Trace analysis of Gunshot residue on different fabrics using locally manufactured ammunition in Pakistan

Muneeba Butt, Makhdoom Saad Ghouri

Abstract --- Gunshot residue (GSR) is also known as cartridge discharge residue (CDR) or firearms discharge residues are the particles produced during the discharge of a firearm. The demonstration of GSR in firearm injuries is of prime importance in forensic science. It creates an understanding of the nature of injury and the direction of the shot which might be clearly demonstrated in the courtroom. GSR has a main role in criminalistics department. The objective of the present study was to detect the chemical components present in Gunshot residues, the elemental and microanalysis of Gunshot residues by using Scanning Electron Microscopy with energy dispersive X-ray spectroscopy. The compostion of Gunshot residues was analyzed by using three chemical tests; Modified Griess test, Sodium rhodizonate test and diphenylamine test. The presence of Gunshot residues evaluate on the basis of different color intensities obtained by different chemical procedures. The more/less GSR detected, the more/less color intensity was observed. Modified Griess test and Diphenylamine test give orange and blue color to indicate the presence of nitrities/nitrates whereas sodium rhodizonate test give pink color for the detection of lead residues. Scanning electron microscopy give the exclusive geometrical images of different GSR particles with high magnification power and the peaks of GSR components can also be plotted with the help of EDX. The persistency of GSR residues on different fabrics cannot depend upon on the variable firing distances rather it depends on the texture of the fabrics. As the distance was increased the GSR intensity was decreased, which was observed by this research. This work will help to reconstruct the shooting scene to estimate muzzle-to-target distance up to 7feet.

Key words --- Ammunition, Diphenylamine test, firearm, Gunshot Residues (GSR), Modified Griess test, Scanning Electron Microscopy/ energy dispersive X-ray spectroscopy (SEM/EDX), sodium rhodizonate test,

1 INTRODUCTION

Gunshot residues in firearm injuries are of prime importance in forensic sciences [7]. GSR has a main role in criminalistics department. It is also used to determine that whether a person discharged a bullet or not. Primer, propellant, metals contained in the bullet, bullet jacket, cartridge case, and gun barrel are the parts from where GSR can be obtained. When a gun is discharged, the resulting hot gases produced by the detonation and ignition of the cartridge are ejected from muzzle and the breech in a dense cloud and the cool quickly. The vaporized materials within the gas condense and are deposited as particulate known as gunshot residue (GSR) or sometimes as firearms discharge residue (FDR) [3]. The combinations of these elements lead (Pb), barium (Ba), and antimony (Sb) have been found in GSR particles based on lead styphnate primers or mercury fulminate primers having little amount of mercury (Hg) [4]. High amounts of lead, barium and antimony considered to be characteristic of gunshot residue [1].
GSR analysis is the most simplest and reliable method for the estimation of shooting distance (muzzle-too-target) [5]. When persons associated with a shooting are arrested, gunshot residue (GSR) samples are collected from all subjects in the vicinity and submitted for forensic analysis. For further examination, the exposure of shooters and bystanders to GSR generated by a variety of handguns should be compared [8].
The ability of the SEM/EDX technique to visualize the individual GSR particles and to determine their elemental composition has led to its widespread adoption in forensic science laboratories in spite of purchasing the high cost necessary equipment and maintaining it [2].

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2 EXPERIMENTAL PROCEDURES

2.1: REVIEW

Tests were performed in an indoor firing range to establish the distance gunshot residue. Sample collection devices were positioned at the firearm's ejection port and horizontally positioned down range up to a distance of 18 m. The collection devices were analyzed for gunshot residue (GSR) using scanning electron microscopy / energy dispersive X-ray analysis (SEM/EDX). The area of highest GSR particle deposition was observed to be 13.5 m downrange of the firearm, however small numbers of particles were also found at a distance of 18 m. It was found that GSR particles travel in close association with the projectile as it was observed that the concentration of GSR particles on the rear target was an order of magnitude higher than the concentration on the forward target. GSR particle numbers cannot be used to distinguish between a shooter, an individual near the path of the projectile, or a shooting victim. (Gerard et al. 2011)

2.2: CHEMICALS

For Modified Griess test, 15% Glacial Acetic acid solution, 0.5g sulphanilic acid, 0.28g alpha-napthol in 100mL methanol was required. The fabrics were soak in these solutions and then sprayed by 15% Acetic acid. Orange color was indicated the presence of nitrite residues. For sodium rhodizonate test, 0.2g sodium rhodizonate solution formed dark tea color solution. Sodium bitartrate and Tartaric acid were used to make buffer solution. Hot plate heating (agiriting) was applied with magnetic stirrer and pH: 2.8 adjusted by 2.5N NaOH. With yellow background pink color indicate the presence of lead (Pb) residues. For Diphenylamine Solution, 0.3g diphenylamine to 20mL conc. sulfuric acid. Add this mixture to 10mL glacial acetic acid. GSR particles were separate on the basis of size, shape and color. Dark blue color reaction on an unknown GSR particle was indicating the presence of nitrates and nitrites. All the chemicals import from ACROS ORGANICS, New Jersey. USA.

2.3: INSTRUMENTATION

Mettler Toledo weighting balance was used for the appropriate weighing of chemicals. Sony Digital Camera (14.1 megapixel, 4× Optical Zoom) was used for photography. Scanning Electron Microscope with energy dispersive X-ray and GSR analysis software (SEM/EDX) was used for microanalysis and elemental study of GSR particles.

2.4: SAMPLING

The fabrics of 5 different types (Cotton, KT Cotton, Polyester, Silk, and Raw-silk) were collected from a local market of Lahore, Pakistan as the targets for Gunshot residues. The fabrics were cut into squares of appropriate sizes (18×18 inch). Each fabric was labeled according to different distances (0, 1,3,5,7 feet) and placed over cardboard, pulled tightly and stapled to stretch it. The cardboard mounted on wooden frame for shooting. AK-47 rifle and 9mm pistol with un-registered locally manufactured ammunition was use for shooting. The entire shooting took place at the shooting range of Punjab Forensic Science Agency, Home Department, Lahore. The targeted fabrics were covered by using butter papers/ plastic sheets to avoid the environmental effects.

2.5: METHODOLOGY

Modified Griess test and Sodium rhodizonate test was applied on the Gunshot residue samples on fabrics. Lead and nitrite residues can be observed by colors of chemicals tests. Lead turns violet/purple colored ring and Nitrite residues turns orange-red. For modified Griess test, the sheets of filter paper were submerged in the solution mixture. Then, the GSR fabric samples were sprayed by 15% acetic acid on entrance hole. Then, fabric was placed on the dampened filter paper. Again, 15% acetic was sprayed on the exit hole of the fabric sample. These layers were pressed by uncontaminated iron. Acetic acid steam was help in color producing reaction. The fabric sample was separated from the filter paper and orange color was appeared on filter paper which indicated the presence of nitrite residues [6]. For sodium rhodizonate test, Bashinsky Transfer Technique was used. The sheets of filter paper were damped with Acetic acid. Place this paper on the tested area. Place a second piece of paper over the first and apply a hot uncontaminated iron for 5seconds. Remove both pieces of filter paper and spray Sodium rhodizonate solution on the tested area. Spray the tested area with Buffer solution. The tested area was sprayed by Hydrochloric acid solution. Repeat this process on the whole area to be tested. For diphenylamine test, the sample fabric was examined macroscopically and microscopically. GSR particles were separated on the basis of size, shape and color. Dark blue color reaction on an unknown GSR particle indicated the presence of nitrates and nitrites. For positive control diphenylamine mix with 15% acetic acid and dark greenish blue color observed.

2.6: SEM/EDX

SEM/EDX is a high magnification microscopy for high resolution of samples. The scanning electron microscopy is a type of electron microscope that images the GSR by scanning it with a high energy beam of electrons in a scan pattern. The electrons interact with the atoms that make up the sample producing signals that have information about the topography, composition and other properties such as electrical conductivity of GSR. The residues don't require any preparation and can be examined in their natural state. Microstructure and elemental analysis of GSR will be accomplished by using scanning electron microscopy with energy dispersive X-ray.

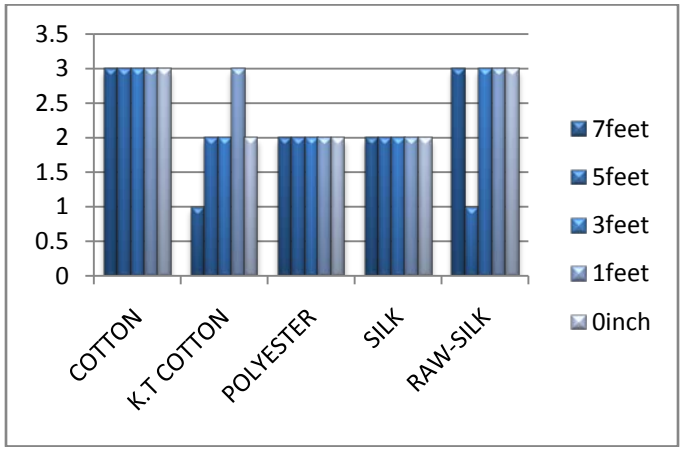
2.7: RESULTS AND DISCUSSION

The diphenylamine test results show different color intensities within different fabrics with different firing distances. The more/less intensity of the color shows more/less presence of GSR on fabrics. Diphenylamine test give greenish blue color for the detection of nitrates and nitrites. The below mentioned results show the presence of GSR components with 9mm pistol with the help different intensities.

Figure 1: DPA test performed on cotton and K.T Cotton with 9mm Pistol

Fabric types	Firing Distance 7 ft	Firing Distance 5 ft	Firing Distance 3 ft	Firing Distance 1 ft	Firing Distance 0 inch	Positive Control
Cotton	Greenish blue color detected	Greenish blue color detected	Greenish blue color detected	Greenish blue color detected	Greenish blue color detected	Blue color detected
K.T Cotton	Light blue color detected	Dark blue color detected	Dark greenish blue color detected	Greenish blue color detected	Dark blue color detected	Blue color detected
Polyester	Dark blue color detected	Dark blue color detected	Dark blue color detected	Dark blue color detected	Dark blue color detected	Blue color detected
Silk	Dark blue color detected	Dark blue color detected	Dark greenish blue color detected	Dark greenish blue color detected	Dark blue color detected	Blue color detected
Raw-silk	Greenish blue color	Light blue color	Greenish blue color	Greenish blue color	Greenish blue color	Blue color

Table no. 01: Diphenylamine test with 9mm Pistol

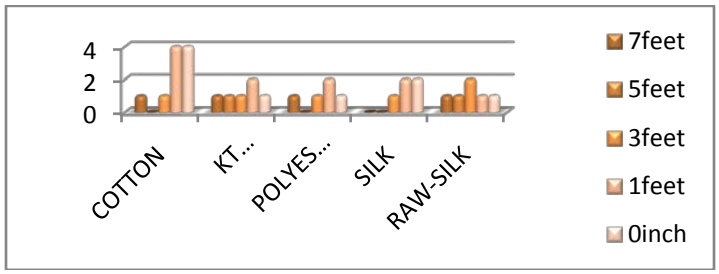


Diphenylamine test with 9mm Pistol
KEY: Very light Blue color '0', Light '1', Dark '2', Very Dark '3', Deep '4'.

The modified griess test results show different color intensities within different fabrics with different firing distances. The more/less intensity of the color shows more/less presence of GSR on fabrics. Modified griess test give orange color for the detection of nitrates and nitrites. The below mentioned tabulated results show the presence of GSR components with 9mm pistol with the help different intensities.

Table no. 02: Modified Griess test with 9mm Pistol

Fabric types	Firing Distance 7 ft	Firing Distance 5 ft	Firing Distance 3 ft	Firing Distance 1 ft	Firing Distance 0 inch	Positive Control
Cotton	Light orange color detected	Very light orange color Detected	Light orange color detected	Deep orange color detected	Deep orange color detected	Dark orange color detected
K.T Cotton	Light orange color detected	Light orange color Detected	Light orange color detected	Dark orange color detected	Orange color detected	Dark orange color detected
Polyester	Light orange color detected	Very light orange color Detected	Light orange color detected	Dark orange color detected	Light orange color detected	Dark orange color detected
Silk	Very light orange color detected	Very light orange color Detected	Light orange color detected	Dark orange color detected	Dark orange color detected	Dark orange color detected
Raw-silk	Light orange color detected	Light orange color detected	Dark orange color detected	Light orange color detected	Light orange color detected	Dark orange color detected



Modified Griess test with 9mm Pistol

KEY: Very light Orange color '0', Light '1', Dark '2', Very Dark '3', Deep '4'.

Figure 2: 9mm pistol shot on fabric

Figure 3: Orange color detected on fabric by Modified Griess test

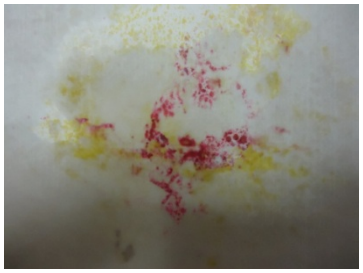


Figure 4: Pink color detected on fabric by Sodium rhodizonate test

The Scanning Electron Microscopy with energy dispersive X-ray give the length, Width, shape, 3D dimension of the GSR residues with different magnification powers and also give the spectrum which estimate the components present in GSR of variable distances.
Here, is a figure and spectrum of 0 inch 7.62×39 rifle.

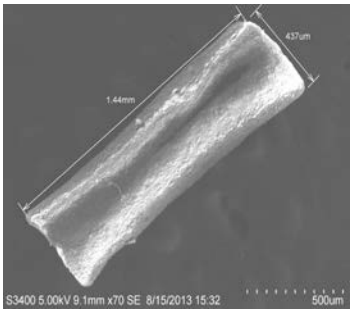


Figure 5: 7.62×39 rifle GSR image on SEM/EDX and its spectrum

The data was analyzed with one way ANOVA using Statistical Package for Social Sciences (SPSS);
$P < 0.05$ was considered significant.

This data of present study correlates with the research work of Izzharif et al. (2010). In this study, the GSR on cotton cloth from close range fire of 3inchs to 12 inches was analyzed. Two types of ammunition used. The nitrite residues were confirmed using modified Griess test. On the other hand, Scanning Electron Microscope (SEM) micrograph of GSR particles discharged from semiautomatic pistol revealed a mean particle size of 2.6µm with spherical shapes. The modified Griess test revealed that revolver pistol generated tiny amount of nitrite residues.
The shooting distance estimated on the detection basis of organic Gunshot Residues. This study was processed on the target cloth material using various distances. Modified Griess test was performed. It results that as muzzle to target distance increase the intensity of the color decrease. GSR particles were measured by SEM 10000X magnification (Ananth et al. 2011). According to my study, GSR contains nitrites/nitrates and lead components on five different fabrics with five different distances. The intensity of GSR varies from distance to distance and fabric to fabric.

2.8: CONCLUSION

Information gained from this study that the persistency of GSR residues on different fabrics cannot depend upon on the variable firing distances rather it depends on the texture of the fabrics. As the distance was increased the GSR intensity was decreased, which was observed by this research. This work will help to reconstruct the shooting scene to estimate muzzle-to-target distance up to 7feet.

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REFERENCES:

Nesbitt RS, Wessel JE, Jones PF. 1976. Detection of Gunshot Residue by Use of the Scanning Electron Microscope. Journal of Forensic Sciences. 21(3).

Zeichner A, Foner HA, Dvorachek M, Bergman P, Levin N. 1989. Concentration Techniques for the Detection of Gunshot Residues by Scanning Electron Microscopy/Energy Dispersive X-Ray Analysis (SEM/EDX). Journal of Forensic Sciences. 34(2), 312-320.

Harald A. Wrobel, John J. Millar, Marek Kijek. 1998. Identification of Ammunition from Gunshot Residues and Other Cartridge Related Materials—A Preliminary Model Using .22 Caliber Rimfire Ammunition. Journal of Forensic Sciences. 43 (2), 324–328.

Kage S, Kudo K, Kaizoji A, Ryumoto J, Ikeda H, Ikeda N. 2001.A simple method for detection of gunshot residue particles from hands, hair, face, and clothing using scanning electron microscopy/wavelength dispersive X-ray (SEM/WDX). Journal of Forensic Sciences. 46(4), 830–834.

Margherita N, Emanuela T, Irene R, Vittorio F. 2007.The determination of firing distance applying a microscopic quantitative method and confocal laser scanning microscopy for detection of gunshot residue particles. International Journal of legal medicine. 121, 287–292.

Mohamed Izzharif A Halim, Umi Kalthom Ahmed, Choong-Hui Yew, Muhammad Koey Abdullah. 2010. A nalysis of Gun ShotResdiue Deposited on Cotton Cloth Target at Close Range Shooting Distances. Malaysian Journal of Forensic Sciences. 1(1).

Salvatore A, Guendalina G, Alessio B, Cristina C, Riccardo. 2011. Forensic Applications of Sodium Rhodizonate and Hydrochloric Acid: A New Histological Technique for Detection of Gunshot Residues. Journal of Forensic Sciences. 56(3), 771-774.

Elspeth Lindsay, Michael J McVicar, Robert V Gerard, E Dale Randall. 2011.
Observations of GSR on the Hands of Employees at Firearms Manufacturing Facilities, Journal of Canadi

An society of Forensic Science. 44(3), 105-109.