

Investigation of Pseudo-residue Existence Obtained from the Hands of Employees in Various Business Lines

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ABSTRACT

Handswap analysis is carried out in criminal laboratories in order to illuminate the crime scenes in which firearms are used. The reliability of the results obtained from the analyzes is the subject of the chemistry department and is based on sample reliability. In this study, the hand swabs of people working in fifteen various business lines were obtained using transfer kit in order to acquire a scientific response to the issue of whether there would be shot residues in the hands of people in everyday life. Collected samples were first examined by Scanning Electron Microscopy (SEM-EDS). The elements of Sb&Ba&Pb, as well as their combinations, were investigated in the samples. After SEM-EDS analysis, all samples were analyzed using graphite furnace atomic absorption spectrometry (GFAAS) method to determine the amount of antimony element. According to the results, antimony element was detected on the surface of the caliper used in the printing house via GFAAS. Sb&Ba&Pb particles were also detected by SEM-EDS analysis in the swabs taken from the hands of the automobile battery repairman and the market cashier.

Çeşitli İş Kollarında Çalışanların Ellerinden Elde Edilen Yanıltıcı Kalıntı Varlığının Araştırılması

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ÖZ

Ateşli silahların kullanıldığı poliseye olayların aydınlatılması için kriminal laboratuvarlarında el svap analizi yapılmaktadır. Analizlerden elde edilen sonuçların güvenilirliği, kimya anabilim dalının konusu olup örneklem güvenilirliğine dayalıdır. Bu çalışmada, insanların günlük hayatta ellerinde kurşun kalıntısı olup olmayacağı konusuna bilimsel bir cevap verebilmek için on beş farklı iş kolunda çalışan kişilerin el sürüntüleri transfer kiti kullanılarak elde edilmiştir. Toplanan numuneler ilk olarak Taramalı Elektron Mikroskobu (SEM-EDS) ile incelenmiştir. Numunelerde Sb&Ba&Pb elementleri ve bunların kombinasyonları incelenmiştir. SEM-EDS analizinden sonra tüm numuneler GFAAS yöntemi kullanılarak antimon element miktarının belirlenmesi için analiz edilmiştir. Elde edilen sonuçlara göre matbaada kullanılan kumpasın yüzeyinde GFAAS aracılığıyla antimon elementi tespit edildi. Otomobil akü tamircisi ve market kasiyerinin ellerinden alınan sürüntülerde ayrıca SEM-EDS analizi ile Sb&Ba&Pb partikülü tespit edildi.

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1. Introduction

Determining gunshot residue (GSR) evidence is an essential part of forensic investigation in firearm-related crimes (Fidan and İzgi, 2009; Kara et al., 2015; Goudsmits et al., 2016; Bender et al., 2021; Feeney et al., 2022; Yüksel et al., 2022). When firing occurs in firearms, a cloud of dust spreads from the muzzle of the gun and other cavities such as the barrel ejection cavity, thanks to the high heat and pressure created by firing (Aliste et al., 2020; Montoriol et al. 2021). This gas cloud is referred to as "GSR" because it comprises burnt or partially burned gunpowder remnants, metal particles, and lubricants (Chang et al., 2014; Ulrich et al., 2004). Antimony (Sb), lead (Pb), and barium (Ba) are the most common elements found in it (Tugcu et al., 2005; Dalby et al., 2010; Chang et al., 2013). Various volatile chemicals such as nitroglycerine, nitro guanine, and others make up the organic component, while heavy metals such as Sb, Pb, Ba, and others make up the inorganic component (Maitre et al., 2018; Shrivastava et al., 2021). These gunshot residues, which are often spherical and have distinct characteristics, might get up on the person carrying the gun's hands, face, or clothing (Dalby et al., 2010; Rijnders et al., 2010; Maitre et al., 2018; Blakey et al., 2019; Rosengarten et al., 2021). There are a variety of ways for collecting GSR samples, and choosing the best one is critical to maintaining optimal collection efficiency. Tape lifting, swabbing, vacuuming, and adhesive lifting are some of the methods used to collect data (Dalby et al., 2010; Gassner and Weyermann, 2020).

Color tests and instrumentation methods are used in analytical methodology to detect GSR (Tarifa and Almirall, 2015; Werner et al., 2020). Color spot tests such as paraffin test, cutaneous nitrate test, Walker test, Marshal and Tiwari test are used. However, it is stated that these procedures are insufficient for analysis as they will damage the sample and cause interaction with other media elements (Shrivastava et al., 2021). Many instrumental analysis instruments can perform qualitative and quantitative analyses with great precision and sensitivity. Examples of these devices are Scanning Electron Microscope (SEM) (Rosengarten et al., 2021), Atomic Absorption Spectrometer (AAS) (Yüksel et al., 2016), Neutron Activation Analysis (NAA) (Koons et al., 1987) and Inductively Coupled Plasma Mass Spectrometer (ICP-MS) (Aliste et al., 2020) etc. There are many studies have been obtained in the literature comparing the benefits and disadvantages of shot residue analysis methods (Matricardi and Kilty, 1977; Brozek-Mucha and Jankowicz, 2001; Taudte et al., 2014; Ali et al., 2016; Shrivastava et al., 2021). GFAAS, which has advantages such as cost, sensitivity, and quick analysis findings, and SEM, which has advantages such as analyzing the elemental composition of the particles and studying their morphological properties, are two procedures that are often employed (Chang et al., 2013; Hearn et al., 2015; Kara and Yalçinkaya, 2017; Bender et al., 2021; Caccia et al., 2021). Especially the analysis of Sb, Ba and Pb in the GSR can be obtained with high accuracy and precision by GFAAS and SEM-EDS. Although these particles are found in GSR, people hands may be contaminated by Sb, Ba and Pb, depending on the working environment in different business lines (Costa et al., 2016). As a result, these three elements, particularly Sb, were given considerable attention in the study of criminal (Di Maio, 1999; Yüksel et al., 2016; Stamouli et al., 2021; Tahirukaj

et al., 2021). It is known that Sb is used in some Pb alloys, as well as Sb, Ba and Pb components in the hands of people working in certain professions such as electrical installers and car mechanics (Romolo and Margot, 2001; Romolo et al., 2019) . In the study of Tucker et al. (2017), samples of a large number of particles containing both Ba and its derivatives were taken from the brake pads, rims and tires of various automotive manufacturers, as well as from the hands of workers who came into contact with these parts. As a result, Sb was found. Although this raises doubts about GSR, no particles containing all three components have been discovered. In fact, all three components must all be present for a particle to be termed GSR. Therefore the study is very important in terms of showing that pseudo-positive results cannot be accepted for these occupational groups in determining GSR.

In this study GSR-business line relations were expand and it was undertaken on participants from various business lines in order to get actual data on whether people can have gunshot residues in their hands as a result of their job and to provide a source for the literature.

2. Materials and methods

2.1. Sample Collection

This work was ethically approved by Diyarbakır Regional Criminal Police Laboratory, Turkish National Police, Diyarbakır, Turkey (Decision Number: 61956719-4590.(51368)-631/01.12.2021). A signed information form was supplied to each participant from whom the sample was obtained, in line with the principles of the Declaration of Helsinki, signed by the World Medical Association in 1964.

Scanning Electron Microscope with Energy Dispersive X-ray Analyzer (SEM-EDS) was used to analyze the collected materials. For this purpose CARL ZEISS EVO SEM equipped with an Oxford Instruments AZTEC energy dispersive x-ray (EDS) spectrometry analysis system was used. The SEM-EDS analysis was conducted in accordance with ASTM Designation E 11496-8 by using Plano GSR and particle analysis calibration kit (Serial: 60806-5). Analysis was achieved via AZTEC software to maintain control of the SEM stage movements and electron beam positioning. The quantity of particles on the samples, their combinations, and the surface areas of Sb&Ba&Pb particles were all determined during the tests. The information about the SEM-EDS device and analysis conditions were given in Table 1.

Table 1. Information about the SEM-EDS device and analysis conditions

Brand/Model	Zeiss/Evo MA15	
Flament Type	LaB6	
Detector	Collector Bias 300 V	
System Vacuum	1,07 e-06 mbar	
Diaphragm	Fil I Target	1.700 A
	EHT	20.000 kV
Electron Gun	Beam Current	30 μ A
	EHT Target	20.00 kV

Agilent AA280Z brand graphite furnace atomic absorption spectrometer, included with Zeeman background correction was used to determine Sb concentration. Boosted discharge hollow cathode

lamps were used as the excitation source. The GFAAS instrumental operating parameters were listed in Table 2.

Table 2. Operating Parameters for GFAAS System

Instrument	Zeeman
Element – Matrix	Antimony (Sb)
Instrument Mode	Absorbance
Current	12.0 mA
Wavelength	217.6 nm
Slit Width	0.2 nm
Measurement Mode	Peak Height
Concentration Unit	µg/L
Replicates Standard	3
Number of Sample Readings	1
Calibration Algorithm	Linear
Control concentration	10 µg/L
Standard Antimony Concentration and Standard Concentrations used for Calibration Curve	100 µg/L (Standard 1: 20 µg/L, Standard 2: 40 µg/L, Standard 3: 60 µg/L, Standard 4: 80 µg/L)

2.2. Procedure

The stock solution, which was 5% HNO₃ containing 100 µg/L Sb (High Purity Standards, Charleston, USA), was diluted by using 5% (v:v) HNO₃ for preparing the calibration standards which were 20, 40, 60 and 80 µg/L Sb solutions. Analysis samples were shaken at 200 rpm (revolutions per minute) for 30 minutes by adding 5 mL of 5% nitric acid.

2.3. Optimization and Sample Treatment

In order to obtain the best results in GFAAS and GSR measurements, it is necessary to pay attention to some important parameters such as choosing the appropriate wavelength for the matrix (217,6 nm for Sb), choosing the most suitable furnace program (in Table 3), and maintaining the linearity in the calibration curve (in Figure 1). As can be seen in Figure 1, the calibration plot obtained for Sb showed good linearity with 0,9998 regression coefficient (Absorbance = 0,0091xC + 0,0048).

Table 3. Graphite Furnace Temperature Program

Step	Temperature (°C)	Time(s)	Argon gas flow rate time (L/min)	Reading and signal recording
1	95	5,0	0,3	No
2	120	20,0	0,3	No
3	300	20,0	0,3	No
4	750	10,0	0,3	No
5	750	10,0	0,3	No
6	750	2,0	0,0	No
7	2300	0,8	0,0	Yes
8	2300	0,8	0,0	Yes
9	2700	0,3	0,3	No

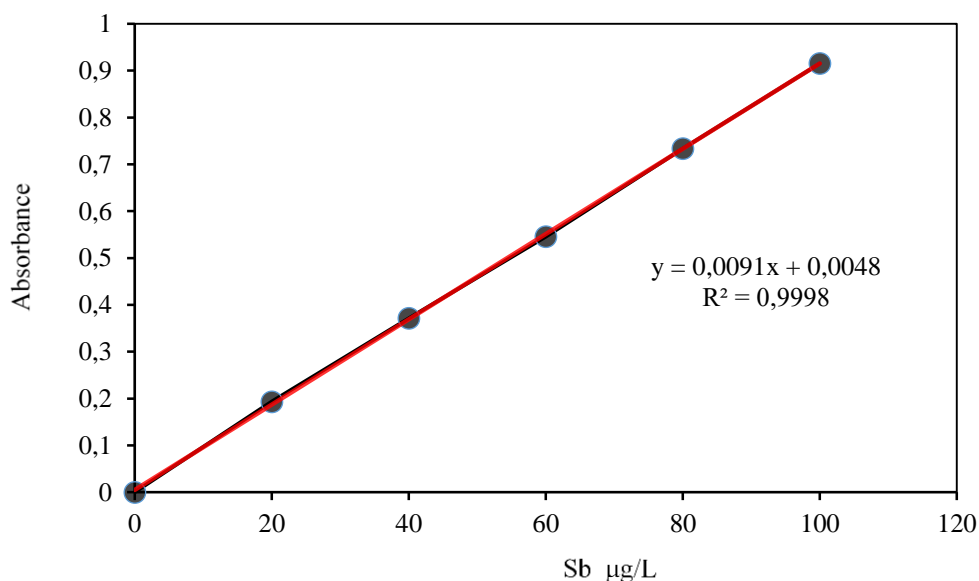


Figure 1. The calibration plot obtained for Sb analysis.

2. 4. Validation of Method

Certified reference solution of 100 µg/L Sb and prepared solutions were used ten times with triplicate measurements to verify the technique in terms of accuracy, precision, and recovery. The relative standard deviation (RSD) of 10 independent measurements of certified reference materials was used to determine the accuracy of the procedures. The certified value for Sb was 100±0,5 µg/L, while the measured values for Sb was 103.28±1,16 µg/L. The recovery and relative standard deviation (RSD) for Sb was 103,28% and 1,12%, According to these results, good agreement was obtained between the certified values and the measured Sb concentration.

2.5. Limit of Detection and Quantification

Limit of detection (LOD) and minimum limit of quantification (LOQ) values are crucial in determining the significance of identified GSR elements; because LOD is the lowest amount (void) of a substance that can be distinguished from its absence. value) has a specified confidence level and the LOQ can be defined as the lowest concentration at which the analyte can not only be accurately detected but also meets certain targets for bias, precision and total error.

For the purpose of this study, participants working in 15 different business lines in Diyarbakır were selected. The hand swabs of the participants were taken separately with the shot residue transfer kit before and after they washing hands. The business lines, sample numbers and abbreviation of sample code of the participants were given in Table 4.

Table 4. Business lines, sample numbers and abbreviation of sample code of the participants whose hand swabs were taken.

Business lines	Sample Numbers	Abbreviation of Sample Code
Phone Repairman*	1+1	1a, 1b
Tinsmith*	1+1	2a, 2b
Coppersmith*	1+1	3a, 3b
Jeweler*	1+1	4a, 4b
Printing Press Worker*	2+1	5a, 5b, 5c
Silversmith*	1+1	6a, 6b
Blacksmith/Welder*	1+1	7a, 7b
Textile Worker*	1+1	8a, 8b
Auto Engine Mechanic*	1+1	9a, 9b
Auto Battery Repair*	1+1	10a, 10b
Auto Body Repair*	1+1	11a, 11b
Auto Electrical Mechanic*	1+1	12a, 12b
Market Employee (Cashier)**	1+1	13a, 13b
Shooting Range Employee (Shot)**	1+1	14a, 14b
Person Carrying a Firearm (Has a License to Carry a Gun)**	1+1	15a, 15b

* were selected from the employees working in the workshop/production departments.

*** hand swabs were taken in line with their consent, at work and while performing their business line. A shot residue transfer kit was used for the right and left hands. “a” refers to samples taken without washing hands, “b” refers to samples taken after washing, and “c” refers to the caliper used in the printing press.

The analyses were obtained with two step procedure. Firstly SEM-EDS measurements were achieved in order to detect Sb&Ba&Pb and then samples were placed in swab boxes which were contained 5% (v/v) nitric acid solution, they were shaken at 220 rpm for 20 minutes before the GFAAS measurements in order to detect the presence of Sb. After shaking, 2 mL of solution were transferred to sample cups and used for the GFAAS analysis. 5% (v/v) nitric acid solution was used as the dilution solution, 1% (v/v) nitric acid solution was used as the washing solution. The calibration of the device was checked by reading the standard Sb solution every ten samples.

3. Results and Discussion

The determination of the chemical and physical characteristics of firearm residues is crucial criminalistic evidence (Tahirukaj et al., 2021). For this purpose, two main analyses can be done intensively. SEM-EDS is one of the most practical and speedy method. The technological advancements, analysis automation, advances in image processing and EDS spectra treatment software, as well as statistical techniques, have increased the feasibility of SEM analysis and the relevance of data interpretation. The GFAAS is the other alternative and cheap method. AAS also tell whether a person shot gun or not based on the amount of element or metal components present. The results obtained utilizing AAS can indicate at least 90% accuracy success (Shrivastava et al., 2021).

Due of the intense heat and pressure, GSR particles are released from the gun's barrel and muzzle blast during a shooting event. Many complicated processes are involved in the formation of these particles, which contain a variety of metal particles (aluminum, calcium, sulfur, copper, potassium, zinc, magnesium, sodium, and phosphorus, among others) as well as Sb, Ba, and Pb (Kara and Yalçinkaya, 2017).

Table 5. Analysis results of GSR according to business lines

Sample No.	Business lines	SEM-EDS analysis			GFAAS analysis		
		Total Number of Particles (pcs)	Element Combinations	Sb&Ba&Pb number of particles (pcs)	Area of the largest Sb&Ba&Pb particle (μm^2)	Sb concentration ($\mu\text{g/L}$)	10 ppb control concentration ($\mu\text{g/L}$)
1a	Phone Repairman	12724	Pb, Sb, PbBa, PbSb			1,32	9,61
1b		662	Pb, Ba			0,50	
2a	Tinsmith	25749	PbSb, Sb, Ba			0,69	
2b		331	Pb			0,67	
3a	Coppersmith	59901	PbSb, Sb, Ba			0,97	
3b		1552	Pb, PbSb, Ba			0,53	
4a	Jeweler	98763	Pb			0,49	
4b		13789	Pb, Sb, Ba			1,16	
5a	Printing Press Worker	1386	Pb, PbSb, Sb, Ba			2,51	
5b		394	Pb, PbSb, Sb			0,70	
5c		4307	Pb, PbSb, Sb, Ba			11,76	9,69
6a	Silversmith	200.000	Pb, PbSb, Ba			0,86	
6b		91531	Pb, PbBa, Sb, Ba			2,05	
7a	Blacksmith/Welder	148627	Pb, Sb, Ba			3,44	
7b		54348	Pb, Sb, Ba			0,86	
8a	Textile Worker	13253	Sb, Ba			1,36	
8b		9481	Pb, Sb, SbBa, Ba			0,99	
9a	Auto Engine Mechanic	25127	Pb, PbSb, PbBa, Sb			1,43	
9b		10481	Pb, PbSb, Sb, Ba			1,59	
10a	Auto Battery Repair	7374	Pb, PbSb, PbBa, Sb&Ba&Pb, Sb, SbBa, Ba	1	0,34	4,22	
10b		485	Pb, Ba			0,03	9,58
11a	Auto Body Repair	38233	Pb, PbSb, Sb, Ba			0,46	
11b		24297	Pb, PbSb, Sb, Ba			0,16	
12a	Auto Electrical Mechanic	22586	Pb, PbSb, PbBa, Sb, Ba			1,51	
12b		1116	Pb, Ba			-0,07	
13a	Market Employee (Cashier)	23948	Pb, Sb&Ba&Pb, Sb, SbBa, Ba	1	1,85	0,48	
13b		444	Pb, Sb, Ba			0,71	
14a	Shooting Range Employee (Shot)	200.000	Pb, PbSb, Sb&Ba&Pb, PbBa, Sb, SbBa, Ba	65671	2133,39	295,10	
14b		3315	Pb, PbSb, Sb&Ba&Pb, PbBa, Sb, SbBa, Ba	485	172,60	7,20	
15a	Person Carrying a Firearm (Has a License to Carry a Gun)	630	Pb, PbSb, Sb&Ba&Pb, Sb, SbBa	38	25,38	12,87	
15b		210	Pb, Sb, Ba			3,90	

The determination of the chemical and physical characteristics of firearm residues is crucial criminalistic evidence (Tahirukaj et al., 2021). For this purpose, two main analyses can be done intensively. SEM-EDS is one of the most practical and speedy method. The technological advancements, analysis automation, advances in image processing and EDS spectra treatment software, as well as statistical techniques, have increased the feasibility of SEM analysis and the relevance of data interpretation. The GFAAS is the other alternative and cheap method. AAS also tell whether a person shot gun or not based on the amount of element or metal components present. The results obtained utilizing AAS can indicate at least 90% accuracy success (Shrivastava et al., 2021).

Due of the intense heat and pressure, GSR particles are released from the gun's barrel and muzzle blast during a shooting event. Many complicated processes are involved in the formation of these particles, which contain a variety of metal particles (aluminum, calcium, sulfur, copper, potassium, zinc, magnesium, sodium, and phosphorus, among others) as well as Sb, Ba, and Pb (Kara and Yalçinkaya, 2017). To acquire significant data on whether a suspect has fired or handled the firearm in samples collected from his hand, quantitative and qualitative examination of the key components of GSR, Sb, Ba, and Pb, should be undertaken. Antimony analysis is common, particularly using the GFAAS approach, because the risk of contamination from the environment is low. For this reason, it was preferred to determine the amount of Sb in the swabs examined in the determination of GSR residues. In order to minimize the variables in determining the persistence of the GSR particles on the hands, the participants were asked to wash their hands for a certain time (20 seconds), and samples were taken again after drying with a napkin.

The results of SEM-EDS and GFAAS measurements were presented in Table 5. As seen in Table 1, SEM-EDS analysis of the results numbered "10a" and "13a" revealed one piece characteristic GSR particle. The obtained areas were approximately 0,34 μm and 1,85 μm , for "10a" and "13a", respectively. These particles were relatively small and were detected after careful analysis. They were also proved with GFAAS measurements. The Sb levels detected in GFAAS analyses of "10a" and "13a" were similarly low which were 4,22 and 0,48 $\mu\text{g/L}$. These findings suggest that every "Sb&Ba&Pb" particle seen on the sample may not have come from the shot. The typical GSR particle (Grima et al., 2012; Romolo et al., 2019), which was belongs to shooting range employee (14a), micrograph was presented in Figure 2.

As seen from Figure 2, the main components of particle were Sb&Ba&Pb with the weight ratio of 15,6; 19,4 and 52,2%, respectively. The homogeneous dispersion was seen on the particle for each element. The samples "14a" "14b" and "15a" according to SEM-EDS analysis, were determined as GSR particles that might be transmitted from gunshot occurrences.

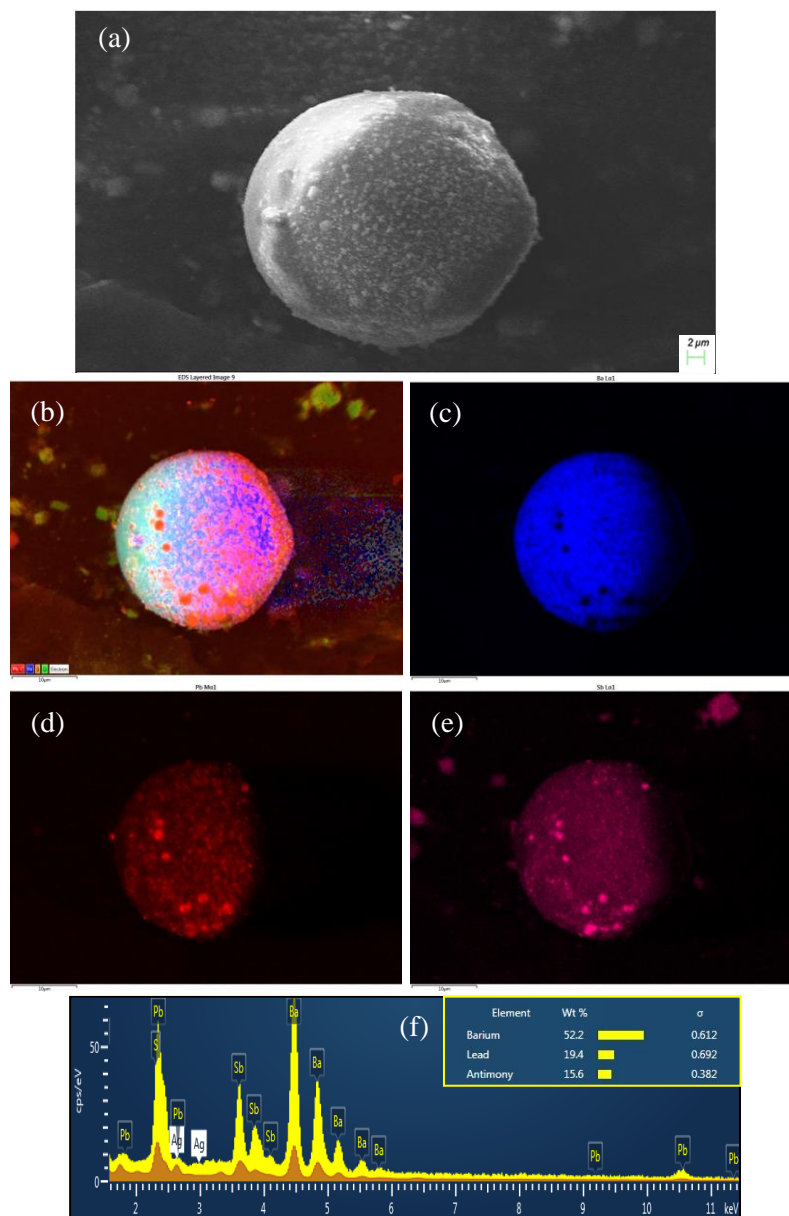


Figure 2. (a) A GSR particle with a typical spherical shape and a variety of components, (b) The energy-dispersive Xray (EDX) dot mapping of GSR particle, (c) Ba blue dot, (d) Pb red dot, (e) Sb purple dot and (f) The EDX spectra of GSR particle

In Table 5, the Sb&Ba&Pb particles numbers were 65671, 485 and 38 for "14a" "14b" and "15a", respectively. According to analysis of the standard sample, it was from the proficiency testing GSR 2005 PT by ENFSI GSR 2005 PT it was achieved by Tahirukaj et al., (2021) particles with diameter 0,5, 0,8, 1,2, and 2,4 μm were detected. The Total no of detected particles were 98, from 0,5 to 2,4 μm ; and the standard deviation (SD) was 2.06. For the particles sizes which were higher than 1.2 μm , SD was zero. During the four week for investigated particles the sensitivity varied between 97%-93%.

Romano et al., (2020) investigated the particles which were collected from both the cartridge cases and the shooters' hands after shooting tests from six volunteers fired two shots for each ammunition. Several elements, such as aluminium, potassium, silicon, sulphur, titanium and zinc were found in gunshot residue (GSR) particles. The GSR particles were found on the hands of shooters until 6 h after the shots. Particles were varied between 0,5 and 14,5 µm. The total number of particles were varied between 36 – 5 for 6h duration time.

In this study, the particles were varied between 630- 200.000 but the GSR particles number were varied from 38 to 65671 (Table 5). The GFAAS analysis confirmed the SEM-EDS results, the obtained Sb concentrations (µg/L) were 295,10, 7,20, and 12,87 for "14a," "14b," and "15a," respectively. Although the Sb, which is the main component of GSR, was discovered by the GFAAS analysis of the all samples examined, none of the other results could be detected in significant concentrations, with the exception of the findings "5c," "14a," "14b," and "15a". Despite the fact that the analysis of 5c, performed with the GFAAS technique, revealed the amount of Sb was 11,76 µg/L, in the analysis of SEM-EDS, there was not any "Sb&Ba&Pb" characteristic particle was seen. These findings revealed that not all Sb-positive results recorded by the GFAAS were caused by the gunshot.

4. Conclusion

Hand swabs from persons working in fifteen various business lines were gathered for this study and were investigated using SEM-EDS and GFAAS analyses to provide a scientific response to the question of whether people will have lead residue in their hands in daily life. Since the Sb concentration determined by GFAAS analysis is quite high compared to other samples, the focus is on the samples "14a", "14b" and "15a". Furthermore, the results of the analysis with SEM-EDS showed that the numbers of characteristic particles in these samples ("14a", "14b" and "15a") were higher and the surface areas were larger. The typical GSR particle was not detected in the SEM-EDS analysis for "5c," on the other hand, but we observed the positive result achieved by the GFAAS. This finding demonstrated that, despite the presence of Sb in the sample, it could not be classified as GSR. On the basis of SEM-EDS and GFAAS analysis of GSR suspected samples, we also investigated "the washing effect," with the findings showing that gunshot remains on samples taken after washing the hands either vanished completely or were significantly reduced, particularly for "14b."

As a consequence, it was advised that the detected antimony element concentration should be above a threshold value when evaluating the positive AAS analysis result. Furthermore, while evaluating the SEM-EDS analysis results, it has been advised that the discovered distinctive particle counts, surface areas, and morphologies be taken into account.

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Conflict of Interest

There is no conflict of interest between the authors.

Contribution of authors

All authors contributed equally to the experiments and writing of the text.

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