounds or materials to propel projectiles, aiming to achieve comparable performance to conventional ammunition while simultaneously mitigating the risk of exposure to hazardous heavy metals like lead, barium, and antimony for both the shooter and the environment [4]. Previously published articles related to the examination of non-toxic ammunition have undoubtedly provided significant contributions to forensic investigation. Early in the 1990s, Gunaratnam [5] demonstrated the presence of zinc and titanium in the inorganic residue particles released by Sintox lead-free ammunition, suggesting its uniqueness from another source of ammunition and the environment. Apart from that, aluminium, calcium, copper, iron, potassium, silicon, sodium, strontium, and tin were also detected, depending on the lead-free or non-toxic ammunition [6]. Chang et al. [7] also reported the varying experimental composition of non-toxic ammunitions, highlighting the absence of three commonly detected elements in conventional ammunitions, which were lead, barium, and antimony. Among conventional ammunitions, the presence of lead is the key composition, and therefore, eliminating lead in the design of non-toxic ammunition makes detection more difficult. However, it was essential to emphasize that the primer composition contributed such elemental compositions of gunshot residues (GSR) but not made up of the projectiles.

In relation to the composition of projectiles, Udey et al. [8] differentiated two types of projectiles, non-jacketed and jacketed, by comparing the concentrations of lead, copper, and antimony using inductively coupled plasma-mass spectrometry (ICP-MS). Note that a non-jacketed projectile is made entirely of lead or lead alloys. In contrast, the jacketed projectile is the projectile with the lead core encased in a layer of metal, typically copper or copper alloys. It was shown that the ability of ICP-MS to detect multiple elements simultaneously makes it a powerful analytical technique. In another study, the presence of lead, tin, copper, and zinc originating from the cores of various projectiles was also detected through inductively coupled plasma-optical emission spectrometry [9]. Newland et al. [10] reported the detection of both lead and antimony in the ten ammunitions under investigation using atomic absorption spectroscopy. Apart from that, the varying ratios of these elements, ranging from 6:1 to 43:1, had allowed for the discrimination of projectiles, which could be further applied to the metal deposits on the impact marks. Literature suggests that lead making up the core of most projectiles is the key marker during forensic examination. However, to the authors' knowledge, with emphasis on the primer composition, the composition of the projectile originating from non-toxic ammunition is not reported. Therefore, its emergence has required firearm examiners to characterise and interpret the output from their analyses, whether a shooting was caused by a specific firearm with certain ammunition, particularly through the comparison with an impact mark.

Previous studies suggested the possibility of elemental profiles from the varying projectiles for discrimination, particularly when they were made up of different compositions by the same or different manufacturers. However, exploration of the elemental profiles of non-toxic ammunitions, specifically their respective projectile cores, is lacking and deserves investigation. Information regarding the composition of a projectile, both released from conventional or non-toxic ammunitions, is crucial to determine the cause of an impact mark on any surface. For instance, an impact mark could be found at a shooting scene, and its cause shall be determined. Therefore, the current study would aid in determining if the mark was caused by a projectile and even track back to the possible source of ammunition that made an impact. Examination of the GSR did not allow for such determination during the forensic investigation; therefore, the elemental profiles of projectiles deserve exploration to keep pace with the current developments in ammunition.

Materials and Methods

Chemicals and reagents

Ultrapure reagent grade nitric acid (HNO_3, 65%, w/w) and hydrogen peroxide (H_2O_2, 30%, v/v) were purchased from Thermo Fisher Scientific (Waltham, MA, USA). Ultrapure water (18.2 MΩ) was prepared in-house from a Millipore water purification system (Bedford, NY, USA). A multi-element standard containing 25 elements was obtained from Agilent Technologies (Whitehouse Station, MA, USA). The standard contained five major elements [calcium (Ca), iron (Fe), magnesium (Mg), potassium (K), sodium (Na)], each at 1000 µg/mL, and twenty minor elements [aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), manganese (Mn), molybdenum (Mo), nickel (Ni), selenium (Se), silver (Ag), thallium (Tl), thorium (Th), uranium (U), vanadium (V), zinc (Zn)], each at 10 µg/mL.

Ammunition samples

In this study, two general types of ammunitions were tested, namely ammunition of 9 mm and .38 calibres. Among the eight ammunition types, three were of .38 calibre (i-iii), and another five were 9 mm ammunition (iv-viii). All the ammunitions were supplied by the Royal Malaysia Police as follows:

i. Winchester .38 Special (Full Metal Jacket (FMJ), United States)
ii. CBC .38 Special (Lead Round Nose (LRN), Brazil)
iii. SME .38 Special (LRN, Malaysia)
iv. SME 9 mm (FMJ, Malaysia)
v. Geco 9 mm Luger Hexagon (Ruag Ammotec, Germany)
vi. Inceptor-Polycase 9 mm (Advance Rotation Extreme (ARX), United States)

vii. Ruag Ammotec 9 mm (Jacketed Deform Projectile (JDP), Switzerland)
viii. Sellier & Bellot 9 × 19 (FMJ, Czech Republic)

Two calibres were chosen due to their more common prevalence in forensic firearm cases. Note that the ammunitions of Geco 9 mm Luger Hexagon, Inceptor-Polycase 9 mm, and Ruag Ammotec 9 mm appeared not alike with the conventional ammunitions (**Figure 1**). In contrast, Geco 9 mm ammunitions were labelled as lead-free ammunition by the manufacturers.

Collection of projectiles from live ammunition

The projectile was removed from ammunition using a bullet puller and collected into 10 mL clear glass headspace vials (Supelco, Bellefonte, PA, USA). Since the cartridge case and smokeless powders were excluded from this study, they were separately collected into other vials. Three projectiles were collected for each type of ammunition.

Acid digestion of projectiles

A self-developed acid digestion was carried out to extract the target elements into solution form. Projectiles collected from each ammunition were firstly cleaned with a cotton swab moistened with methanol and subsequently placed in a 25 mL beaker. In the fume hood, 6 mL of HNO_3 and 2 mL of H_2O_2 were added into the beaker, followed by gentle heating on a stirring hotplate controlled at 100°C with the speed adjusted at 500 rpm. The heating procedure was monitored carefully to avoid the acid from being wholly dried off, with acid solutions added when necessary. Upon completion of the acid-digestion process with complete dissolution of the projectile, the digested sample was transferred to a 10 mL volumetric flask and topped up with deionised water to the mark. The sample was then filtered using a regenerated nylon syringe filter of 0.45 µm pore size (Corning Inc., NY, USA), followed by the elemental determination. A reagent blank was also prepared according to the above procedure.

Elemental profile determination

In this study, an ICP-MS instrument (iCAP Triple Quadrupole ICP-MS, Thermo Fisher Scientific, Waltham, MA, USA) with an integrated autosampler was used. It was operated with an integral peristaltic pump for sample uptake, along with a spray chamber and nebuliser to generate fine aerosol. Each digested sample was aspirated into the ICP-MS system in triplicate. The presence of the target element in each sample was determined, and its composition was evaluated to investigate the relative abundance within a single sample.

Data analysis

To characterise the tested projectiles, principal component analysis (PCA) was carried out based on the relative abundance of detected elements for each sample using Minitab 21 software (Minitab Inc., State College, PA, USA). Any potential grouping or discrimination of projectiles according to the source of ammunition was assessed from the PCA score plot.

Results and Discussion

Elemental profiling of projectiles

In this study, seven target elements were priorly set for elemental determination as suggested by FBI Laboratory [11], namely arsenic, antimony, bismuth, cadmium, copper, silver, and tin. Previously, lead was not analysed when weighing bullet-related evidence due to the common lead core in most ammunition [12]. However, it was unclear whether lead was absent or present, especially for those non-toxic ammunitions, and therefore, it was included for detection. Apart from that, zinc was added to the list for comparison, especially with its usage in jacketing a projectile together with copper. Iron was also included due to the

(a) (b) (c)

Figure 1. Physical appearance of (a) Geco 9 mm Luger Hexagon, (b) Inceptor-Polycase 9 mm, and (c) Ruag Ammotec 9 mm ammunitions.

literature reporting the manufacture of steel-made projectile cores in certain newly developed ammunition [13]. The ten elements were assessed, and the detection of respective elements in each sample was based on their concentration levels above the background equivalent concentration (BEC) values. Note that the BEC values referred to the blank value in concentration units, obtained by dividing the signal generated upon aspiration of a blank against the slope of the calibration curve of a target element. **Table 1** shows the relative abundance in percentage of each target element for the eight tested projectiles in this study, each with three projectiles.

The elemental profiles of each projectile were determined, firstly by detecting the presence of an element, followed by the division of each signal to the total signals of all detected elements. Taking the Winchester .38 Special as an example, six elements were detected in its projectile, including antimony, bismuth, copper, lead, silver, and zinc. Lead was found to dominate the composition of a projectile, occupying 99.3% of the total composition. Other elements appeared as minor compositions, in which the silver only contributed to less than 0.01% of the projectile.

Through the simultaneous detection by ICP-MS, lead was the primary composition that made up a projectile in most tested ammunitions. All three projectiles extracted from ammunitions of .38 calibre were detected with high percentages of lead, generally greater than 98.9%. It continued to be the key composition used to make the core of a projectile, either with or without alloy, particularly for conventional ammunition. Antimony was the second most abundant element detected from these projectiles, ranging from 0.28% to 0.92%. The presence of antimony could have suggested an alloy combination consisting of lead and antimony in manufacturing the projectile [13]. Out of the three bullets fired from .38 ammunitions, two of them, i.e. CBC .38 Special and SME .38 Special, have made up the full lead bullets, lacking any jacket or coating on each projectile. Therefore, their composition of copper and zinc was detected at relatively lower levels compared to the projectile retrieved from the Winchester .38 Special.

Concerning the 9 mm calibre ammunitions, the three jacketed projectiles (i.e., SME, Ruag Ammotec, and S&B) showed the presence of lead at a percentage of more than 95%, respectively. Such a full metal jacket (FMJ) type is common ammunition in which a metal jacket covers the lead or lead-alloy core. As the projectiles used in small arms pistols could have been made of a lead-antimony alloy, the higher percentage of lead in jacketed bullets is common. In addition to lead, the antimony incorporated in the lead-antimony alloy was also detected, ranging from 0.2% to 0.5%, depending on the manufacturers. The lead core could be strengthened by the addition of alloys, commonly antimony. Additionally, a small amount of antimony in the bullet could increase its hardness [12,14]. Detection of both copper and zinc could also suggest jacketed projectiles. The primary function of a jacket is to provide a protective layer to the lead core, providing structural reinforcement to the projectile. By minimising the deformation or fragmentation upon impact, the jacket could enhance a projectile's ability to penetrate targets and maximise its lethality [3,15].

In this study, both copper and zinc were detected from the composition of all tested projectiles. However, it was important to note that the relative proportion of copper-zinc could be varied. For instance, a ratio of approximately 2:1 for copper-zinc was demonstrated in the profiles of Ruag AmmoTec 9 mm projectile; however, the proportion of copper is 200 times higher than zinc in profiles of projectiles originating from SME 9 mm and S&B 9 × 19 ammunitions. This could suggest that the Ruag AmmoTec had jacketed the bullet with soft brass while the other two were made up of copper-plated jackets. Ruag AmmoTec is a type of projectile that incorporates a jacket surrounding its core and forms a jacketed deformation projectile. Still, it does not appear like FMJ, in which the whole core is fully jacketed. Such a design allows for controlled deformation through expansion or fragmentation, creating larger impact holes [16,17]. On the other hand, the very low percentage of iron, either at a level less than 0.002% or undetectable, suggested that the core of the projectile could have been made up of lead and not steel [13].

Comparatively, two types of projectiles were detected with lead at lower levels: Geco 9 mm ammunitions at a percentage of less than 1.5% and ARX at less than 0.01%. These two ammunitions carried projectiles with a high percentage of copper but low lead, which differed from other ammunition, both full-lead and full-jacketed projectiles. Besides copper, zinc was also detected at a higher level. ARX is made of an injection-molded polymer-copper matrix to provide adequate toughness for an all-purpose defense round [18,19]. Its elemental profile was found to be varied from conventional ammunition, with a significant absence of lead and antimony, making up nearly 99.9%

Table 1. The mean elemental percentages of target elements in eight tested projectiles

Element	Elemental Percentage (%)							
	Winchester .38 Special	**CBC .38 Special**	**SME .38 Special**	**SME 9 mm**	**GECO 9 mm**	**ARX 9 mm**	**Ruag 9 mm**	**S&B 9 × 19**
Antimony	0.640 ± 0.042	0.481 ± 0.033	0.921 ± 0.056	0.498 ± 0.021	0.008 ± 0.001	0.002 ± 0.0001	0.305 ± 0.042	0.216 ± 0.016
Arsenic	ND	0.005 ± 0.001	0.001 ± 0.0002	0.001 ± 0.0001	ND	ND	ND	0.004 ± 0.0006
Bismuth	0.025 ± 0.003	0.040 ± 0.003	0.062 ± 0.002	0.038 ± 0.004	NA	0.001 ± 0.0002	0.002 ± 0.0004	0.033 ± 0.003
Cadmium	ND	ND	ND	ND	ND	ND	ND	ND
Copper	0.068 ± 0.009	0.013 ± 0.001	0.024 ± 0.002	2.104 ± 0.698	93.844 ± 1.767	99.933 ± 0.054	2.962 ± 0.338	2.178 ± 0.297
Iron	ND	ND	ND	0.002 ± 0.042	ND	0.001 ± 0.042	ND	ND
Lead	99.247 ± 0.575	99.445 ± 0.065	98.982 ± 0.608	97.340 ± 1.175	1.455 ± 0.042	0.029 ± 0.002	95.084 ± 1.032	97.557 ± 0.842
Silver	0.003 ± 0.001	0.001 ± 0.0003	0.006 ± 0.001	0.004 ± 0.0002	0.008 ± 0.001	0.004 ± 0.0002	0.232 ± 0.011	0.002 ± 0.0002
Tin	ND	0.005 ± 0.0003	ND	ND	ND	0.006 ± 0.0002	ND	0.003 ± 0.0004
Zinc	0.016 ± 0.002	0.010 ± 0.003	0.004 ± 0.0004	0.012 ± 0.0002	4.686 ± 0.442	0.024 ± 0.001	1.415 ± 0.102	0.007 ± 0.001

ND: Not detected

of copper. On the other hand, Geco ammunition is engineered with the desired precision and accuracy [20,21]. Like ARX, its major composition was copper, with low levels of lead and zinc. These two projectiles could be confidently distinguished from the other conventional projectiles through their elemental profiles.

Based on the tested list, cadmium was found absent in all instances. Tin was not always found. Even detected, it appeared in exceedingly low amounts (less than 0.01% of the total composition). In this study, only three types of projectiles were detected with tin, which carries a similar function to antimony in hardening the lead of a projectile [22]. Bismuth and silver were present only in very restricted ranges with less than 0.07%, except in the projectile of Ruag AmmoTec detected with approximately 0.23% silver from the total abundance. Bismuth was reported as a green substitute for lead in the production of ammunition [23]; however, such an element was not evident among the tested ammunition where lead was more common. Apart from that, the presence of bismuth and silver could also be introduced at the refining stage during the production of ammunition, appearing as impurities. It was suggested that both bismuth and silver were not very discriminatory among the projectiles [24].

Discrimination of projectiles through PCA

PCA allows for data visualisation based on the elemental profiles of each sample. **Figure 2** shows the three-dimensional score plot decomposing the data, in which the first PCs demonstrated 45.3%, 22.1%, and 13.0% of the explained variance. The first three PCs described a cumulative variation of up to 80.4% of the data.

It was observed that data points indicating those projectiles originated from the same manufacturers or types of ammunitions tend to cluster together. The four types of projectiles, namely ARX 9 mm, Geco 9 mm, Ruag Ammotec 9 mm, and SME 9 mm, formed their respective clusters in the score plot. Such findings suggested the unique elemental profiles of these projectiles, discriminating them from other projectiles tested in this study. Differences in the relative abundance of elements within the projectiles were found to be possible to differentiate them.

Comparatively, data points of projectiles sourced from WCC and SME, both in .38 calibre, were located closely to each other, probably due to their similar elemental compositions. All six elements detected in the projectile originated from WCC .38 ammunition, with arsenic in addition to the latter. The presence of arsenic was found to be insufficient to discriminate between these two projectiles. Although they could still be distinguished from each other through the score plot, careful interpretation was required. On the other hand, the very similar elemental profiles of projectiles from CBC .38 and S&B 9 mm led to the proximity of their data points at the centre of the score plot. Referring to **Table 1**, both projectiles exhibited similar compositions, except for the presence of tin in the projectile of S&B 9 mm, which did not contribute to discrimination through PCA.

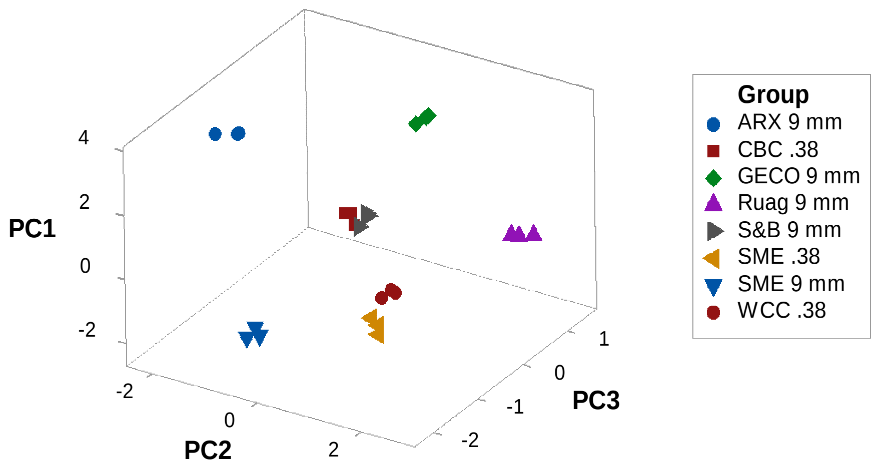

Figure 2. Three-dimensional score plot decomposing the elemental profiles of eight projectiles

In short, different types of ammunitions share the same calibre and can be fired from the same firearm. These different ammunition types were found to have carried different compositional profiles through their elemental analyses. Since 2005, the FBI Laboratory announced that it no longer examines the lead composition in a projectile. Previously, the projectile lead examination was carried out to compare to projectiles associated with a suspect, especially when a firearm has not been recovered or when a fired bullet is too mutilated for comparison of physical markings. However, the association between the projectiles, both known and questioned, was said to have carried limited significance due to the probative value. Instead of serving as a routine-based analysis, such an examination will only be conducted whenever required [11].

Although the discontinuation of projectile lead analysis is routine based, it remains important, as evident in this study. Comparing the eight ammunitions, certain projectiles could be differentiated from those conventional types, especially for non-toxic ammunitions. Their profiles did not report high compositional lead, but copper existed as the more common element that made up the profiles. Such information would be useful in distinguishing the questioned projectile from a crime scene with the known specimen. Furthermore, zinc and copper were also found to be good indicators to suggest if a projectile is jacketed or non-jacketed, as they could be detected in a high proportion in those jacketed bullets. However, it was also emphasized that the compositional copper-zinc should be interpreted with care as a non-jacketed projectile, which might also show the presence of these two elements, probably due to the introduction of impurities during the manufacturing process [24,25].

Conclusion

In this study, the elemental profiles of projectiles from eight types of ammunitions were established. Lead was found to be the major composition in six, while copper dominated the elemental composition in another two, including Geco 9 mm, which was labelled as non-toxic ammunition. Apart from lead and copper, the presence of antimony, arsenic, bismuth iron, silver, tin, and zinc were also detected as minor compositions, depending on the projectiles. Through PCA, the elemental profiles of the eight projectiles formed six clusters, allowing for discrimination and prediction. The elemental ratios correlating to the type of projectiles can be compared to those projectiles found on the scene to link to the residues on an impact mark or hole made by any projectile. A notable limitation identified in this study was the small sample size, involving eight types of ammunitions, restricting to small firearms. Broader information on the elemental profiles of the varying projectiles and their relationship with the impact holes upon perforation would benefit forensic investigators when investigating firearm-related cases. This will allow the comparison of samples recovered from the impact holes with the profiles of any suspected projectile, perhaps as a future recommendation to confirm the cause of an impact or at least to suggest the possible source of ammunition that could have made an impact.

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