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Araştırma Makalesi / Research Article

GSR Analysis by Spectrophotometry Method in Forensic Chemistry

Adli Kimyada Spektrofotometri Metoduyla GSR Analizi

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ABSTRACT

This study was conducted to analyze residual shots from forensic chemistry applications. The entire analysis process was evaluated with Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy (SEM/EDS) and, Graphite Furnace Atomic Absorption Spectrometry (GFAAS)devices, which are among the methods for obtaining efficient results, and positive results were tried to be obtained by comparison. Following the study, it was discussed which results were more appropriate; in particular, the samples' appropriateness for reanalysis was underlined. The presence of shot residues on all samples was determined in the analyses performed with the SEM/EDS device. When the same samples were analyzed with the GFAAS device, the results remained below the threshold value of 10 µg/L, which is the antimony concentration indicating the presence of shot residues. It should be noted that the values we obtained may vary with the size and weight of the shot residue particles. Due to the ease of sample collection, the capacity to identify elements other than gunshot residues on the sample under examination, and the circumstances surrounding the determination of particles below the threshold value, the analysis method utilizing the SEM/EDS device has been deemed more suitable despite its higher cost.

ÖZ

Bu çalışma, adli kimya uygulamalarından atış artıkları analizlerine yönelik olarak yapılmıştır. Verimli sonuçlar elde etme yöntemlerinden olan Enerji Dağılımlı X-Işını Analizörlü Taramalı Elektron Mikroskobu (SEM-EDS) ve Grafit Fırınlı Atomik Absorpsiyon Spektrometresi (GFAAS) cihazları ile analiz surecinin tamamı değerlendirilmiş, elde edilen verilerin mukayeseleri yapılarak olumlu sonuçlar elde edilmeye çalışılmıştır. Analiz işlemi bitiminde hangi sonuçların daha sağlıklı olduğu ele alınmış,

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numunelerin özellikle tekrar analiz etmeye uygunluğu üzerinde durulmuştur. SEM/EDS cihazı ile yapılan analizlerde tüm numuneler üzerinde atış artıklarının varlığı belirlenmiş, ancak aynı numuneler GFAAS cihazı ile analiz edildiğinde sonuçlar, atış artıkları varlığına işaret eden antimon derişimi olan 10 µg/L'lik eşik değerin altında kalmıştır. Elde ettiğimiz değerlerin atış artığı taneciklerinin büyüklük ve ağırlıklarıyla değişkenlik gösterebileceği unutulmamalıdır. SEM/EDS cihazı ile analiz yöntemi her ne kadar maliyetli bir yöntem olsa da numune toplamadaki kolaylık, analiz sürecine ekstra hiçbir işlem yapmadan geçiş ve analizlemede numune üzerindeki atış artıkları haricindeki başka elementleri de tespit etme özelliği ile derişimi eşik değer altındaki tanecikleri de belirleme durumlarından dolayı daha uygun olduğu değerlendirilmiştir.

1. INTRODUCTION

Firearms-related incidents are among the most dangerous and often deadly ones that law enforcement agencies must investigate. Because certain residues may be found on a person's hands or clothing, gunshot residue analysis, or GSR, is generally a method used to assess whether a human discharged the pistol. The main elements formed in gunshot residue are lead (Pb), barium (Ba), and antimony (Sb). These main elements are commonly found together. Aluminum (Al), sulfur (S), tin (Sn), calcium (Ca), potassium (K), Chlorine (Cl), copper (Cu), strontium (Sr), zinc (Zn), titanium (Ti), or silicon (Si) can also be found [1]. It should not be forgotten that with the detonation of a weapon, not only the person who pulls the trigger, but also those who are close to it, can form shot residue on it, and this should be considered in forensic evaluations [2]. The elemental analysis and imaging systems were used to determine Ba, Pb, and Sb. When determining the GSR on the skin, clothing, wound entrance, or any other material discovered on the target, SEM/EDX is regarded as a reliable tool [3].

The SEM-EDX method provides both morphological data and particle compositions [4]. For this reason, this imaging and detection technique is gaining acceptance in forensic laboratories. The detected particles are classified on their distinctive morphology and elemental composition. One of the most crucial phases of the analysis is the transfer of GSR from the surfaces [5]. The transfer mechanism must be straightforward, easily implementable, trustworthy, and safe for human health. The collection efficiency of adhesive stubs for positive GSR results is statistically significant. So the probable reasons for this are regarded [6].

Establishing the conditions for sample preparation and analysis necessitates evaluating the sample origin. SEM/EDX analysis requires carbon/gold coating to increase the sampling period and to keep the whole analysis period shorter. The electronic charge observation throughout the automatic run is fewer. By using environmental SEM or an SEM operation with low-voltage mode, the non-conducting specimens can be imaged uncoated. Inversely, the GFAAS analysis methods are minimally affected by sample conditions. So GFAAS methods for the determination of Sb, Ba, and Pb at gunshot residue are relatively simple and cheap. However, the morphological structure of the GSR particle cannot be taken into consideration by GFAAS analysis. Therefore, the likelihood of a false-positive result is increased. To avoid this circumstance, it is crucial to identify the sample sources [7]. The morphological and elemental indicators can individuate each ammunition from others. Therefore, by reducing unintentional sample contamination, a firearm discharging a round, round case, and bullet components can be recognized. [8].

Thirteen distinct samples from six different weapons were examined for our study. Antimony was used for this study because it had a lesser risk of contamination than both Pb and Ba. GFAAS and SEM/EDX have determined all the components of interest in gunshot residue by stubbing method.

2. MATERIALS AND METHODS

2.1 Instrumentation

The AAS (PerkinElmer® AAnalyst™ 600) equipped with a background corrector (Zeeman), a graphite furnace with THGATM pyrolytically coated graphite tubes, and an autosampler (PerkinElmer AS-800 autosampler) were optimized with operational circumstances and analytical criteria listed in Table 1.

Table 1. Sb analysis instrument operating conditions.

INSTRUMENTATION		A PerkinElmer® AAnalyst™ 600 AAS		
Lamp		A PerkinElmer® Lumina™ Sb		
Wavelenght		217,6 nm		
Slith Width		0,7 nm		
Dispensed Sample Volume		20 µL		
Step	Temperature (ºC)	Ramp Time (s)	Hold Time (s)	Argon Gas Speed (L/s)
1	110	1	20	0,25
2	130	15	20	0,25
3	700	10	15	0,25
4	2300	0	5	0
5	2450	1	3	0,25
TOTAL PROGRAM TIME: 90 s				

Zeiss Co., United Kingdom, Zeiss EVO LS-15 with X-Max 50 X-Ray Detector (SEM/EDS) was used for imaging and other elemental analysis. The operating circumstances are ideal, as shown in Table 2.

Table 2. SEM/EDS Operating Conditions for stubbed swap analysis.

Voltage	15.0 kV
Working Distance	39 mm
Take-off Angle	35.0^o
Distance	39mm
Elapsed Lifetime	100.0 s

2.2 Standart Solutions and Reagents

Sb standard solution of 1000 mg L^{-1} for AAS from E. Merck (Darmstadt, Germany) was purchased. New Human Power I Scholar-UV (Human Co. Made in Korea) was used for water distillation. The standard solutions of 5, 10, 20, 30, and 40 mg L^{-1} Sb in 1% nitric acid were prepared by dilution of the 1000 mg L^{-1} Sb standard. Nitric acid (65%, Merck) was of analytical purity.

2.3. Sample Collection

Because of its better recovery, TAAB 12 mm diameter carbon discs with 0.5 inch aluminum stubs (TAAB Co, UK) were used for sampling. 30 mL cylindrical polypropylene boxes of 2.5 cm x 5.5 cm (LP Italiana Spa, Italy) were benefited as swab sample containers.

Sixteen adhesive TAAB Stub Kit swabs were collected from six different firearms (Table 3). The Control Group Samples were stabbed and controlled for GSR persistence absence confirmation.

Table 3. Stub Kit swaps taken from the samples.

CONTROL GROUP	Sample 1	Calibration Test kit
	Sample 2	Clean 1 (Unused sample-cartridge)
	Sample 3	Clean 2 (clean sample taken from the firing hand)
SARSILMAZ 9mm pistol (Türkiye)	Sample 4	Sterling (STR) pistol cartridge (9x19mm)
	Sample 5	Codex (CDX) pistol cartridge (9x19mm)
	Sample 6	Ozkursan (OZK) pistol cartridge (9x19mm)
	Sample 7	Yavex (YVX) pistol cartridge (9x19mm)
	Sample 8	MKE pistol cartridge (9x19mm)
Browning 7,65 mm pistol (Belgium)	Sample 9	GECO pistol cartridge (7,65 mm)
	Sample 10	MKE pistol cartridge (7,65 mm)
	Sample 11	OZK pistol cartridge (7,65 mm)
G3 A3 7,62mm Infantry Rifle (Türkiye)	Sample 12	MKE 7,62x51 mm rifle cartridge
AK 47 7,62 mm Infantry Rifle (Russia)	Sample 13	Sterling 7,62x39 mm rifle cartridge
Heckler&Koch 5,56mm HK 33 Rifle (Germany)	Sample 14	MKE 5,56x45 mm rifle cartridge
Fumsan Semi-Automatic 12 caliber Hunting Rifle (Türkiye)	Sample 15	Sterling 12 caliber Hunting Rifle Cartridge
	Sample 16	Yeni-Tür 12 caliber Hunting Rifle Cartridge

2.4. Sample Preparation

Quantitative elemental detection of antimony by GFAAS is one of the analytical GSR detection techniques. The studies were conducted using sixteen different TAAB Stub kits (see Table 3). To replicate samples that the investigators will submit for analysis, samples from the firing hand were swabbed. The samples in the boxes were shaken at 200 rpm speed for 30 minutes with 5 mL 5% nitric acid (v/v) for Sb determination.

The samples were first cleaned with 10 mL acetone in an ultrasonic bath for two minutes before to the examination of the adhesive content. For determination of Sb, the acetone was evaporated and then dissolved in 4 mL of 8% nitric acid (v/v).

For samples 2, 3, and 4, the stubbed swabs were taken on the first day. The remaining stubbed swabs from samples 5 to 16 were obtained once each day daily. Five minutes after the last shot was fired, swaps from the shooters' hands were taken.

3. RESULTS AND DISCUSSION

Technically, both the primer and the gunpowder that formed the main charge in the cartridge are subjected to rapid combustion by firing a weapon. The emerging residues resulting from this combustion are called gunshot residues. After shooting, the remains dispersed in the shape of a white cloud either by adhering to nearby surfaces or remaining on the hands. In the resulting GSR, there are either burned or unburned remains of both the primer and the gunpowder.

In addition to the condensation particles made from the metals employed in the primer, cartridge, and projectile, GSR is a heterogeneous cloud of propellant particles in various stages of combustion [1].

Because of the propellant’s organic character, it is difficult to make a definite identification of GSR from the propellant residue, while it has been reported that some screening methods are acceptable [9,10]. In some cases, only organic components can be detected when the inorganic GSR was not found. In such cases, the organic components, that provide additional information about the sample, help to differentiate GSR from environmental residues. Micron-size inorganic GSR particles are commonly collected with adhesive tape. These tapes lift both the GSR particles and those of environmental origin residues as well [4]. Inorganic residue detection is broadly practiced in forensic laboratories. Today many scientists have begun to concentrate on organic components of GSR derived from propellants.

The limits of detection (LOD) and limits of quantification (LOQ)for each metal were determined as follows: 10 independent analyses of a blank solution spiked with the metal at a level of lower concentration of the analytical curve were performed. The LOD and LOQ were calculated from the standard deviation (s) of these determinations (LOD = 3 x s and LOQ =10 x s) [11].

In the current investigation, the relative standard deviation (%RSD), limit of detection (LOD), and limit of quantitation (LOQ) values of the calibration curves displayed in Table 4 served as the foundation for the confidence parameters produced by GFAAS.

Table 4. Confidence Parameters Based on the Calibration Curve

%RSD	**4,88 µg L^{-1}**
m (slope)	0,0045
n (slide)	0,0046
Linear Range	0-40 µg L^{-1}
S (Standart Deviation)	0,003437
R^2	0,9991
LOD	**0,95** µg L^{-1}
LOQ	**2,85** µg L^{-1}

When a firearm has been fired, there are at least three particles (Sb, Pb, and Ba) of characteristic morphology determined. Consistent particles are two-component particles that can be originated from the environment [12]. The consistent category formation, such as Pb and Sb or Pb and Ba, has been determined in a few occupational residues. These compositions can be characterized only as consistent but not unique [13]. The relevant particles are at least one characteristic particle together with other elements. The results of the analysis with SEM/EDX for characteristic, consistent, and relevant particles are shown in Table 5.

Table 5. The number of characteristic, consistent, and relevant particles

Sample	Number of characteristic particles	Number of Consistent particles	Number of relevant particles
1	126	-	-
2	-	-	-
3	-	-	-
4	117	108	398
5	12	7	66
6	74	43	283
7	135	219	1940
8	71	28	264
9	38	15	142
10	218	154	649
11	41	33	112
12	1	1	24
13	3	5	27
14	2	1	18
15	6	4	35
16	1	1	2

The spectra and images of specified characteristic particles are listed in Figures 1-14**.**

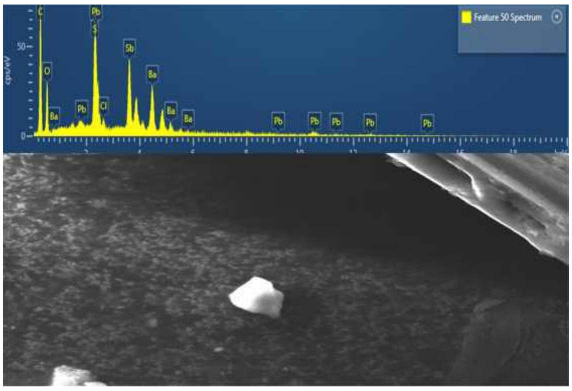

Fig. 1. *The spectra and image of characteristic particle in Sample 1.*

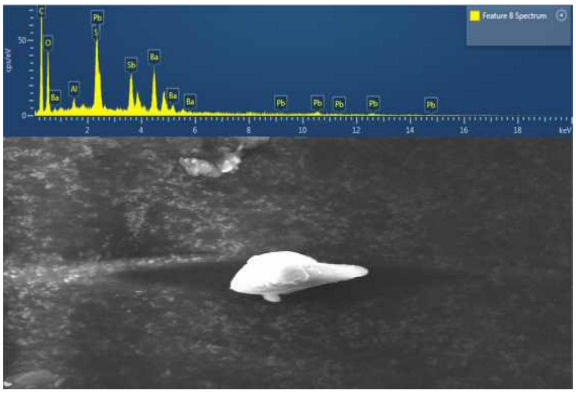

Fig. 2. *The spectra and image of characteristic particle in Sample 4.*

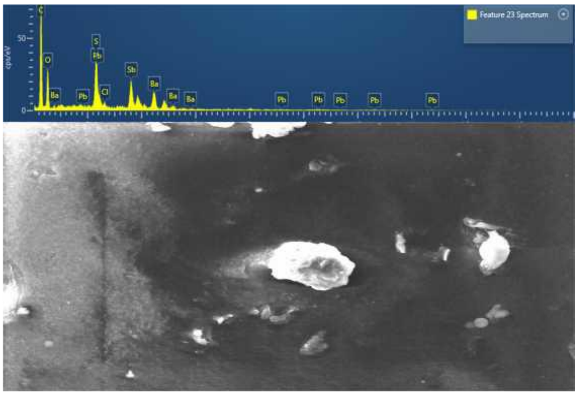

Fig. 3. *The spectra and image of characteristic particle in Sample 5.*

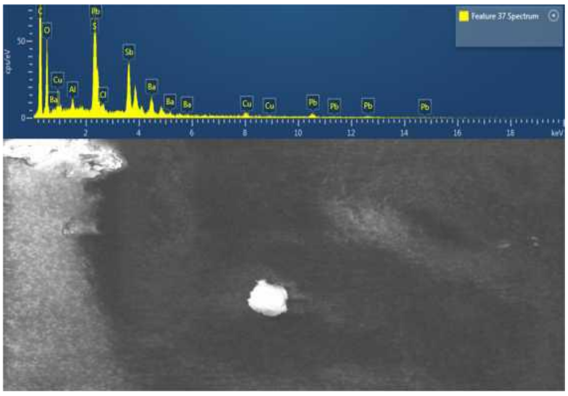

Fig. 4. *The spectra and image of characteristic particle in Sample 6.*

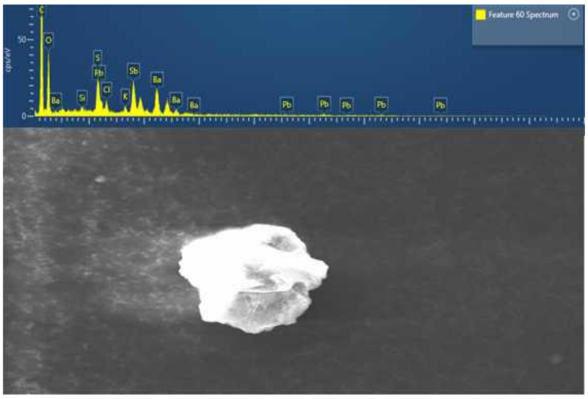

Fig. 5. *The spectra and image of characteristic particle in Sample 7.*

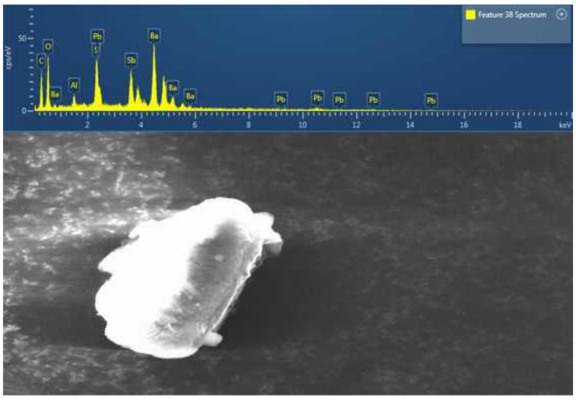

Fig. 6. *The spectra and image of characteristic particle in Sample 8.*

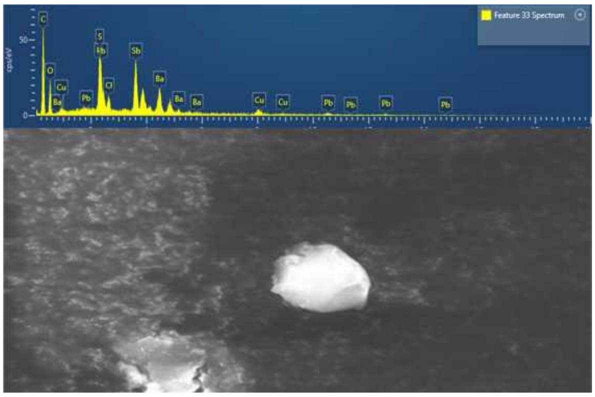

Fig. 7. *The spectra and image of characteristic particle in Sample 9.*

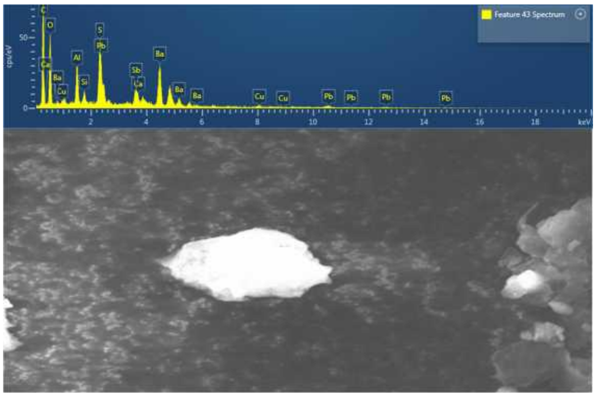

Fig. 8. *The spectra and image of characteristic particle in Sample 10.*

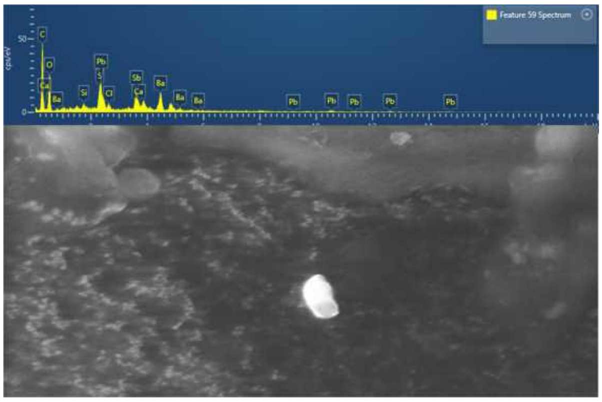

Fig. 9. *The spectra and image of characteristic particle in Sample 11.*

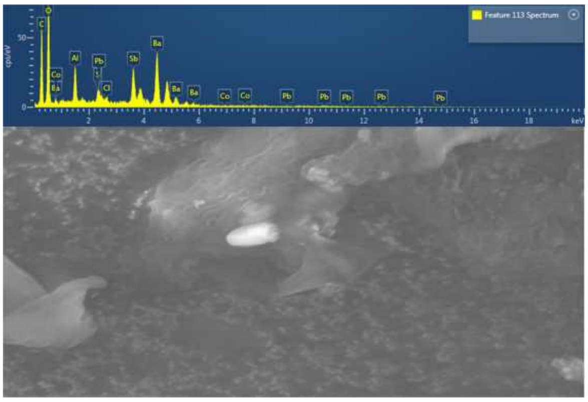

Fig. 10. *The spectra and image of characteristic particle in Sample 12.*

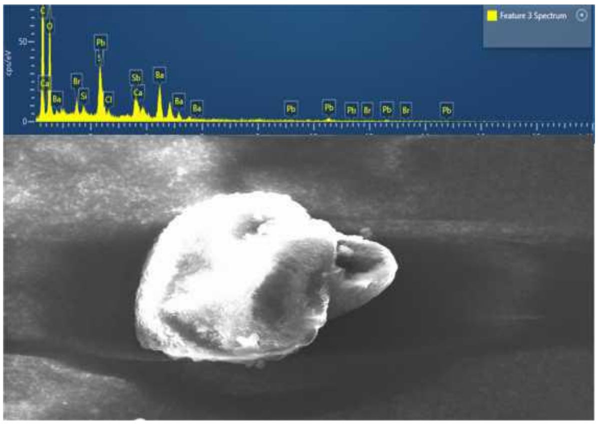

Fig. 11. *The spectra and image of characteristic particle in Sample 13.*

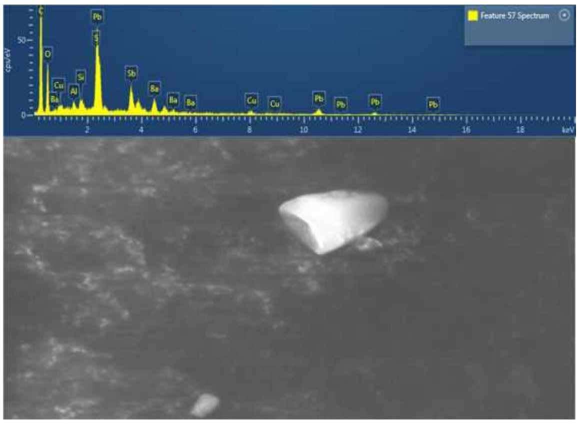

Fig. 12. *The spectra and image of characteristic particle in Sample 14.*

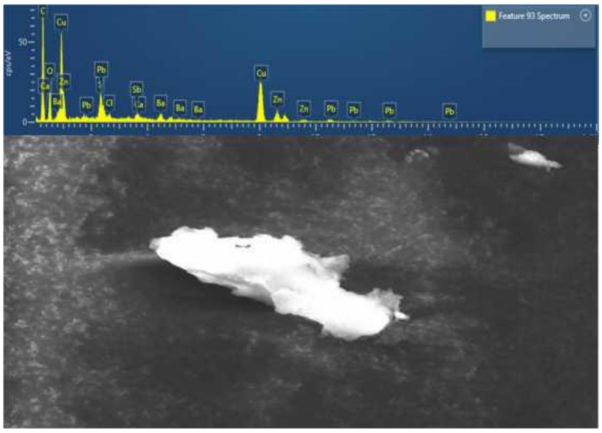

Fig. 13. *The spectra and image of characteristic particle in Sample 15.*

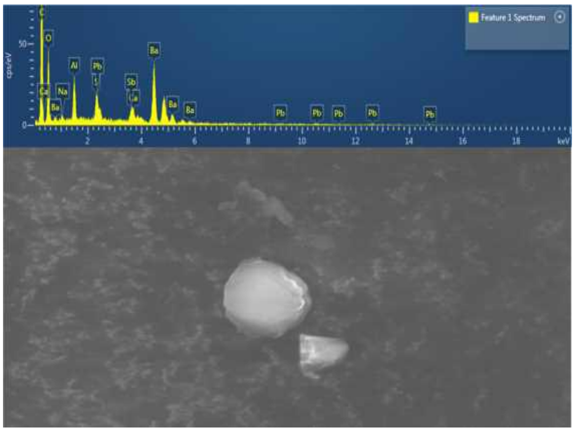

Fig. 14. *The spectra and image of characteristic particle in Sample 16.*

The GFAAS analysis results of the samples are shown in Table 6.

Table 6. The samples' GAAFS analysis results

Sample	Absorbance	Antimony Concentration (μg L^{-1})
2	0,0072	0,58
3	0,0081	0,78
4	0,021	3,62
10 μg L^{-1} Sb	**0,067**	**10,98**
5	0,014	2,83
6	0,027	4,68
7	0,025	5,07
10 μg L^{-1} Sb	**0,051**	**9,25**
8	0,021	4,14
9	0,02	3,57
10	0,03	4,98
10 μg L^{-1} Sb	**0,061**	**11,26**
11	0,024	3,34
12	0,009	1,34
13	0,014	2,37
10 μg L^{-1} Sb	**0,053**	**10,74**
14	0,008	1,15
15	0,011	2,18

Table 6 Continued

16	0,01	1,23
10 µg L^{-1} Sb	**0,057**	**10,92**

The GFAAS analysis results of the Sb and Pb samples in a certain proportion are shown in Table 7.

Table 7. The GFAAS analysis results of the Sb and Pb samples in a certain proportion

Mixing Ratio	Sb Absorbance in Mixture	The Absorbance of *10 µg L^{-1} Sb* Standart	Recovery (%)
1:1 (1 ml 10 µg/L Sb – 1 ml 10 µg/L Pb)	0,0498	0,0526	94,68
1:5 (1 ml 10 µg/L Sb – 1 ml 50 µg/L Pb)	0,0525	0,0547	95,96
1:10 (1 ml 10 µg/L Sb – 1 ml 100 µg/L Pb)	0,0478	0,0491	97,35
1:20 (1 ml 10 µg/L Sb – 1 ml 200 µg/L Pb)	0,0451	0,0488	92,42
1:50 (1 ml 10 µg/L Sb – 1 ml 500 µg/L Pb)	0,0474	0,0512	92,58
1:100 (1 ml 10 µg/L Sb – 1 ml 1000 µg/L Pb)	0,0468	0,0498	93,98

The GFAAS analysis results of the Sb and Ba samples in a certain proportion are shown in Table 8.

Table 8. The GFAAS analysis results of the Sb and Ba samples in a certain proportion

Mixing Ratio	Sb Absorbance in Mixture	The Absorbance of *10 µg L^{-1} Sb* Standart	Recovery (%)
1:1 (1 ml 10 µg/L Sb – 1 ml 10 µg/L Ba)	0,4644	0,4935	94,1
1:5 (1 ml 10 µg/L Sb – 1 ml 50 µg/L Ba)	0,4554	0,4675	97,41
1:10 (1 ml 10 µg/L Sb – 1 ml 100 µg/L Ba)	0,4736	0,4995	94,81
1:20 (1 ml 10 µg/L Sb – 1 ml 200 µg/L Ba)	0,4364	0,4712	92,61
1:50 (1 ml 10 µg/L Sb – 1 ml 500 µg/L Ba)	0,4832	0,4974	97,15
1:100 (1 ml 10 µg/L Sb – 1 ml 1000 µg/L Ba)	0,4771	0,4875	97,87

4. CONCLUSION

SEM/EDS and GFAAS are widely used in the detection of GSR in the firing hands of shooters. Our study is aimed to evaluate the strengths and weaknesses of both devices. When data we obtained from the analysis are examined, it is seen that the optimum parameters of devices are consistent.

After the shots were made with 13 different cartridges in total, GSR was determined at all samples in SEM/EDS analyses. All samples were collected by a single contact from the firing hand. When these samples were analyzed at GFAAS, concentrations less than the threshold value of 10 μg/L, which is accepted as an indication of the presence of GSR, were obtained. Even if the number of particles detected by the analysis of different samples with SEM/EDS is the same, the same values may not be obtained in GFAAS due to the difference in particle size and weight.

A low number of GSR particles in samples obtained by firing a long-barreled weapon is because the barrel length and the distance of the cartridge case ejection part from the firing hand are greater than those of the pistols.

While the morphological structures of the particles can be determined by SEM/EDS, this is not possible by GFAAS. It is stated in the literature that this special morphological spherical structure of particles, does not generally interact with the tissue, has a decisive feature, and belongs only to GSR [14-18]. While antimony in the structure of some materials such as polyester may cause false-positive results by GFAAS, this is not the case with the SEM/EDS [7]. It should be considered that the half-life duration for the amount of gunshot residues on the skin depending on various environmental conditions is approximately 1 hour and the particles can be determined for up to 4 hours [8, 20, 21].

Considering the whole process, the sample obtaining convenience and the resulting sensitivity at the analysis make the SEM/EDS stand out. Although the analysis at SEM/EDS is costly, it is a very important factor that SEM/EDS detects not only antimony but all elements in the unique analysis.

Other elements found next to the detected element may cause chemical interference. This can lead the element concentration of the element analyzed by GFAAS to be seen at either lower or higher levels. Thus, we have chosen Sb, Pb, and Ba which form the main components of GSR. With the results we obtained, an absorbance decrease was observed with the increase of Pb, Pb, and Ba ratios. Emphasis was placed on Sb analysis that Pb and Ba characteristics must be considered as well. At the mixtures of 1:1000 and higher ratios, studies stated that some changes of 10% and above in recovery were observed.

If the samples were obtained by 25 cm^2 (5x5 cm) double-sided adhesive tapes by touching the surface many times instead of 12mm diameter (4,5 cm^2) stub kits by only one touch, we could have obtained 5 times more particles because of the total surface area of adhesive tape. SEM/EDS can easily determine every element while each separate element needs a preparation for GAAFS analysis which is time-consuming and high cost.

The samples for GAAFS are usually obtained with 5x5 cm adhesive tapes. Sometimes the sticky sides can stick to the sample boxes or stick to the other areas of the same tape. This situation reflects negatively on the analysis results. It can be easily understood from our present study that the samples for SEM/EDS can be obtained easily and practically without any negative situation stated above for GAAFS.

The samples remain in good condition for further analysis after being analyzed by SEM/EDS. However the samples analyzed by GAAFS are no longer suitable for further analysis. Additionally, the prepared samples are affected negatively when a sudden malfunction occurs in GAAFS while the samples can be analyzed many times without any additional process in SEM/EDS.

This study represents the superiority of SEM/EDS over GFAAS for GSR analysis when considering the easiness and simplicity in sample collection, other elements detection, and determination of particles below a threshold value.

CONFLICTS OF INTEREST

The authors confirm that this article's content has no conflict of interest.

AUTHORS' CONTRIBUTIONS

Gökhan TEKİN and Fırat AYDIN: Conceptualization, methodology, validation, writing-review and editing, development or design of methodology, supervision, data interpretation. Ercan SEYHAN and Isil AYDIN: Writing-original draft preparation, data collection, data curation, visualization, data interpretation, development or design of methodology, data presentation.

DECLARATION OF ETHICAL CODE

In this study, the authors undertake that they comply with all the rules within the scope of the "Higher Education Institutions Scientific Research and Publication Ethics Directive" and that they do not take any of the actions under the heading "Actions Contrary to Scientific Research and Publication Ethics" of the relevant directive.

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