International Journal of Chemistry, Mathematics and Physics (IJCMP)
[Vol-6, Issue-2, Mar-Apr, 2022]
https://dx.doi.org/10.22161/ijcmp.6.2.2
ISSN: 2456-866X

Differentiation of high velocity ammunition type from Gunshot Residue (GSR) metal element originating from different firearm

Abhijitsinh A Parmar[1*], Rajesh G Babu[1], S.O Junare[1]

[1]National Forensic Sciences University, Gandhinagar - Gujarat, India.
[*]Corresponding author: Abhijitsinh A Parmar; E-mail: abhiparmar57@gmail.com

Received: 30 Mar 2022; Received in revised form: 19 Apr 2022; Accepted: 26 Apr 2022; Available online: 30 Apr 2022

***Abstract*— *The examination of GSR collected from the scene of occurrence is critical to the forensic science community because it provides answers to problems such as distinguishing between entry and exit wounds, estimating the range of fire, and linking an individual to the use of a firearm. Insufficient information exists on the differentiation of ammunition type through the analysis of GSR. This study investigated the potential of differentiating between ammunition types through the analysis of its metallic residue using Energy Dispersive X-Ray Fluorescence spectroscopy (EDXRF) and Scanning Electron Microscope Energy Dispersive X-Ray Fluorescence spectroscopy (SEM-EDS). Three firearms (AK-47 assault rifle, Self-loading rifle, and Carbine rifle) were adopted for the study. Overall, a series of 10 shots were performed from each firearm at three different collection mode (45°, 90° & Barrel wash) The residues were subjected to a non-destructive analysis by energy dispersive X-Ray fluorescence (EDXRF-7000, Shimadzu) without sample preparation and Scanning Electron Microscope Energy Dispersive X-Ray Fluorescence spectroscopy. Gunshot residues originating from 9mm Carbine, AK-47 and Self-loading rifle produced common metal element including Lead (Pb), Copper (Cu), Mercury (Hg), Antimony (Sb) and Zinc (Zn). Considering that, analysis of the sample residue was carried out EDXRF and SEM-EDS; it was unreliable to predict the ammunition type from the particles that originated from the firearm-ammunition employed. In this study element profile of Gunshot residue give some identical point which can helpful in identification of ammunition.*

Keywords*— *Gunshot residue (GSR), Ammunition; Energy Dispersive X-Ray Fluorescence (EDXRF), Forensic ballistics, Self-loading rifle (SLR), AK-47 rifle, 9mm carbine, Scanning Electron Microscope Energy Dispersive X-Ray Fluorescence spectroscopy (SEM-EDS) and Stereo Microscope.

I. INTRODUCTION

Gunshot residues (GSR) are very important forensic evidences in the event of any criminal activity involving shooting incidents [1-5]. Gunshot residue (GSR) comprises a mixture of chemical compounds that are produced from the series of high-pressure compression reactions, which helps to propel the projectile down the barrel of the gun. These residues fundamentally originating from both the primer and the propellant and other components of the ammunition and the firearm are produced concurrently to form a plume of airborne particulates, which exit from the barrel and any other openings in the firearm [6]. Due to their light in weight, GSR does not travel afar and it usually settles on surfaces that are close to the firearm firing vicinity such as on the hands, clothes, and hair of the shooter.

The examination of GSR collected from the scene of occurrence is critical to the forensic science community because it provides answers to problems such as distinguishing between entry and exit wounds, estimating the range of fire, and linking an individual to the use of a firearm. Studies have revealed that the metallic particles that originate from the primer of ammunition are unique to GSR due to their individualistic morphology and chemical composition. The unique nature of GSR increases its evidential value in connecting an individual to a firing scene and the use of a firearm [7, 8].

Many analytical techniques with high sensitivity and specificity have proved valuable for the analysis and examination of GSR although the scanning electron microscope coupled with energy dispersive x-ray spectrometry (SEM-EDX) is most preferred due to its dual functionality and non-destructive nature. It simultaneously provides information about the characteristic morphology and chemical component of the sample under examination (inorganic GSR such as lead, barium, and antimony)[6]. Environmental and occupational sources such as brake lining, fireworks, and paints are known to produce metallic particles that are akin to the inorganic GSR which makes the reliability of the SEM-EDX questionable and may contribute to the risk of false-positive results in some situations [9, 10, 11]. The analysis of Organic Gunshot Residues (OGSR) by using Spectroscopic (Raman) [12-14], Spectrometric (Mass) [15] and Chromatographic coupled with mass (GC-MS, LC-MS) [16-20] techniques have also been demonstrated to identify and characterize GSR samples as forensic evidences. Although these techniques are quite reliable, they still have few limitations including high cost, need of experienced technician and difficulty in getting sufficient samples in many instances.

Insufficient information exists on the differentiation of ammunition type through the analysis of GSR. This study investigated the potential of differentiating between ammunition types through the analysis of its metallic residue using energy dispersive x-ray fluorescence spectroscopy and Scanning Electron Microscope Energy Dispersive X-Ray Fluorescence spectroscopy (SEM-EDS). All the research show hand swab analysis but Gunshot residue distribution at intermediate target collection of Gunshot residue also a part of gunshot residue analysis, there is a sure sign of firing if collection of Gunshot Residue done successfully at intermediate target. High velocity ammunition shows different element profile than other ammunition like pistol and revolver.

II. MATERIALS AND METHOD

The study was conducted in an indoor firing range (Ballistic Research Center and Testing Range) at National Forensic Sciences University, India. Inorganic GSR was the subject of the present study. Three firearms (AK-47 assault rifle, Self-loading rifle, and Carbine rifle) were adopted for the study. These weapons were selected due to their availability at the firing range. Temperature and relative humidity were maintained at 21℃ ± 2.9 ℃ and 50% ± 20% respectively. This helped to limit the influence of environmental conditions on the distribution of GSR particles. The cartridges used were obtained from the same manufacturer (Ordnance Factory of Varangaon) (OFV) and a batch of ammunition. This helped to assure that the primer compositions of the ammunitions were uniform throughout.

Before test firing, the firearm was cleaned with a white cotton tissue to remove residues deposited from previous firings. The firearm was mounted on a secure mobile firing rest to permit a precise adjustment for each firing. Overall, a series of 10 shots were performed from each firearm at a muzzle-target angle 45° (1-meter distance from muzzle), at a muzzle-target angle 90° angle (1-meter distance from muzzle) and the barrel wash using cotton cloth. So, by this three-collection point (45°, 90° & Barrel wash) 30 sample were collected from each firearm. The firearm was cleaned completely after each shot to remove all residues resulting from the previous firing.

2.1. Sample Collection

Gunshot residue was obtained through barrel wash, and at an angle of 45˚ and 90˚ (1m away from the muzzle end). GSR deposited in the bore of the barrel was obtained using Flannel gun clean cotton cloth (a procedure known as barrel wash). Two (2) A4 sized blank sheet (dimensions 210 × 297mm) was placed 1m away from the muzzle end and at an angle of 45˚ and 90˚ to collect dispersed air-borne GSR. The A4-sized sheet was replaced for each firearm-ammunition used.

Table.1. Summary of sample collection

Description	**Firearm**		
	AK-47 7.62x39 mm	SLR 7.62x51 mm	Carbine 9X19 mm
Total number of shots	10	10	10
Duration of sample collection	After every single shot	After every single shot	After every single shot

Collection Mode	45° at 1-meter 90° at 1-meter Barrel wash	45° at 1-meter 90° at 1-meter Barrel wash	45° at 1-meter 90° at 1-meter Barrel wash
Total number of samples	30	30	30

2.2. Energy Dispersive X-Ray Florescence (EDXRF) Analysis

The residues were subjected to a non-destructive analysis by energy dispersive X-Ray fluorescence (EDXRF-7000, Shimadzu) without sample preparation. The instrument was first calibrated using an Aluminum Standard sample. This was essential to check the accuracy and reliability of the instrument and to determine the traceability of the measurement. The residues were introduced into the sampling cups equipped with a transparent Mylar film base. The X-ray beam was focused onto the residues inside the sampling cup and the samples were scanned in an air mode. The qualitative and quantitative data for the element present in the sample was obtained and recorded. Descriptive statistics (mean) were computed for the metal element present in the sample under examination.

Table.2. Experimental Parameter for EDXRF

Parameter	***Element Series***	
	Al-u Series	Na-Sc Series
kV	50	15
µA	130-Auto	414-Auto
Analysis Collimator	0.00-40.00	0.00-4.40
Scan Time	30 Second	30 Second
Mode	Quick Easy Air & Metal	Quick Easy Air & Metal
Filter	NA	NA
DT %	29	30

2.3. Scanning electron microscope coupled with energy dispersive x-ray spectrometry (SEM-EDS) Analysis

SEM (Zeiss) coupled with EDS (EDAX APEX) was used for the analysis of GSR following ASTM 1588–20.25 The instrumental working conditions of the SEM-EDS were optimized at the start of every analytical run: magnification (70x-1000x). SEM analysis was performed using a backscattered electron detector and a secondary electron detector with a high-resolution image. The cartridge primer specimens were manually investigated for the existence of small particles (0.5–100 µm).

Table.3. Experimental Parameter for SEM:

Parameter	
Electron High Tension (EHT)	5.00 kV
Working Distance (WD)	7.7 mm
Signal A	In lens
Magnification	70-1000x

Table.4. Experimental Parameter for SEM EDS:

Parameter	
kV	10 kV
Take off	35.9

Live Time	100 Second
Amplitude time (µs)	3.84
Resolution	126.6ev

III. RESULTS AND DISCUSSION

The distribution and quantity of metal element originating from 9mm Carbine, AK-47 and Self-loading rifle were explored. For 9mm carbine, Lead (Pb) was present from all the collection method employed and antimony (Sb) was only detected in residues obtained from barrel wash technique. The quantity of Pb was highest relative to the other detected metal element. Other metal element detected from the barrel wash technique included; Hg, P, Cu, and Zn with varying concentrations. The distribution of the metal element originating from 9mm carbine is presented in Table 5. Regarding AK-47, GSR elemental trio (Lead, Barium, and Antimony) was present from residues collected using the barrel wash technique. Again, the quantity of the elemental trio was in the order Lead (Pb), Barium (Ba) and Antimony (Sb). The other metal elements that were detected through the barrel wash technique were Fe, P, K, Cu, Ti, and Zn. The overall distribution of the metal element from the AK-47 arm-ammunition is presented in Table 6.

Moving forward, metal element originating from self-loading rifle was also ascertained (Table 7). The barrel wash technique produced allowed for the detection of majority of the metal element relative to the other collection technique. Amongst the metallic element regarded as unique to GSR, only Pb and Ba were detected from residues obtained from the barrel wash technique. Only Ba was obtained from GSR collected 90˚ away from the muzzle end of the firearm. Other metal element that were also detected were; Fe, P, Cu, and Zn. Gunshot residues originating from 9mm Carbine, AK-47 and Self-loading rifle produced common metal element including P, K, Cu, Ti, and Zn. These detected elements however cannot be considered as being characteristic to GSR because there exist other environmental sources that produce GSR-like metal elements. The possibility that there could be other sources of GSR-like particles is very important. If it were found that any other process or activity could produce particles with indistinguishable morphological and / or compositional characteristics to those of GSR then the weight of such particles as forensic evidence would be greatly reduced. Copper amount was higher in 9 mm Ammunition than AK-47 and SLR rifle, Lead amount was higher than SLR and 9 mm carbine weapon so that by using this point identification if weapon become easier. Mercury was not showing presence in SLR Rifle in any of collection mode. SEM-EDS also show the presence of Lead, Antimony, iron and copper Presence in this study which can easily show the element presence in high velocity ammunition. Particle size of element in SEM was 25 µm & 27 µm .

Table.5. 9mm Carbine EDXRF element Distribution mean%

Element	***Mode of GSR collection***		
	45˚	***90˚***	***Barrel wash***
Pb	0.0172	0.016	3.666
Sb	0	0	1.233
Ba	0	0	0
Fe	0.081	0.181	0
Hg	0	0	1.435
P	2.054	1.837	2.665
Cu	0.050	0.054	9.474
Zn	0.024	0.023	2.110
Al	6.222	0	0

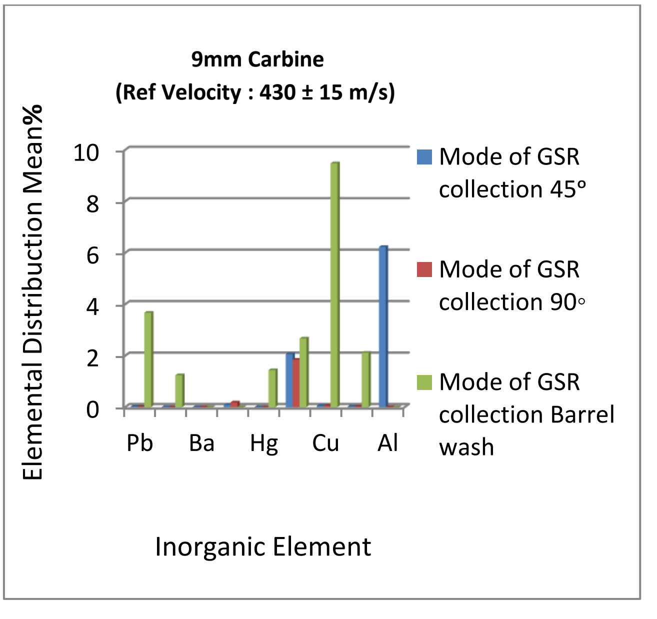


Table.6. AK-47 (HSC) EDXRF element Distribution mean %

Element	*Mode of GSR collection*		
	45°	*90°*	*Barrel wash*
Pb	0	0.021	9.588
Sb	0	0.191	1.255
Ba	0	0	4.747
Fe	0.083	0.086	3.459
Hg	0	0.026	0
P	2.332	1.883	0.554
Cu	0.116	0.336	8.571
Zn	0.022	0.040	3.116
Al	0	0	0

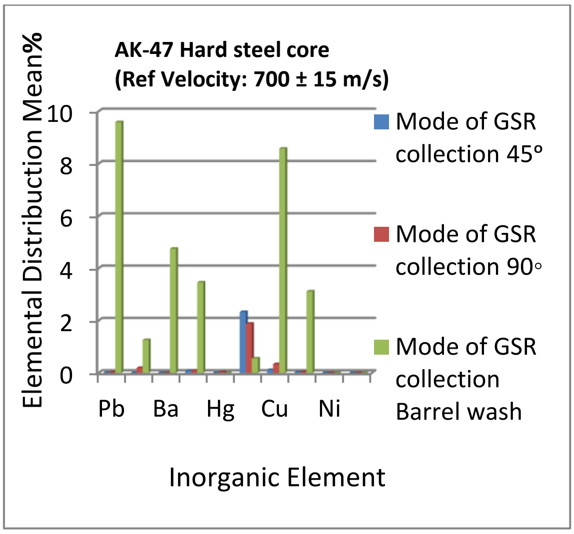


Table.7. Self-loading rifle EDXRF element Distribution mean %

Element	*Mode of GSR collection*		
	45°	*90°*	*Barrel wash*
Pb	0.115	0.025	24.089
Sb	0	0	4.748
Ba	0	0.485	0
Fe	0.526	0.070	3.493
Hg	0	0	0
P	17.433	2.433	2.474
Cu	0.339	0.065	7.434
Zn	0.094	0.021	3.512
Al	34.991	7.576	0

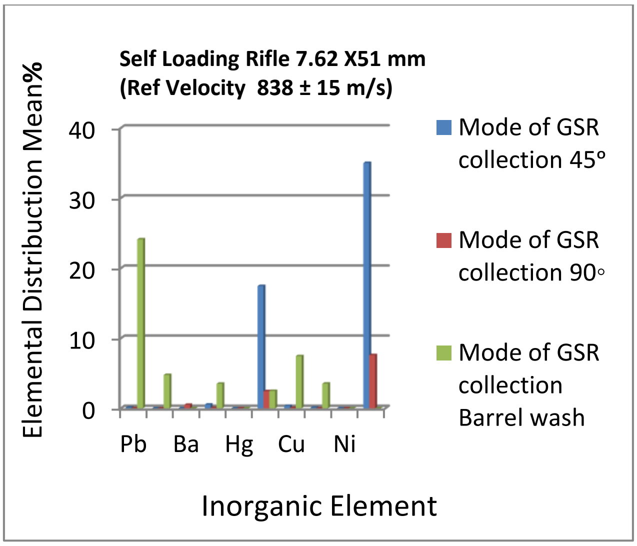


Image.1. SEM Images of Gunshot Residue Element

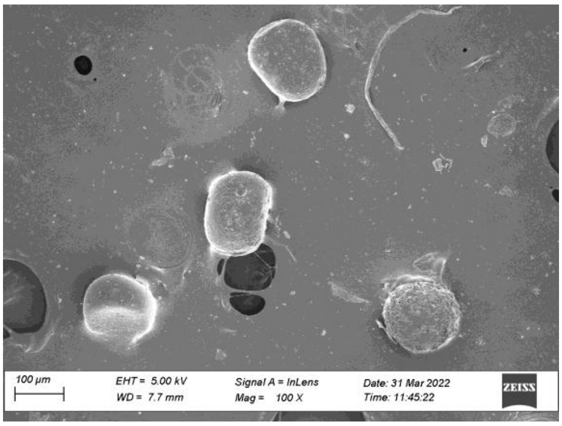
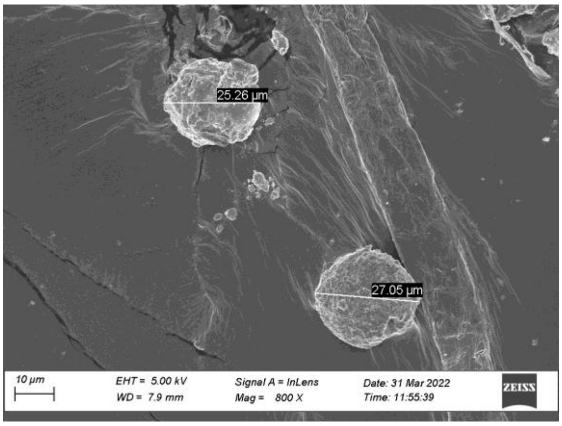


Image.2. SEM Images of Gunshot Residue Element



IV. CONCLUSION

The analysis of both inorganic and organic residues has been shown as a promising method of gaining as much information about any given sample as possible. A combination of these techniques with microscopic or even macroscopic analysis of particle/grain morphologies would be even more favorable. Therefore, this must be seen as the most ideal approach to sample analysis. The interpretation of the results of any sample analysis by an expert witness is incredibly important. A further study into the guidelines which are applied to the interpretation of GSR by independent laboratories/experts would be incredibly valuable in terms of assessing the levels of consistency within the field.

The current study has revealed that, barrel wash followed is best for GSR collection and analysis. Considering that, analysis of the sample residue was carried out EDXRF and SEM-EDS, it was unreliable to predict the ammunition type from the particles that originated from the firearm of high velocity ammunition employed. Identification of different ammunition and firearm will be easy task to solve the crime and other illegal weapon usage cases.

REFERENCES

[1] O. Dalby, D. Butler, J.W. Birkett, Analysis of gunshot residue and associated materials—a review, J. Forensic Sci. 55 (2010) 924–943.

[2] L.S. Blakey, et al., Fate and behavior of gunshot Residue — a review, J. Forensic Sci. 63 (1) (2018) 9–19.

[3] K.H. Chang, et al., Gunshot residue analysis and its evidential values: a review, Aust. J. Forensic Sci. 45 (1) (2013) 3–23.

[4] F. SaverioRomolo, P. Margot, Identification of gunshot residue: a critical review, Forensic Sci. Int. 119 (2001) 195–211.

[5] A.J. Schwoeble, D.L. Exline, Current Methods in Forensic Gunshot Residue Analysis, CRC Press LLC, Boca Raton, FL, 200

[6] Dalby, O., Butler, D., & Birkett, J. W. (2010). Analysis of gunshot residue and associated materials - A review. *Journal of Forensic Sciences*, *55*(4), 924–943.

[7] Brozek-Mucha, Z. (2011). Variation of the chemical contents and morphology of gunshot residue in the surroundings of the shooting pistol as a potential contribution to a shooting incidence reconstruction. *Forensic Science International*, *210*(1–3), 31–41.

[8] Aksoy, C., Akman, Y., Ergun, Z. O., Uzek, U., & Aydin, F. (2013). Determination of antimony in gunshot residues using GFAAS and SEM/EDX. *Atomic Spectroscopy*, *34*(5), 170–174.

[9] Saverio Romolo, F., & Margot, P. (2001). Identification of gunshot residue: A critical review. *Forensic Science International*, *119*(2), 195–211.

[10] Schwoeble, A., & Exline, D. L. (2000). Current Methods in Gunshot residue analysis. In *CRC Press*. Turillazzi, E., Di Peri, G. P., Nieddu, A., Bello, S., Monaci, F., Neri, M., … Fineschi, V. (2013). Analytical and quantitative concentration of gunshot residues (Pb, Sb, Ba) to estimate entrance hole and shooting-distance using confocal laser microscopy and inductively coupled plasma atomic emission spectrometer analysis: An experimental study. *Forensic Science International*, *231*(1–3), 142–149.

[11] Taudte, R. V., Roux, C., Blanes, L., Horder, M., Kirkbride, K. P., & Beavis, A. (2016). The development and comparison of collection techniques for inorganic and organic gunshot residues. *Analytical and Bioanalytical Chemistry*, *408*(10), 2567–2576.

[12] Z. Abrego, N. Grijalba, N. Unceta, M. Maguregui, A. Sanchez, A. Fernández-Isla, M.A. Goicolea, R.J. Barrio, A novel method for the identification of inorganic and organic gunshot residue particles of lead-free ammunitions from the hands of shooters using scanning laser ablation-ICPMS and Raman micro-spectroscopy, Analyst 139 (2014) 6232–6241.

[13] J. Bueno, V. Sikirzhytski, I.K. Lednev, Raman spectroscopic analysis of gunshot residue offering great potential for caliber differentiation, Anal. Chem. 84 (2012) 4334–4339.

[14] J. Bueno, I.K. Lednev, Raman microspectroscopic chemical mapping and chemometric classification for the identification of gunshot residue on adhesive tape, Anal. Bioanal. Chem. 406 (2014) 4595–4599.

[15] M. Morelato, et al., Screening of gunshot residues using desorption electro-spray ionisation-mass spectrometry (DESI-MS), Forensic Sci. Int. 217 (1–3) (2012) 101–106.

[16] L. Ali, et al., A study of the presence of gunshot residue in Pittsburgh police stations using SEM/EDS and LC-MS/MS, J. Forensic Sci. 61 (4) (2016) 928–938.

[17] A.L. Gassner, C. Weyermann, LC-MS method development and comparison of sampling materials for the analysis of organic gunshot residues, Forensic Sci. Int. 264 (2016) 47–55.

[18] J.L. Thomas, D. Lincoln, B.R. McCord, Separation and detection of smokeless powder additives by ultra-performance liquid chromatography with tandem mass spectrometry (UPLC/MS/MS), J. Forensic Sci. 58 (3) (2013) 609–615.

[19] M. Gallidabino, et al., Development of a novel headspace sorptive extraction method to study the aging of volatile compounds in spent handgun cartridges, Anal. Chem. 86 (2014) 4471–4478.

[20] M. Gallidabino, F.S. Romolo, C. Weyermann, Characterization of volatile organic gunshot residues in fired handgun cartridges by headspace sorptive extraction, Anal. Bioanal. Chem. 407 (23) (2015) 7123–7134