Qualitative and Quantitative Differences between GSR and non-GSR Source Particles Revealed by Raman Spectroscopy

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Abstract: - Analysis of Gunshot Residue (GSR) is relevant to forensic science by providing a correlation between an individual and a firearms incident. Detection of GSR is usually made by using Scanning Electron Microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (SEM-EDX). Particles originating from some manufacturing trades could represent false positive threats to SEM-EDX analysis of GSR. In particular, GSR-like particles arising from brake pads are very similar in composition. In this study, we propose a non-destructive complementary method for the discrimination of GSR samples by using Raman Spectroscopy. The analysis of Raman spectra obtained from GSR and brake pads is able to differentiate the origin of the particles in a qualitative and quantitative manner. In particular, the center frequency of the G-band is shifted to lower frequencies for brake pad dust; on the other hand, the center frequency of the band is shifted to higher frequencies for GSR samples. In addition, to assess quantitative changes due to chemical structural modifications, we computed the intensity ratio R between the detected band centered around 1350 cm^{-1} and 1585 cm^{-1}, respectively. The R values for GSR samples were, in all cases, lower than the R values computed for brake pad samples. In addition, scanning SEM was employed to characterize the morphology of the samples, coupled with EDX to determine the elemental composition of the sample surface. Since elemental compositions detected via traditional SEM-EDX can overlap between brake and GSRs, detailed morphological and structural analyses are mandatory for an unambiguous attribution. Although SEM-EDX remains the gold standard for GSR analysis, this study demonstrates that its combination with Raman spectroscopy provides a crucial advantage. Leveraging its non-destructive nature, Raman spectroscopy offers vital complementary

characterization, establishing a promising framework to enhance the reliability of traditional forensic technical investigations.

Key-Words: - Raman Spectroscopy, Gunshot Residues, Brake Pads, Frequencies shifts, SEM-EDX, Vibrational Spectroscopy.

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1 Introduction

Inside the ammunition there are two different types of energetic materials that cover different roles: the priming mixture and the launch charge. The action of the firing pin on the bottom of the primer capsule causes the energy substance to be ignited. The resulting explosion in the volume of the trigger capsule induces adiabatic conditions for just over a millisecond. The thermobaric evolution involves peaks in temperature (from 2900°K to 3400°K) and pressure (from 30 to 60 atm), sufficient to induce the sublimation (transition from the solid to the gaseous state) of the metallic salts present, the consequent condensation and formation of protocorpuscles. The latter, confeyed through the flash hole, ignites the launch charge, causing it to explode. When a gunshot explodes, a complex cloud of particulate matter emerges from the weapon, made up of a mixture of chemical substances which includes not only residues from the primer and gunpowder, but also elements derived from the weapon, from the bullet and the cartridge case, [1]. Gunshot residue (GSR) is the name of this complex mixture of organic and inorganic particles, [2]. GSRs can be divided into inorganic (IGSR) and organic (OGSR). The inorganic components of gunshot residue (IGSR) are mainly derived from the used primer and the projectile, while the organic components of gunshot residue (OGSR) are derived from the propellant and stabilizers, [3], [4]. The most common characteristics of IGSR particles contain the elements lead (Pb), barium (Ba) and antimony (Sb) [5], but also irregular particles with different concentrations of aluminum (Al), silicon (Si), magnesium (Mg), potassium (K), sulfur (S), iron (Fe), titanium (Ti), copper (Cu), nickel (Ni) and zinc (Zn) [6], while the organic compounds, coming from the powder propellant, the priming mixture and/or the black powder, are mainly nitrocellulose, nitroglycerin, nitroguanidine, diphenylamine, ethyl-centralite, akardite, phthalates and dinitrotoluene (therefore based on carbon, oxygen, hydrogen and nitrogen). GSR is relevant to forensic science. In fact, a correct determination of both IGSR and OGSR provides a correlation between an individual and a firearms incident. After the shooting, discharged particles are deposited around several locations of the shooting incident, including the body and clothing of the shooter [7] and, for this reason, particulate samples are collected from the hands and clothes of suspects.

However, environmental and occupational sources of particles could be confused with GSR, thus "generating" false positives, [8]. In particular, many studies have found that the dust produced during the friction of brake pads contains particle residues that share similarities with particles found in GSR, [9], [10], [11].

Moreover, the elements that are found in the particles of GSR have several commercial and industrial applications, for example in batteries, solder, paint, armor, glass, dyes, paints, paper products, brake pads, and fireworks, [12], [13].

This scenario is relevant because many people could be exposed to particles that can be confused with GSR because of changing a wheel.

In forensic applications, different methods for collection and detection of GSR are used. The analysis of primer gunshot residue (pGSR) traditionally relies on automated Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectrometry (SEM-EDX), in strict accordance with the ASTM E1588 standard practice included in the NIST OSAC Registry of forensic standards, [14]. This technique is well suited for detecting small particles containing heavy metals such as Pb, Ba, and Sb, and it allows for the determination of the correlation between the morphology and the chemical composition of individual particles, [15], [16]. However, these elements are not exclusively found in GSR.

In addition, this technique requires excessive time, sampling procedures, and instrumentation, [17].

On the other hand, different techniques are emerging in forensic science. In particular, Infrared spectroscopy and Raman spectroscopy represent valuable methodologies in GSR analysis, [14]. Infrared Spectroscopy is usually used for the detection of organic compounds of GSR [18], while Raman Spectroscopy represents a feasible approach for the study of gunfire traces, [19].

In this study, we employed Raman Spectroscopy to obtain information about any possible difference in the vibrational spectra of GSR and brake pad samples in order to establish if it is possible to distinguish between them. In fact, Raman spectroscopy is an inelastic light-scattering phenomenon that provides vibrational spectra containing information on the chemical bonds and the symmetry of a specific molecule, [20], [21]. For this reason, Raman spectroscopy is considered the "fingerprint" of material identification, [22]. It is commonly used in different research fields such as human and veterinary medicine, cultural heritage, and chemistry, [23], [24], [25], [26]. In fact, the Raman spectroscopy technique does not require reagents to perform measurements, and it is very easy to execute. The obtained Raman spectra provide abundant information about structural composition in a single step; in particular, Raman spectroscopy provides detailed information regarding vibrational, rotational, and other low-frequency transitions within molecules, making it a powerful tool for molecular characterization, [27]. Any alterations in the molecular environment, such as structural changes or interactions, are reflected in the Raman spectrum as variations in peak position, width, and intensity. These changes offer valuable insights, allowing for the identification and analysis of molecular properties, interactions, and transformations, thus making Raman spectroscopy highly sensitive to even subtle shifts in molecular structure or composition.

In this preliminary study, we characterized GSR and Brake pads dust by using SEM technique, coupled with EDX, representing the gold standard and Raman spectroscopy to verify if it is possible distinguish between GSR and brake pads dust, providing an alternative methodology that even if does not represent a new gold standard, can be used coupled with SEM analysis, exploiting the fact that it is a non-destructive technique.

2 Problem Formulation

2.1 Sample Collection

Both GSR particles and brake pad dust were collected using the standard procedures prescribed for this type of investigation by international police forces, procedures that require the use of appropriate personal protective equipment (PPE - laboratory suit, latex gloves, etc.) in order to prevent any possible contamination.

The sampling process was carried out using the standard method for collecting particulate matter for analysis at the SEM, i.e., using a carbon tape placed on an aluminum support (stub). These stubs allow optimal collection of particulate matter, concentrating the particles in a circular area of 12mm diameter, ideal for subsequent SEM or Raman analysis.

To avoid contamination, the working environment and the operator performing the sampling were monitored by the use of a sterile stub (blank test), which is used to swab gowns, gloves, and work surfaces to capture any polluting particles.

A total of 20 samples were collected and analyzed as follows:
- 10 samples from samplings on 10 people after shooting
- 10 samples from samplings on 10 mechanics after working in the workshop on disc brakes.

The ten shooters fired three shots each (in an open environment), using three different pistols: 9 para-Beretta 92 FS (usually used by police forces in Italy), 9x21 Glock 45, and 357 Mag Smith & Wesson 686.

The cartridges used were produced by the Giulio Fiocchi factory (Lecco, LC, Italy). These cartridges mount standard Pb-Ba-Sb-based primers. The decision to focus our attention on this type of ammunition stems from two decisive factors: they are the most widespread on the Italian market, and previous studies have shown that they contain primers with compositions compatible with disc brake residues.

The stubs were then used by pressing their adhesive surface onto the hand holding the weapon of the ten shooters.

For the disc brake samples, samples were taken from ten different workshops of ten mechanics.

Stubs were used on the hands and clothing of the ten mechanics after they had worked on brake pads or on changing a wheel. Seven samples were taken from the hands and three samples from the overalls worn during work.

2.2 SEM Analysis

Morphological characterization was performed using two Scanning Electron Microscopes (FEG TESCAN MIRA 4 – THERMO SCIENTIFIC PHENOM XL GSR). This instrument allows for high-resolution imaging and semi-quantitative X-ray microanalysis of both conductive and non-conductive specimens at the nanometer scale. To avoid the need for conductive coating (e.g., gold or carbon), the samples were imaged in low vacuum mode (LV). The electron beam, generated at an

accelerating voltage of 15-25 kV, scanned the sample surfaces in the X-Y direction. Images were acquired in back-scattered electron (BSE) mode with a working distance (WD) ranging from 7 to 14 mm. Multiple magnifications were explored (x300-x15000). The resulting 16-bit images were saved in .tiff format without any subsequent digital processing. Elemental microanalysis was conducted via Energy-Dispersive X-ray Spectroscopy (EDX) integrated into the microscopes. This technique provided X-ray spectra where peak positions identified the elements present, and peak intensities allowed for their quantitative characterization. Compositional data, including relative errors, were generated based on the instrument's internal calibration library. Both qualitative and quantitative analyses were performed at four distinct points on each sample surface; the reported data represent the mean values of these four measurements per target sample.

2.3 Raman Analysis

Raman measurements were performed using a DXR-SmartRaman Spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). The DXR-SmartRaman Spectrometer uses a diode laser with an excitation wavelength of 785 nm. All the Raman spectra were acquired over the wavenumber range of 400–2800 cm^{-1}, with a resolution of about 2 cm^{-1} and irradiated with a laser power of 10 mW, coming out from a circular 50 μm spot. Before the measurements, the DXR-SmartRaman Spectrometer was calibrated using standard samples of known wavenumber furnished by the manufacturer.

All the stubs containing the dust collected from the shooters' hands and from mechanics were accommodated into the appropriate sample holder, and the 180-degree sampling accessory for the DXR-SmartRaman Spectrometer was used for measurements. To obtain high signal-to-noise ratio (S/R) spectra, each Raman spectrum was acquired after collecting 16 sample frames, and the duration of each exposure for each frame was set equal to 30.0 s. Total acquisition time was 8 minutes for each spectrum.

In addition, a spectrum of the blank stub was obtained in triplicate with the same acquisition parameters, and the average spectrum was computed.

For each obtained spectrum, a spline baseline correction was performed to remove the noise due to fluorescence background and scattering.

To eliminate the contribution of the blank stub, each spectrum was spectrally subtracted (dust stub minus the blank one).

The average Raman spectrum and the standard deviation were computed for shooters and mechanics.

To assess quantitative changes due to chemical structural modifications, we computed the intensity ratio of:

$$R = \frac{I_{1350}}{I_{1585}} \quad (1)$$

where R represents the intensity ratio; I_{1350} and I_{1585} are the intensity of peaks located at about 1350 cm^{-1} and 1585 cm^{-1}, respectively. Calculating R allows us to mathematically normalize the spectral data. This ratio serves as a sensitive, quantitative indicator of the specific molecular and structural configurations, providing an objective parameter to distinguish GSR from non-GSR particles.

2.4 Statistical Analysis

To examine data distribution, we have used scatterplots and boxplots. In our small-sample-size study, scatter plots were used to differentiate between the two datasets (GSR and Brake Pads), while boxplots were used to show the distribution of numerical data and skewness by displaying the data quartiles and averages.

A comparison between R values for brake pads and GSR samples was performed with a Mann-Whitney U test. A value of $p<0.05$ was considered statistically significant. All statistical analyses were performed with MATLAB®, R2020b (MathWorks, Inc., Natick, MA, USA).

3 Problem Solution

Figure 1 and Figure 2 illustrate the elemental characterization of the GSR and disc brake pads, respectively, where the traditional triad of Lead (Pb), Antimony (Sb), and Barium (Ba) emerges as the most representative and critical combination of elements.

However, a comparative analysis of the two figures reveals a significant challenge: due to the close similarity or potential overlap in the spectral data, it is not readily possible to clearly distinguish which specific sample these key elements belong to. While Figure 1 shows these dominant peaks as the definitive signature of ignition residues, the complex and inhomogeneous composition of the brake pads in Figure 2—which includes prominent peaks of Sulfur (S), Copper (Cu), Zinc (Zn), Sodium (Na), and Chlorine (Cl)—complicates the clear-cut differentiation between the two sources.

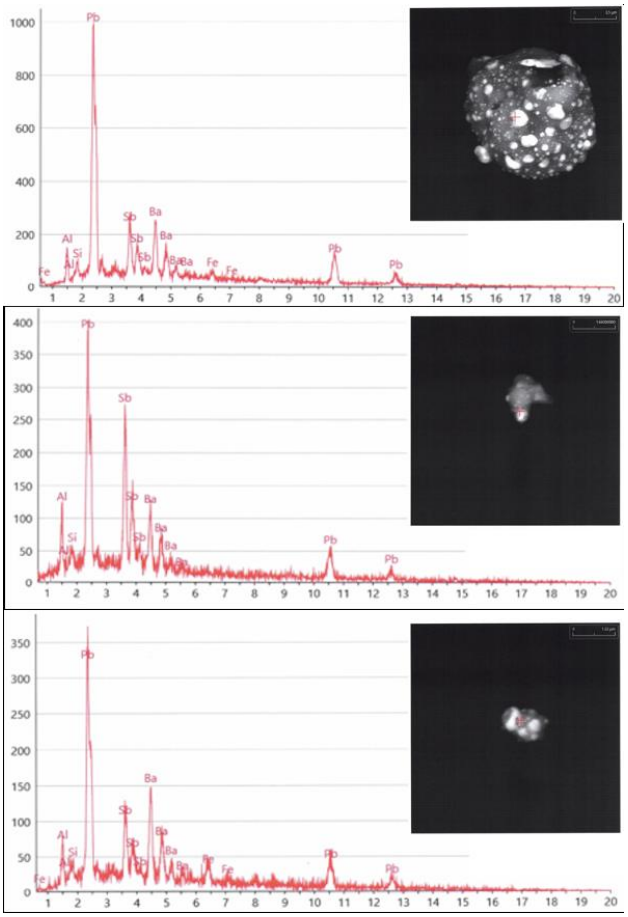

Fig. 1: SEM micrographs and corresponding EDX spectra of the analyzed particles: morphological view and EDX elemental analysis of gunshot residue (GSR) particles. In all three different acquisitions and related spectra, Pb, Sb, and Ba are the most representative elements
Source: created by the authors

In Figure 3, the Raman spectra of blank stub (a), GSR (b), and brake pad dust (c) are shown. The spectra exhibit the main features attributed to carbon [28], with two main peaks assigned to D-band (~ 1350 cm-1) and G-band (~ 1585 cm-1).

Possible contamination of the stub could induce peak position shift, peak broadening, and changes in their intensities. In this context, spectral subtraction was performed to eliminate the contribution of the blank stub. The result of the spectral subtraction is reported in Figure 4. In this figure, the spectral difference between the stub containing GSR (mean value and standard deviation) and the blank stub is indicated as a red line, whereas the difference between the spectra of the stub containing brake pad dust (mean value and standard deviation) and the blank stub is reported as a black line.

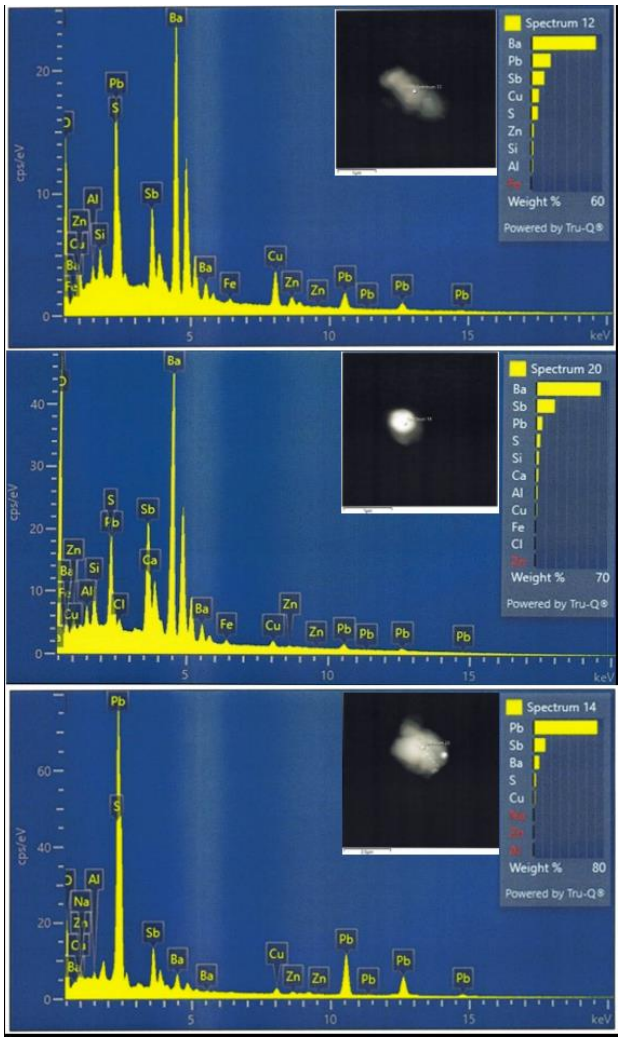

Fig. 2: SEM micrographs and corresponding EDX spectra of the analyzed particles: morphological view and EDX elemental analysis of brake pad particles. Pb, Sb, and Ba are the most present elements
Source: created by the authors

Figure 5 describes the discrete distribution of data by using a box plot graph. The box represents the first and third quartiles of the data. Inside it, the median value is drawn as a horizontal line. The whiskers only extend to the most extreme observations within 1.5 times the difference between the third quartile and the first one. The observed data points outside the boundary of the whiskers are plotted as outliers. In the same figure, for all the analyzed samples, the R ratio is reported as a scatter plot. The scatter plots show the differences between the measurement data, allowing the authors to quickly assess their magnitude and distribution.

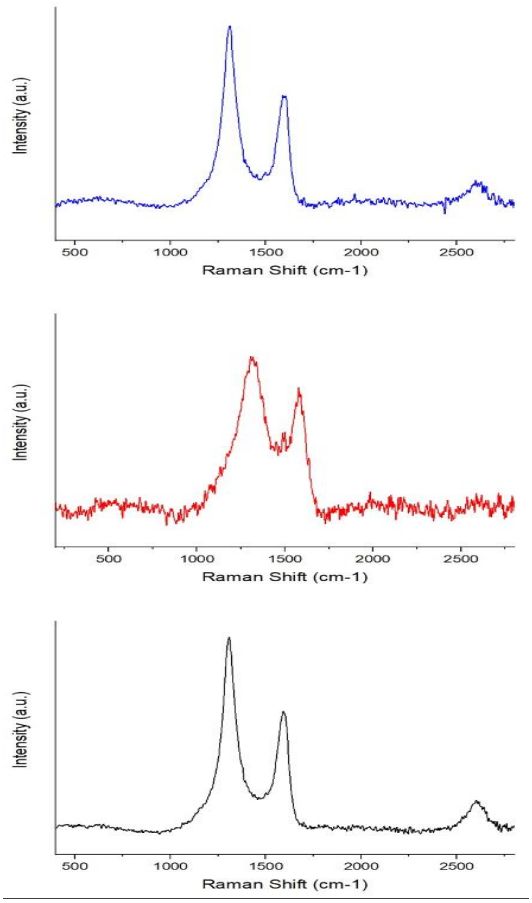

Fig. 3: Raman spectra of blank stub (a), GSR (b) and brake pad dust (c)
Source: created by the authors

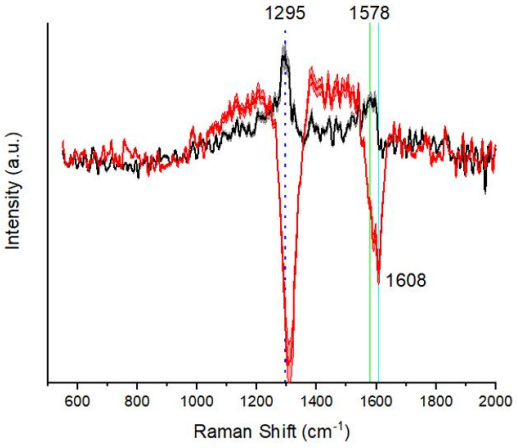

Fig. 4: Spectra differences (mean value and standard deviation) between brake pad dust minus blank stub (black line) and GSR minus blank stub (red line). The vertical blue dotted line is related to the center frequency of the D band; the vertical green line is related to the center frequency of the G band of the resulting spectrum of brake pad dust minus blank stub; the cyan vertical line is related to the center frequency of G band of the resulting spectrum of GSR minus blank stub
Source: created by the authors

The relevance of the ratio R as a quantitative discriminator is clearly demonstrated by the statistical analysis of our experimental data. As shown in Figure 5, the R values calculated for GSR samples were consistently and significantly lower than those obtained for brake pad residues ($p<0.05$). The statistical parameters extracted from the distribution of the R ratio highlight a clear-cut separation between the two analyzed populations. For the brake pad group, the data exhibited a tight distribution centered around a median R value of approximately 1.79, with the first and third quartiles spanning from 1.64 to 1.91, and a single isolated outlier. The GSR group demonstrated a distinct, lower distribution, with a median R value of approximately 1.30 and quartiles ranging from 0.99 to 1.50. The analysis of the averages and the non-overlapping quartile intervals between the two categories mathematically confirms the structural differences of the samples. The Mann-Whitney U test confirmed that the difference between the median R values of GSR and brake pad dust is highly significant ($p < 0.05$), statistically validating that the two groups belong to distinct, non-overlapping populations.

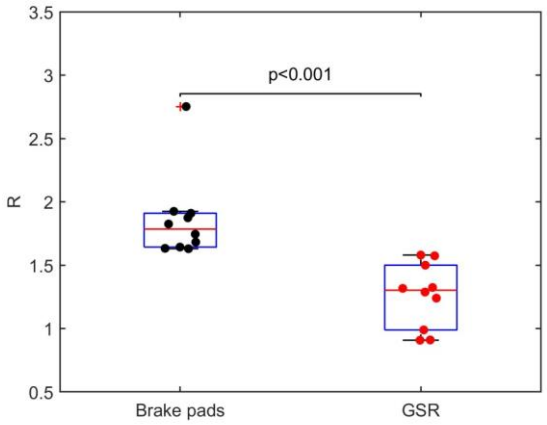

Fig. 5: Box plot and distribution graph of R ratio referred to brake pads (black) and GSR samples (red)
Source: created by the authors

Numerous occupational groups are routinely exposed to particles that share characteristics with gunshot residue (GSR). In particular, particles generated from automotive brake pads are associated with potential false positives for GSR, [9], [29].

The purpose of the present study was to discriminate, by using the Raman Spectroscopy technique, non-GSR sources of particles, which have a composition consistent with GSR, that could

determine a false positive when analyzed by conventional techniques. In particular, SEM+EDX, which represents the gold standard in GSR analysis, could experience difficulties in distinguishing between GSR and particles originating from automotive brake pads, [30]. The starting point was the acquisition and the following analysis of blank stub, GSR, and brake pad dust samples. All the obtained spectra exhibited two main bands attributed to the D-band (~ 1350 cm-1) and the G-band (~ 1585 cm-1) of graphite, deriving from stub coats. Other authors maintain that propellant grains are coated with varying amounts of graphite [31], usually used to prevent static electricity from causing undesired ignition. It can be detected in GSR as graphite dust in the samples.

The spectral subtraction revealed the main differences between GSR and brake pad dust. In fact, we can observe that the center frequencies of the D-band and G-band are shifted when the blank stub contribution is subtracted from GSR and brake pad dust samples. In particular, as evident in Figure 4, the center frequency of the D-band is around 1295 cm-1 for both the GSR and brake pad samples, after the removal of the blank stub contribution. The more notable results of the spectral subtraction are obtained for the center frequency of the G-band. In this case, we can notice that the center frequency of the brake pad dust samples is around 1578 cm-1, then it moved to lower frequencies after the blank stub subtraction; on the other hand, the spectral subtraction revealed that the center frequency of the G-band moved to higher frequencies (around 1610 cm-1) in the case of GSR samples. This experimental evidence could be used to discriminate GSR samples from brake pad dust samples.

To assess quantitative changes due to chemical structural modifications, we calculated the intensity ratio as expressed in (1). The forensic relevance of Equation 1 lies in its capacity to overcome the intrinsic limitations of conventional elemental analysis. While SEM-EDX frequently encounters challenges in distinguishing GSR from brake pad particles due to overlapping elemental profiles of Lead, Antimony, and Barium, the Raman-derived R ratio probes the structural fingerprint of the samples.

We observed that, in all cases, GSR samples have an R value lower than the R value calculated for brake pad samples. The results of the statistical analysis are evident in Figure 5. Encouraged by the obtained results, the next step of our research will concern the possibility of applying the proposed Raman-based methodology for an early categorization of non-GSR sources of particles, which could originate false positives.

4 Conclusion

According to the literature, Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDX) is the most widely recognized method for the characterization of gunshot residue (GSR). While highly effective, this technique has several limitations, including lengthy analysis times and relatively high cost. Moreover, a significant challenge with SEM-EDX lies in the potential for false positives, as particles originating from certain manufacturing sectors, such as welding or metalworking, can exhibit similar characteristics to GSR, complicating the analysis and interpretation of results. These factors underscore the need for complementary techniques or enhanced specificity in GSR detection methodologies, [30]. Methods for the chemical analysis of organic compounds are still in development, [32], [33]. In the present preliminary study, a non-destructive method to distinguish between GSR and GSR-like objects by using Raman Spectroscopy is reported. We have used stubs to collect GSR samples from shooters and the clothes and hands of mechanics after managing brake pads, which contain particles similar to GSR. The detected spectral modifications, particularly the frequency shift and the calculation of the intensity ratio R, could provide a means to differentiate between gunshot residue (GSR) and brake pad dust. This demonstrates that Raman spectroscopy has the potential to reliably distinguish between GSR samples and brake pad dust. The results obtained can be considered a significant starting point for further studies aimed at enhancing the accuracy of this technique in forensic analysis. Future research could focus on a broader range of samples and operating conditions, further strengthening the reliability of Raman spectroscopy as a tool to differentiate between similar particles originating from various industrial and environmental sources.

In this context, we believe that combining the gold standard method for analyzing GSR particles (SEM-EDX) with Raman spectroscopy could offer a promising pathway for future forensic research. The integration of these two techniques, which rely on distinct analytical methodologies, may significantly enhance the ability to achieve this goal. Our research does not propose a replacement for the current gold standard, but rather highlights an alternative technique that can differentiate GSR from non-GSR dust in a non-destructive manner. Furthermore, Raman spectroscopy, when coupled with advanced statistical analysis, holds the potential to be employed as a complementary approach in forensic investigations. It could be

particularly useful as a preliminary screening method for GSR, augmenting the accuracy and efficiency of existing procedures while minimizing the risk of false positives and preserving the integrity of the sample for further analysis.

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- Giuseppe Acri and Marco Romeo Conceptualization; Validation.
- Marco Romeo and Rosanna Abbruzzo carried out investigation and resources.
- Barbara Testagrossa, Alberto Scoglio and Giuseppe Acri: methodology, formal analysis, writing—original draft preparation.
- Valentina Hartwig: data curation, writing—review and editing, statistical analysis
- Matteo Marino and Elisa Ruello: visualization.
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