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Gunshot residue and brakepads: Compositional and morphological considerations for forensic casework

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ABSTRACT

Previous research has raised the possibility that automotive brake pads can produce particles that are both compositionally and morphologically similar to gunshot residue (GSR). These studies, published in the early 2000s, coincided with the reduction or removal of unnecessary sources of lead from the automotive industry. The question therefore arises whether modern brake pads might still be a relevant source of 'GSR-like' particles.

In the present study, a total of 75 brake pads taken from a range of cars currently on the road in Adelaide, South Australia, were collected from service centres. These pads were screened by XRF, and 12 were selected as representatives of the types of pads available on the market. Some pads generated XRF results for elements particularly relevant to GSR analysis. Signals for barium and antimony were commonly observed, with some pads showing results possibly attributable to lead. The surfaces of these 12 pads were directly sampled using aluminium stubs equipped with carbon-filled adhesive tape. Following this, they were screened using SEM-EDS and an automated GSR particle search in order to detect particles containing lead, barium or antimony. No particles containing all of these elements were found, although a large number of particles containing both barium and antimony were located.

Other particulate samples were collected using stubs from persons or objects associated with brakes in order to examine whether particles similar to GSR might be present on them. No three-component particles were detected in samples collected from the wheel rims or the hands of those exposed to automobiles.

From this study of common, contemporary cars, brake pads and brake pad technicians, it was determined that the possibility of obtaining three-component 'GSR-like' particles from brake pads appears to be much lower now than when this issue was first raised in the early 2000s. While some brake pads do produce particles containing barium and antimony, they are often angular particles that contain sulphur. Furthermore, these 'GSR-like' particles are commonly found together with an abundance of particles containing iron. The particle evidence evaluated in total allows clear differentiation between residues originating from brakes and residues originating from firearms.

1. Introduction

Gunshot residue is the term used to refer to the substances that form as a result of the discharge of a firearm and remain to be collected during the investigation of a shooting incident. These residues originate from the primer and propellant and may incorporate traces of the projectile, cartridge casing, or barrel of the firearm [1,2]. The American Society of Testing and Materials (ASTM) has established guidelines for the analysis of GSR and defines a hierarchy of particles. Particles considered characteristic of GSR (most likely to be associated with the discharge of an explosive cartridge or firearm), are spheroidal, non-crystalline, and contain the elements lead (Pb), barium (Ba) and antimony (Sb) [3]. Other particle types, considered consistent with a firearm source, are also defined in this standard, but are considered less strongly supportive of a firearms origin. Further, several 'non-toxic' or 'green' ammunition formulations exist that do not include heavy metal elements as a component of the primer. These ammunition formulations produce their own characteristic and consistent GSR particles, which are separately defined within this standard.

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Regardless of whether the heavy metal free compositions or traditional Pb/Ba/Sb primers are involved, GSR may be used to provide an association between an individual and a firearms incident. As GSR is distributed from the firearm during firing, particles of these compositions naturally settle on the shooter, any bystanders, and in the surrounding environment [4]. With this in mind, particulate samples are often collected from the hands of suspects in shooting cases, in order to ascertain if they have fired, handled, or been in close proximity to a firearm.

Environmental and occupational sources of particles that could be confused with GSR in casework situations are, therefore, important to the interpretation of GSR evidence. Trace evidence can be evaluated using a logically correct framework that produces a likelihood ratio. At the lowest level, called the sub-source level, the likelihood ratio provides a consideration of the competing probabilities that the result observed would be seen if the residues originated from a firearm or cartridge source, or if they originated from a non-firearm source. Accordingly, the magnitude of the likelihood ratio is inversely proportional to the prevalence of GSR particles, and particles that cannot be distinguished from GSR, within a particular population (e.g. present on a ‘random man’).

In the early-2000s, several studies [5–7] found that the friction surfaces of brake pads could generate particles that contain combinations of lead, barium and antimony. Brake pads, and the vehicles to which they are fitted, are abundant in the wider population. The studies were therefore significant because a large cross-section of the population could be exposed to particles that might resemble GSR on a frequent basis as a result of activities such as changing wheels, inflating tyres, and even washing or cleaning their car. If even a small proportion of the particles produced by braking resemble GSR, then the presence of these ‘GSR-like’ particles on a random member of the population and certain other populations could be relatively high. However, the studies showed that these particles were associated with an abundance of particles that are not usually associated with firearm discharge. Additionally, these ‘GSR-like’ particles lacked the characteristic spheroidal morphology that is typical of GSR, and tended to be irregular. As a consequence, while individual particles from brake pads considered in isolation may resemble individual particles from firearm sources, it was concluded that it was possible to determine that a population of particles arose from a brake source rather than a firearm source. Despite the clarity of the findings in the early studies, the fact that brake pads can produce some particles that resemble GSR was on occasion, cited as evidence that GSR examination was unreliable, lacked specificity, or was prone to false positives. This view is exemplified by an article in a popular science magazine [8] and alluded to even in more scholarly articles [9].

Friction materials used in brake pads often contain barium sulfate, which is used to control the heat stability of the material [10]. Further, a number of metal sulfides are used as additives in brake pads in order to modify the frictional characteristics of brake materials, in particular antimony trisulfide is used as a solid lubricant [11]. In 1996, the United Nations [12] released a declaration on the risks of lead exposure, and a recommendation that the potential for lead exposure be minimised. One recommendation targeted the reduction of automotive sources of lead through the removal of the element from fuel, brake pads, and other automotive components. Many countries acknowledged the risks of lead exposure and over the next five years, new legislation was passed, or existing legislation tightened, in a variety of countries including the USA, Australia, the UK and a number of EU countries [13–16]. Currently existing automotive sources of lead include lead-acid batteries, automotive paints and wheel weights.

The transition period when lead was being phased out of the automotive industry broadly corresponded to the period when the research into the automotive origins of GSR-like particles was being carried out. Furthermore, modern surveys do not seem to reveal a widespread occurrence of GSR-like particles on “random man” [17,18].

Consequently, an investigation of the types of particles produced by modern brake pads in modern vehicles in light of the latest ASTM definitions of GSR was warranted. The work presented here is an evaluation of particles generated from a number of brake pads and an assessment of their significance to GSR examinations.

2. Methods

A total of 75 brake pads were collected. All pads were collected from mechanics’ workshops after removal from vehicles. Although in some cases, the vehicles to which these pads may be fitted could be identified based on part numbers, the actual vehicle from which the pad samples were taken was not known. Most of the brake pads were at the end of their useful life, where others had moderate wear. Consequently, these pads had been exposed to the typical heat and abrasion conditions of the braking process over an extended period, and therefore represented the best-case scenario for the production of ‘GSR-like’ particles to which a random member of the population may be exposed.

2.1. Elemental composition of brake pads—XRF analysis

A Tracer III–V+ portable handheld XRF (Bruker, USA) (Rhodium X-ray tube and Peltier cooled Si-PIN detector) was used to generate XRF spectra for the brake pads analysis. The S1PXRF Bruker AXS Handheld Inc. Software (version 3.8.30) was used. All spectra were taken using X-ray energy of 40 KeV, a current of 3 μA and using the yellow filter (0.001 in/0.0025 cm Ti and 0.012 in/0.0305 cm Al), and counting for 30 s. This setup enabled the potential measurement of elements from Ti to Ag (K lines) and W to Bi (L lines). Six measurements were taken from different points across the surface of each brake pad in order to ensure results were representative of the entire brake pad. A comparison between an unworn pad and worn pad, with sample sites marked can be seen in Fig. 1. These data were analysed using Bruker AXS MA ARTAX version 7.4.0.0 in order to assign peaks and determine peak intensities. Software peak assignments were verified. Samples for SEM analysis were chosen to be representative of a wide variety of brake pad compositions (Table 1), by choosing pads with various combinations of forensically relevant elements. Furthermore, it was decided to include a mix of OEM (Original Equipment Manufacturer) and aftermarket pads from a variety of different manufacturers to account for possible differences in composition

2.2. Particle search of brake pad dust—SEM-EDS

All pads selected from the XRF survey were sampled by dabbing across the entire surface of the pad with GSR SEM stubs equipped with a circle of double-sided adhesive carbon tape (Tri-Tech Forensics Inc. North Carolina, USA). An Inspect F50 Scanning Electron Microscope (FEI Inc., Oregon, USA) equipped with an EDS detector (EDAX Inc., New Jersey, USA), GSR Magnum particle analysis system (FEI Inc., Oregon, USA), and TEAM software (EDAX Inc., New Jersey, USA) was used to detect and analyse particles collected on the stubs. An accelerating voltage of 25 KeV with a 30 μm^2 aperture and an average current of ~100 μA were used for elemental analysis.

The GSR Magnum software brightness and contrast settings were calibrated using a gold, niobium, germanium, silicon and carbon (Au/Nb/Ge/Si/C) standard. Particle identification settings were arranged to analyse particles where the average $Z \geq 30$, in

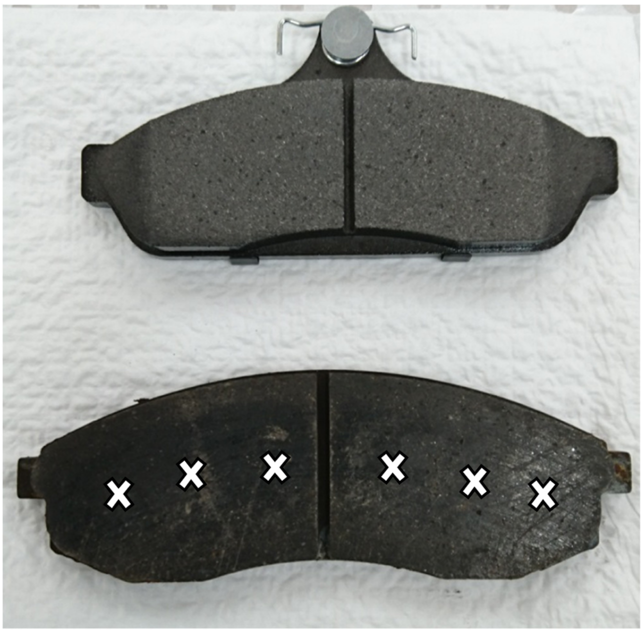

Fig. 1. Exemplar brake pad as used for sampling. Comparison between unworn (top) and worn (bottom) pads. The six points across the surface of the pad analysed by XRF are designated with an 'x'.

order to filter out some iron (Fe) signals and to reduce analysis time.

A synthetic particle standard (PLANOW Plannet GmbH, Wetzlar, Germany, SPS-5P-2A), consisting of deliberately deposited Pb/Sb/Ba films of known size between 0.5 um and 10 um, was analysed at the start and end of every sample run as a positive control.

2.3. Collection and examination of particles from brake rotors, wheel rims, and hands

Particulate samples were collected from brake rotors (n = 3), wheel rims (n = 22), and hands of individuals (n = 11) known to have recently handled brake pads or wheels, or regularly come into contact with brake pads through their work. All samples were collected using adhesive aluminium sample stubs.

Samples from brake rotors were collected by directly sampling from the rotor surface. Samples were collected by applying the adhesive stub to the rotor surface at a point directly accessible from between the wheel spokes.

Wheel samples were taken directly from vehicles in a car park. They were collected by dabbing wheel rims with a stub. The cars sampled were from various manufacturers and models, but the brake pad types present on the vehicles were unknown.

The hands of a number of mechanics who had handled brakes and other automotive components were also sampled.

All samples collected were examined using SEM-EDS and GSR Magnum particle search software under the same settings and conditions used for the brake pad particle analysis; particle composition assignments were verified manually.

All particles found on pads, components, and hands were evaluated in the context of the ASTM guidelines [3].

A summary of the total samples collected and analysed as a part of this work can be seen in Table 2.

3. Results and discussion

3.1. Elemental composition of brake pads—XRF analysis

The XRF analysis of 75 brake pads obtained from local automotive mechanics identified 12 samples that represented the population, with some samples indicating the possible

Table 1
Brake pad samples chosen for SEM analysis, manufacturer and vehicles compatible with these pads (if known).

Sample number	Manufacturer	Part no.	Vehicles	Relevant composition by XRF
C1	Bendix	DB1474	Toyota Camry (2002–2006) Toyota Avalon (2003–2006)	Moderate Ba, Cu, Mo, Pb, Sb
C2	Bendix	DB1455	Mitsubishi Colt (2006–2013) Mitsubishi Lancer (2003–Present)	As, Ba, Nb, Pb, Sb
C8	Bendix	DB1808	Holden Astra (2005–2010) Holden Zafira (2001–2006) Holden Combo (2002–2012)	No Ba
C12	Toyota (OEM[a])	PC554	Toyota Celica (2002–2005)	High Mo, Low Sn
C18	PBR	B746-1169	Unknown	Ba, Mo, Pb, Sb,
C21	Bendix	DB1473	Ford Falcon (2008–2014)	Lowest Fe, No Zr.
C24	Mazda (OEM)	D15748784	Mazda Ford	Ba, Pb, Sb,
C38	Mazda (OEM)	D15748784	Mazda Ford	Ba, (High K), Pb, Sb, Zr,
C42	TMD Friction	PA645	Volkswagen, Audi,	Bi, Cr, low Sb, Ba, Pb
C46	Jurid	D1375	Audi A6 (1994–1997) Audi A8 (1994 –2002)	Co, Rb
C57	ATE	ATE7895	Mercedes, BMW,	As or Pb
C60	Premier	DB1085A	Holden Commodore (1978–2001) Toyota Lexcen (1989–1997)	High tin, low Mo

[a] Original Equipment Manufacturer.

Table 2
Summary of samples analysed.

Sample	Number of samples
XRF	
Brake pads	75
SEM-EDS	
Brake pads	12
Brake rotors	3
Wheel rims	22
Hands	11

presence of lead, mostly in low concentration around the limit of detection. The majority of pads were found to contain higher concentrations of barium and antimony. These 12 representative pads were further analysed by SEM-EDS.

3.2. Particle search of brake pad dust—SEM-EDS

These 12 brake pads were stubbed to collect loose particles off their surface and the stubs were reviewed manually to see if there were particles that were similar to GSR. Particles exhibiting morphological characteristics of GSR were not observed; the particles were angular, flaky and not spheroidal or regular (Fig. 2).

The spheroidal morphology of GSR particles is attributable to the high temperatures and pressures that they are exposed to during the discharge of a firearm [19]. While automotive braking can generate temperatures in excess of 500 °C under extreme conditions, usual temperatures are in the range of 40–250 °C, at atmospheric pressure [20]. The morphology of the particles found, and the distinct lack of spheroidal or globular particles, suggests that temperatures and pressures attained during typical braking are not sufficient to replicate the formation of the characteristic morphology of particles seen in GSR.

Table 3 is a list of relevant particles detected using SEM-EDS. The automated particle screening software detected one 'characteristic' Pb/Ba/Sb particle. This was a false-positive result; rather than being a single particle containing all three components, it was a lead particle that was resting on a larger particle containing both barium and antimony. Overall, the occurrence of single element lead particles was relatively low, with 0.12% of all particles detected being of this type. The combined frequency of all other particle types containing lead (Pb/Ba, Pb/Sb, Pb/Sn, Pb/Ti, Pb/Ca, Pb/Cl/Br and Pb/Sb/Sn) was 0.35% overall. In total, all particles in this survey containing lead amounted to 0.47% of all detected particles. The particles identified as consistent with GSR by the software were all detected in low to very low frequencies (<2% of detected particles), with the exception of Ba/Sb particles, which were more frequently observed, making up 10% of all detected particles from direct sampling of brake pads. These particles were produced by six of the twelve pads examined. Although the software identified these particles as being consistent with GSR, manual review of a sample of the elemental data and images collected was able to eliminate all as being of firearm origin either on the basis of morphology or the presence of forbidden elements. It was found that the vast majority of particles originating from automotive sources had a compositional heterogeneity that is atypical of GSR. That is, these particles had a tendency to exhibit discrete zones, each containing a large number of elements within the one particle, whereas GSR tends to possess a distribution of a small number of elements. A comparison of the form and composition of brake pad particles and GSR is shown in Figs. 3 and 4. This is consistent with particles from brake pads arising through the physical process of abrasion rather than through a random condensation of elements from a gaseous mixture, as is thought to take place when GSR are produced [19].

Fig. 3 shows the typical spectrum of brake pad particle observed in this study, showing a particle classified as "consistent—Ba/Sb", but it contains iron, titanium, potassium, sulphur, silicon, copper, aluminium and magnesium at similar or greater peak levels than barium and antimony. These data are comparable to the results reported by Torre et al. [5] and Garafano et al. [6], who also frequently observed particles containing these elements. By comparison, the GSR particle in Fig. 4 exhibits far fewer of these additional elements and at lower levels.

As might be expected for modern vehicles produced after the initiative to remove lead from products, a total of only 579 particles containing lead were found in brake pad dust from the 12 pads examined. The overwhelming majority of these particles (566) contained lead and barium and all particles that were manually examined could be eliminated as originating from a firearm. Likewise, single-element Pb, Ba or Sb particles reviewed were obviously not from a firearm source.

Iron was a ubiquitous component on all sample stubs, with 50–70% of the particles detected containing iron as a major component. This result is comparable to particles observed in the work by Torre et al. [5], who found iron to be present in all brake pad particles, frequently at a major level. However, the result observed in the present study is a gross under-representation of the true iron content of the samples, as the search was carried out using settings that were designed to exclude iron particles and many particles containing iron as a major element were classified as other particle types. Under the current ASTM guideline, while a trace level of iron in a particle is acceptable, a major contribution of iron would result in the particle being classified as not likely to originate from a firearms source.

These results indicate that while samples collected from brake pads may result in a few false positive results through the software, a trained analyst would resolve the situation readily. This is in support of the conclusions previously drawn by Garofano et al. [6].

3.3. Particles from brake rotors, wheel rims, and hands

Random members of the public may encounter particles from brakes as a result of activities such as changing a wheel on their car,

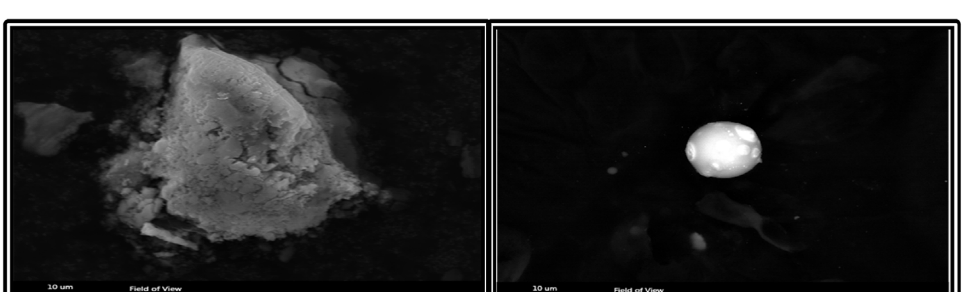

Fig. 2. Morphological comparison of a typical brake pad particle (left) from direct sampling and a typical GSR particle (right).

Table 3
Frequency (%) and absolute number (n) of selected particle types automatically categorized by software for particles with average 30 < Z < 82 by BSE-SEM-EDS.

	Pads (n = 12)		Hands (n = 11)		Wheels (n = 22)	
	%	n	%	n	%	n
Characteristic of GSR						
PbBaSb	0	0(1)[a]	0	0	0	0
Total characteristic	0	0	0	0	0	0
Consistent with GSR						
PbBa	0.634	566	0.00694	8	0.0155	23
BaSb	10.5	9374	0.182	210	2.62	3879
PbSb	0.00224	2	0.0165	19	0	0
BaCaSi	1.09	977	1.332	1536	1.59	2358
BaAl	0.038	34	0.036	41	0.038	56
Total consistent	12.3	10953	1.6	1814	4.3	6316
Single element						
Sb	0.232	207	0.0416	48	0.0128	19
Ba	2.84	2530	0.923	1064	7.23	10722
Pb	0.0112	10	0.305	352	0.0412	61
Particles indicative of other sources						
BaS	10.5	9386	3.81	4399	6.53	9679
SbS	0.629	561	0.317	366	0.0189	28
CuZn	0.738	659	0.268	309	0.0331	49
Fe	50.9	45,458	59.0	68,064	65.8	97,594
Cu	2.72	2429	1.27	1466	0.173	257
Ti	1.19	1060	0.589	679	0.198	293
Sn	1.13	1010	0.202	233	0.0344	51
Sr	0.00224	2	0.00087	1	0.00135	2
Subtotal[b]	83.22	74,263	68.33	78,795	84.4	125,071
Total	100	89,240	100	115,314	100	148,211

Remaining categories (not shown) include commonly encountered environmental particles such as lighter flint, coinage or jewellery, or particles that were unclassified by the software.

[a] One sample registered a false positive, detecting a singular 3-component particle. On review, it was discovered to be a lead particle sitting on a flake that contained antimony and barium, amongst other elements.

[b] Total indicates total percentage of detected particles described by the above categories.

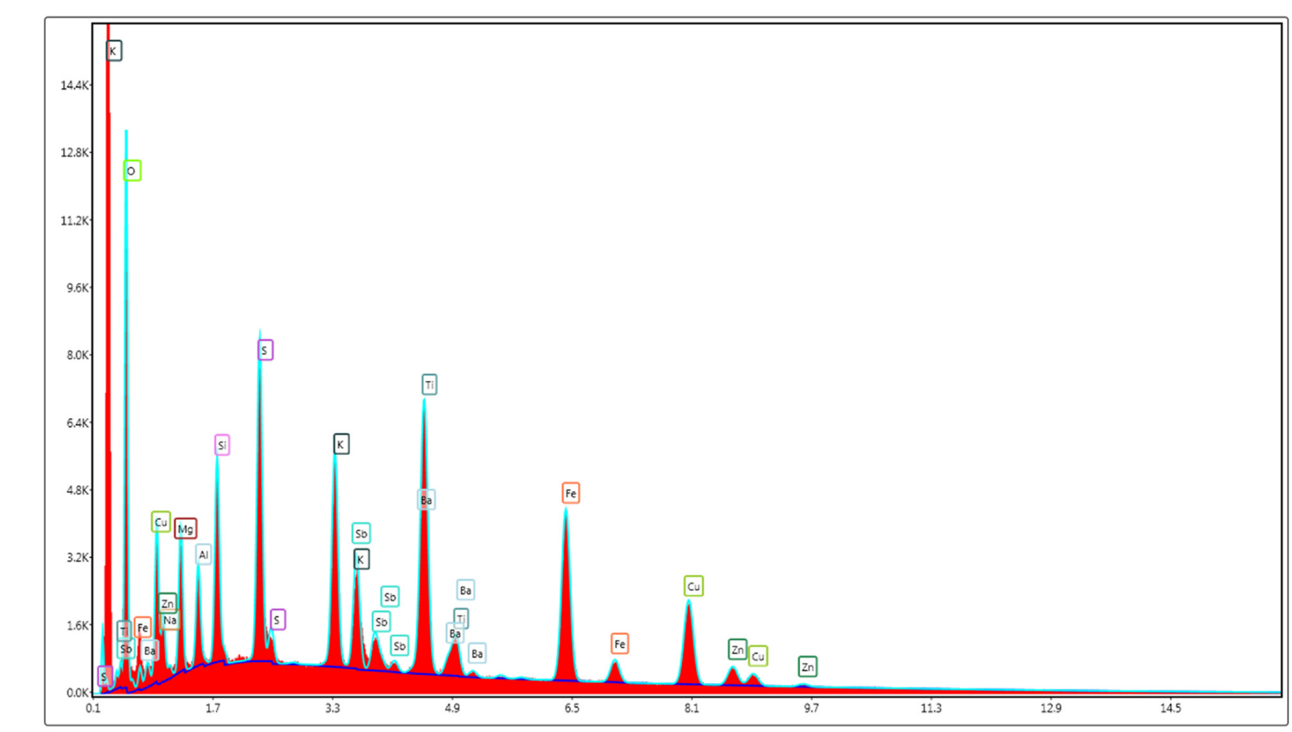

Fig. 3. Typical EDS spectrum of a brake pad particle.

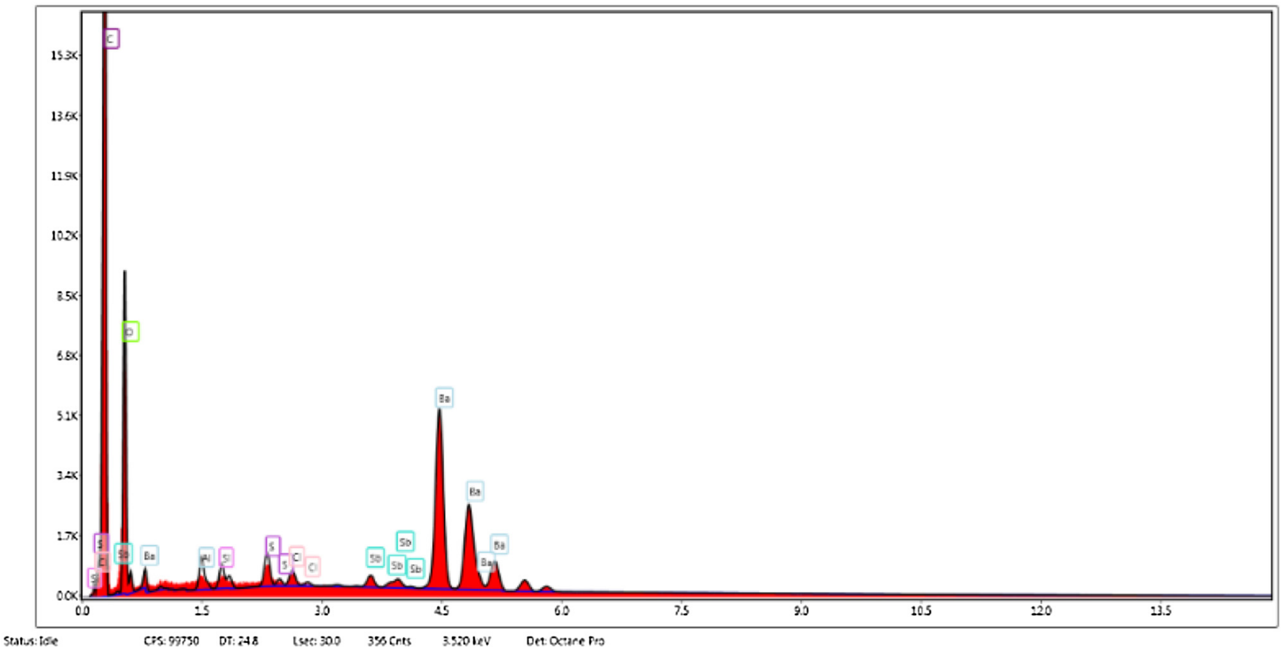

Fig. 4. Typical EDS spectrum of a GSR particle.

touching a wheel rim during inflation of tyres, or while cleaning their car. Figs. 5 and 6 show the distributions of particles found on brake pads and wheel surfaces, respectively, as classified by the particle screening software. Additionally, the hands of mechanics and volunteers who had handled wheels were sampled (see Fig. 7 for the distribution of particles). This cohort was chosen because these individuals handle a broad range of automotive components that might expose them to GSR-like products, specifically those containing lead.

Particles were collected from three brake rotors and they showed few compositional or morphological differences from those collected directly from brake pads, as displayed in Fig. 2.

When the particles depicted in Figs. 6 and 7 were reviewed, other elements were detected in abundance, especially sulphur and iron, which allowed the particles to be ruled out as being derived from firearm discharge. In addition, the particles observed in this case did not appear to be spheroidal, which is a requirement for particles to be consistent with or characteristic of GSR. An

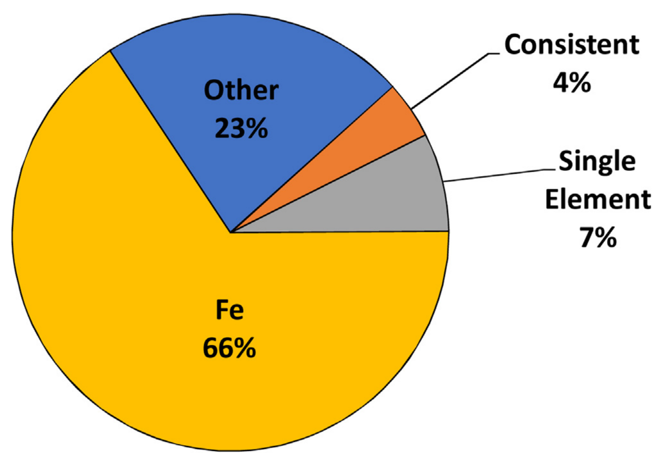

Fig. 6. Distribution of particle classifications for samples collected directly from wheels (n = 22).

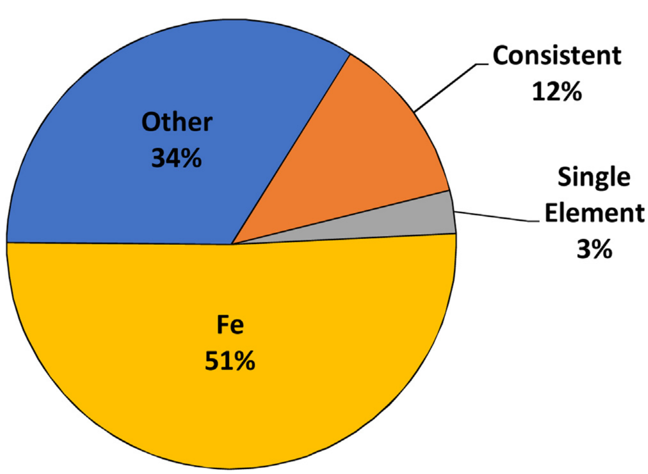

Fig. 5. Distribution of particle classifications for samples collected from brake pads (n = 12).

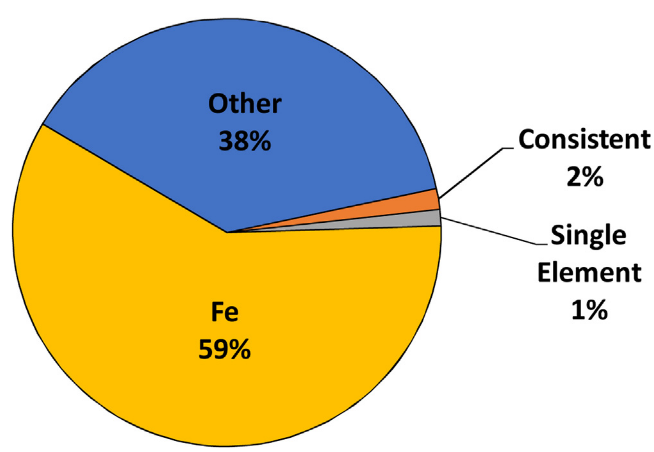

Fig. 7. Distribution of particle classifications for samples collected directly from hands (n = 11).

experienced GSR examiner would therefore exclude these as possible GSR particles.

4. Conclusions

Seventy five used brake pads collected at random were analysed using XRF. Twelve were selected as representatives of the population and dust from the surfaces of them was collected and analysed using SEM-EDS. Wheels and disc rotors were sampled for particles as were the hands of people who had recently handled brake pads and wheels in order to determine if “random man” or “random mechanic” might become contaminated with GSR-like particles.

Torre et al. in their study in 2002 [16] detected lead in many particles collected from automotive parts and workers who handle them. For example, in their examination of dust from the wheel rims of 40 automobiles 16 provided particles containing lead. In the present study, very few particles containing lead were found on either components (brake pads and wheel rims) or the hands of individuals who had touched them. It would appear that initiatives introduced after the work of Torre et al. that were aimed at minimizing the usage of lead in automotive components has had an impact on the composition of dust derived from brake pads.

No particles containing lead, barium, and antimony simultaneously were found during this survey. However, a relatively large number of particles containing barium and antimony, or just one of those elements, were observed through this study. Particles observed from all sources had both morphologies and compositions that were inconsistent with GSR; the particles were composed of a large number of elements and many “non-GSR” elements were more abundant than is considered consistent with GSR. Particles from brake pads appeared to be conglomerates of smaller particles, particles were not rounded, and the compositional variation between particles from the same pads and different pads was low.

Even though lead-containing particles now appear to be a rare component of brake dust, the present study re-emphasises the importance of both a thorough review of EDS data reports produced by automated particle classification software and the evaluation of the overall population of particles recovered in connection with an investigation. Once these measures have been implemented, it is difficult to envisage scenarios in which brake dust constitutes a GSR false positive. Any scenario would require selective retention of a small minority of particles from a population (i.e. only those that contain barium and/or antimony) and the selective loss of particles containing iron and sulphur. On the other hand, scenarios that might lead to a false negative result are more plausible, for example when a surface is sampled that contains a mixed deposit of brake pad dust and a few particles of GSR.

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