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Research Article

Comparing sampling procedures for trace residues from bullet hole circumferences to determine the striking of projectiles

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Abstract
The recovery of ballistic trace residues from bullet holes is crucial for forensic investigations to link projectiles, firearms, and crime scenes. Nonetheless, there remains a lack of consensus on the most effective sampling procedures to retrieve elemental information from the inner circumference of a bullet hole found on materials of varying properties. This study compares three sampling procedures, namely swabbing, tape lifting, and scraping, for their efficiency in recovering lead (Pb), copper (Cu), and zinc (Zn) residues from bullet holes under controlled conditions. Using a semi-automatic pistol with 9 mm ammunition, 21 shots were fired on each material, including Perspex, plywood, Formica board, gypsum board, and two metal sheets of different thickness. Seven bullet holes per material were sampled by each sampling procedure, followed by acid digestion and elemental analysis *via* graphite furnace-atomic absorption spectroscopy. The two-way ANOVA statistical test revealed significant differences in the recovered concentration of Pb, Cu, and Zn from bullet holes in relation to the sampling procedures ($p<0.001$) and surface materials ($p<0.001$). Tape-lifting procedure consistently recovered higher concentrations of Pb and Cu by 45.7% and 13.7%, respectively. For Zn, both tape-lifting and swabbing procedures allowed for better recovery up to 21.1% compared to scraping procedure. In term of target surfaces with bullet holes, higher elemental contents could be recovered from malleable materials, regardless of the sampling procedures. As a less destructive, requiring no solvent and more practical sampling procedure, this study proposes tape-lifting as the preferred procedure to collect ballistic trace evidence from bullet holes for shooting event reconstruction.

Keywords: bullet hole, sampling, tape-lifting, swabbing, scraping, GF-AAS

Introduction

During the routine examination of a shooting-related incident, the determination of any hole on a surface made by a gunshot frequently relies on the observation of its morphological features, depending on the nature of the impacted surface [1,2]. In addition to morphological changes, the striking of a projectile onto any surface also tends to deposit the trace materials, composing gunshot residue (GSR), metallic fragments, or ballistic-related particles, onto the bullet holes. These trace residues contribute to forensic investigation, particularly for the reconstruction of shooting events to link the firearm, the projectile, and the scene [3]. In most instances, they are found surrounding a hole or an impact due to transference of materials during a shooting event and play an important role in confirming it to be caused by a projectile, frequently through the detection by scanning electron microscope-energy dispersive x-ray (SEM-EDX) detector [3-5].

Previously, articles and technical aspects related to the collection and detection of GSR particles from a suspect's hand have provided significant contributions in relation to their sampling efficiency [6,7], time elapsed since a shooting event [8-9], and the effect of hand washing [10]. Apart from that, GSR particles could also be detected from various surfaces and materials, frequently on people, objects, and the surrounding environments after the discharge of a firearm, highlighting the importance of sampling procedures in recovering these trace residues.

Research by Zeichner and Levin [11] explored the effectiveness of adhesive lifting methods for recovering GSR particles, revealing significant differences in the recovery rates due to the adhesive type and surface texture. Commercially available adhesive lifters were used to collect potential GSR particles from the walls and clothing [12], while two-sided adhesive carbon tapes affixed on aluminium stubs were utilised for the sampling of secondary transfer GSR particles [10]. Instead of using a stub where the carbon tape was attached, Rodriguez-Pascual et al. [13] also used adhesive film to sample the GSR adhered to various surfaces, including stainless steel, particleboard, and polyvinyl chloride foam board, followed by laser-induced breakdown spectroscopy (LIBS) to investigate the elemental spatial distribution. LIBS also allowed for the detection of GSR around bullet holes from the adhesives applied on the plywood, float glass, and drywalls for shooting distance determination [14].

Generally, the efficiency of sampling methods was found to vary greatly across diverse surfaces, especially porous and non-porous surfaces. More importantly, these studies were mainly targeted at the recovery of GSR particles and not the trace residues at the inner circumferences of bullet holes. With a distant shooting activity, these GSR particles might not be able to reach the target surface, restricting the possibility of confirming the striking of the projectile [5]. There remains a lack of consensus on the most effective sampling procedures for various surfaces commonly encountered at crime scenes, particularly from the inner circumference of a bullet hole. Their efficiency in recovering trace residues from bullet holes found on diverse materials of varying properties also remain inadequately established. This study provides empirical evidence comparing three sampling methods, namely swabbing, tape lifting and scraping, under controlled conditions. By evaluating each sampling procedure's performance across surface types, we seek to propose the best protocol in maximising the value of ballistic trace evidence, contributing to the improvement on the reliability of forensic analyses.

Materials and Methods

Chemicals and reagents

Individual standards for lead (Pb), copper (Cu), and zinc (Zn), each with a concentration of 1000 mg/mL, were purchased from Agilent Technologies (Santa Clara, CA, USA). Ultrapure reagent-grade nitric acid (HNO_3, 65%, w/w), hydrogen peroxide (H_2O_2, 30%, v/v), and analytical-grade methanol were obtained from Thermo Fisher Scientific (Hampton, NH, USA). Ultrapure water (18.2 MΩ) was prepared in-house from a Millipore water purification system (Bedford, NY, USA).

Shooting

A semi-automatic pistol, Sig Sauer model SP 2022 (Schweizerische Industrie Gesellschaft and J. P. Sauer and Sohn, Switzerland), was used for the shooting of SME 9 mm ammunition (Full Metal Jacket, Round Nose, Selangor, Malaysia). A series of shots was performed on six different surface materials, commonly encountered in household construction in local settings, as follows:

i. Perspex
ii. Plywood
iii. Formica board
iv. Gypsum board
v. Thin metal sheet
vi. Thick metal sheet

Each surface materials were of consistent thickness and cut into 30 cm × 30 cm dimension. Prior to shooting, the surface material was placed on top of a bench in a vertical position and securely held in place. The outer side of each surface material was positioned in front of the shooter. Shot locations were marked on a grid, with each impact spaced at least 10 cm apart to prevent overlapping of bullet hole and potentially contamination by residues originated from the different shots. The order and position of shots were also randomised to avoid positional bias. At a three-meter distance from the target, a total of 21 bullet holes were produced per target material by a trained shooter at 90º level. Each target material was then removed from the frame, wrapped properly, and transported to the laboratory for further examination

Sampling of trace residues from bullet holes

Sampling procedure applied in this study was to collect the trace residues adhered to the internal circumference of bullet holes and not at the peripheral area surrounding the bullet hole. The total 21 bullet holes were allocated to the three sampling procedures, seven holes per sampling method. They were sampled separately and assessed for their reproducibility. Each residue sample was also analysed separately, with triplicate analytical measurements performed per sample. Note that both swabbing and tape-lifting procedures were adapted from Reid et al. [6] which had demonstrated their usefulness in recovering GSR from shooter's hand. Scraping procedure was proposed in this study for a comparative study as materials from a projectile could be strongly adhered to the bullet hole, requiring physical removal of such trace elements.

Swabbing

The swabbing procedure was applied by swabbing the internal circumference of a bullet hole using a cotton swab stick moistened with analytical grade methanol. Methanol was used due to its choice of solvent to recovery inorganic GSR from shooter's hand and adapted in this study [6]. The moistened swab circled the bullet hole clockwise three times, followed by anticlockwise three times. The sampled swab stick was then cut and transferred into a polypropylene tube, sealed, and labelled.

Tape-lifting

Double-sided adhesive carbon tape was first wrapped around a wooden stick. Next, it circled around a bullet hole clockwise three times, followed by an anticlockwise direction three times. After the sampling, the section with adhesive tape was cut from the wooden stick and transferred into a polypropylene tube, sealed, and labelled.

Scaping

Using a plastic cutter, the internal circumference of a bullet hole was carefully scraped onto a piece of white A4 paper. The scraping procedure was performed by circling the bullet hole clockwise and anticlockwise, respectively, three times each. Then, the particles scraped off from the bullet hole were transferred into a polypropylene tube, sealed, and labelled.

Sample processing and preparation

The tested samples (swabbed, tape-lifted, and scraped samples) recovered from bullet holes were acid-digested. Due to the possible presence of unburnt propellant powders on the bullet holes, an open acid-digestion procedure was performed in all instances to avoid any accidental explosion. Each sample was gently heated with the presence of 6 mL of HNO_3 and 2 mL of H_2O_2 on a stirring hotplate at 100°C and 500 rpm for one hour. Upon digestion, each was filtered through a nylon syringe filter into a 10 mL volumetric flask and topped up with ultrapure water to the mark. Each surface material tested in this study also served as the negative control. Using a razor blade, a weight of 2.00 g of each material was cut from the larger piece and acid-digested following the above procedure.

Elemental profile determination

An Analyst 800 graphite furnace-atomic absorption spectroscopy (GF-AAS) (PerkinElmer, Waltham, MA, USA) was used to detect three elements (Pb, Cu, Zn) of each bullet hole from the six target surfaces. GF-AAS was chosen for elemental quantification due to its adequate detection limits for trace Pb, Cu and Zn, requires minimal sample volumes, and allows for background correction to minimise matrix effects. This technique offers both sensitivity and ease of use, making it suitable for forensic applications involving limited sample quantities. The furnace temperature programmes covered sequential drying, pyrolysis, atomisation, and cleaning steps. Wavelengths of the detector were set at 283.3 nm, 324.8 nm and 213.9 nm for Pb, Cu and Zn, respectively. Zeeman background correction was applied during the analysis.

Pb, Cu and Zn were analysed as they represent the major metallic constituents of jacketed bullets, particularly lead from the bullet core, and copper and zinc from the brass or gilding-metal jacket. These elements are typically transferred to the target surface upon impact, forming measurable residues suitable for elemental analysis [15-17]. Calibration standards for Cu and Zn were prepared in a range between 0.05 and 0.25 µg/mL, respectively, while a calibration ranges from 0.50 and 2.50 µg/mL was used for Pb. Calibration standards were run to develop the calibration curves for each element. Tested samples were aspirated into the GF-AAS system in triplicate to evaluate the repeatability of the analytical procedure and minimise random measurement errors. The presence of target element in each sample was evaluated and compared based on the concentration recovered from the bullet holes. All data were also exported into Microsoft Excel® for data treatment (Redmond, WA, USA).

Statistical analysis

Elemental profiles by the sampling procedures and surface materials were compared through Two-way Analysis of Variance (ANOVA) statistical test using IBM® SPSS Statistics version 27 (SPSS Inc., Chicago, IL, USA). A p-value of <0.05 was considered statistically significant.

Results and Discussion

Elemental profiles of negative controls

Among the negative controls, no surface materials were detected with Cu and Pb; however, Zn was found to be present in acid solutions containing Formica board and metal sheets. The presence of Zn in Formica board could originate from the materials that are impregnated with resin and pressed together at high pressure and temperature [18]. On the other hand, zinc-iron alloy could be the contributor of such element in the metal sheet, which involved the coating through electroplating [19]. In this study, elemental profiles of the tested samples recovered from the respective bullet holes shall be interpreted carefully, particularly with Zn.

Development of calibration curves

Calibration curves were constructed for each element to establish the relationship between absorbance and concentration. They show good linearity with correlation coefficients (R^2) greater than 0.995 (**Figure 1**), suggesting the reliability of quantitative measurements. The quality assurance procedures covered the analysis of procedural blanks and multi-element calibration standards before and after each sample batch. Recoveries were verified using reference standards with relative standard deviation (%RSD) values below 10%, indicating good analytical precision.

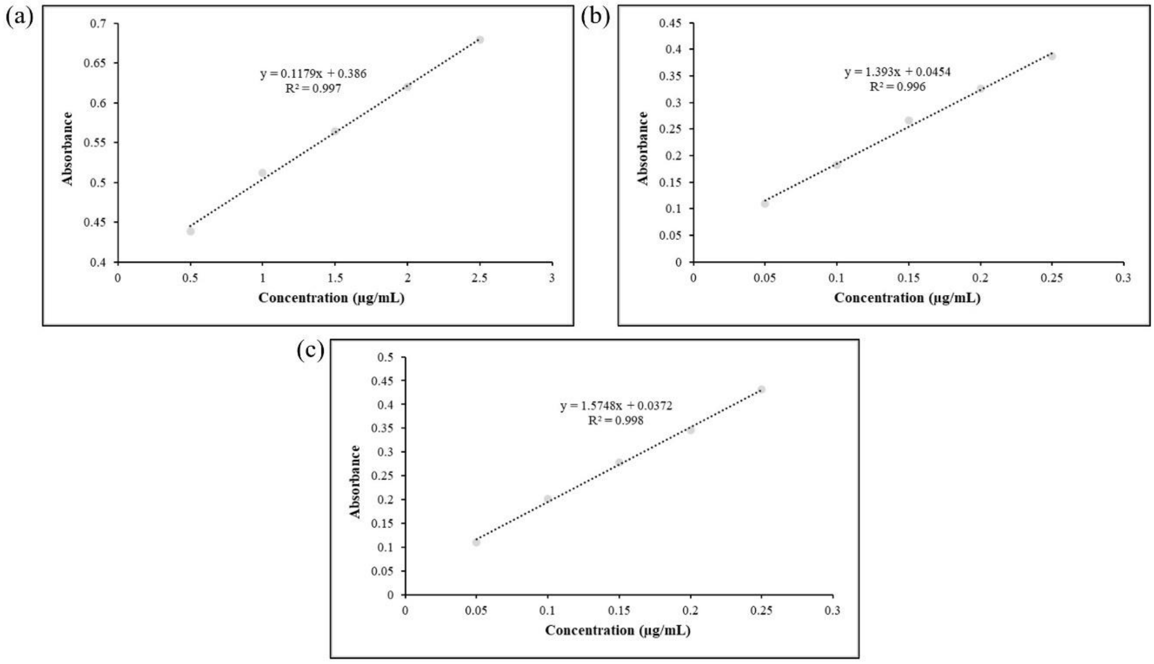

Figure 1. Calibration curves of absorbance values against the concentration of (a) Pb, (b) Cu and (c) Zn

Comparison of the concentration of lead recovered from bullet holes by sampling procedures

Two-way ANOVA was carried out to determine the effect of two independent factors, namely the sampling procedures and the surfaces with impact by projectiles, on the concentrations of Pb. These factors consisted of more than two levels, and the assumptions of the statistical tests were checked. The Shapiro-Wilk test with a *p*-value greater than 0.05 demonstrated the normality of the dataset, and **Table 1** shows the statistical output comparing the sampling procedures based on the concentrations of Pb.

From **Table 1**, the simple main effect analysis showed that the tape-lifting procedure had significantly recovered a greater amount of Pb residues by 45.7% as compared to another two sampling procedures. In terms of surface materials, the trace residues recovered for the different surface materials were found varied. The Perspex and metal sheet surfaces subjected to the impact of projectiles were detected with a higher concentration of Pb as compared to plywood, Formica board, and gypsum board. The amount of Pb recovered from the Formica board and gypsum board upon impact was found to be relatively low at a level of less than 1 µg/mL.

Table 1. Adjusted mean and 95% confidence interval of the main effects of sampling procedure and target surfaces on the detection of Pb

Factors		Adjusted Mean (95% CI)	F-Statistics (df)	*p*-value
Sampling procedures	Swabbing	1.326 (1.228, 1.424)	63.248 (2)	< 0.001*
	Tape-lifting	1.952 (1.854, 2.050)		
	Scraping	1.225 (1.127, 1.324)		
Surfaces	Perspex	2.068 (1.929, 2.206)	72.509 (5)	< 0.001**
	Plywood	1.265 (1.126, 1.404)		
	Formica board	0.888 (0.749, 1.027)		
	Gypsum board	0.780 (0.642, 0.919)		
	Thin metal sheet	2.018 (1.879, 2.156)		
	Thick metal sheet	1.989 (1.851, 2.128)		

Post-hoc* test: **Swabbing vs Tape-lifting, *p* <0.001; Swabbing vs Scraping, *p*= 0.326; **Tape-lifting vs Scraping, *p* <0.001**.

***Post-hoc* test: **Perspex vs Formica board, *p* <0.001**; **Perspex vs Plywood, *p* <0.001**; **Perspex vs Gypsum board, *p* <0.001**; Perspex vs Thin metal sheet, *p*= 0.615; Perspex vs Thick metal sheet, *p*= 0.431; **Formica board vs Plywood, *p*= 0.003**; Formica board vs Gypsum board, *p*= 0.887; **Formica board vs Thin metal sheet, *p* <0.001**; **Formica board vs Thick metal sheet, *p* <0.001**; **Plywood vs Gypsum board, *p* <0.001**; **Plywood vs Thin metal sheet, *p* <0.001**; **Plywood vs Thick metal sheet, *p* < 0.001**; **Gypsum board vs Thin metal sheet, *p* <0.001**; **Gypsum board vs Thick metal sheet, *p* <0.001**; Thin metal sheet vs Thick metal sheet, *p* =1.000.

Two-way ANOVA also allowed for the determination of whether there was an interaction between the two independent variables (*i.e.*, sampling procedure and surfaces) on the dependent variable (*i.e.*, concentration of Pb detected by AAS). There was a statistically significant interaction between the effects of sampling procedures and target surfaces on the detected concentration levels of Pb [$F(10, 108) = 18.715$, $p < 0.001$]. To compare among the sampling procedures for Pb in each surface, separate one-way ANOVA tests were carried out (**Table 2**). Note that a *post-hoc* test was done to determine which sampling procedures differed from each other and only reported when there was a significant difference among these sampling procedures ($p < 0.05$).

Table 2. Means and standard deviations in the concentration of lead recovered by the different sampling procedures

Surfaces	Sampling Procedures	Mean	SD	F-Statistics (df)	*p*-value
Perspex	Swabbing	1.301	0.4373	15.943 (2)	<0.001
	Tape-lifting	2.484	0.3623		
	Scraping	2.417	0.5086		
	Post-hoc test: **Swabbing vs Tape-lifting, *p* <0.001; Swabbing vs Scraping, *p* <0.001**; Tape-lifting vs Scraping, *p*= 0.958				
Plywood	Swabbing	1.069	0.2577	3.122 (2)	0.069
	Tape-lifting	1.394	0.2029		
	Scraping	1.331	0.3040		
Formica board	Swabbing	0.624	0.2923	7.612 (2)	0.004
	Tape-lifting	1.258	0.2985		
	Scraping	0.782	0.3547		
	Post-hoc test: **Swabbing vs Tape-lifting, *p*= 0.004;** Swabbing vs Scraping, *p*= 0.630; **Tape-lifting vs Scraping, *p*= 0.029**				
Gypsum board	Swabbing	0.716	0.2876	2.933 (2)	0.079
	Tape-lifting	1.027	0.2419		
	Scraping	0.623	0.3738		
Thin metal sheet	Swabbing	1.965	0.2691	31.505 (2)	<0.001
	Tape-lifting	2.572	0.2747		
	Scraping	1.515	0.1989		
	Post-hoc test: **Swabbing vs Tape-lifting, *p*= 0.001; Swabbing vs Scraping, *p*= 0.009; Tape-lifting vs Scraping, *p* <0.001**				
Thick metal sheet	Swabbing	2.281	0.3621	97.413 (2)	<0.001
	Tape-lifting	3.002	0.2388		
	Scraping	0.685	0.3391		
	Post-hoc test: **Swabbing vs Tape-lifting, *p*= 0.001; Swabbing vs Scraping, *p* <0.001; Tape-lifting vs Scraping, *p* <0.001**				

From the six surfaces tested for the recovery of Pb, the choice of sampling procedures did not demonstrate a significant difference for plywood and gypsum board. In other words, whenever a bullet hole was found on such surfaces, the trace residues could be recovered from either swabbing, tape-lifting, or scraping. However, from other tested surface materials, tape-lifting was found to be a better choice, particularly when the trace residues were required to be taken from relatively harder and malleable surface materials, such as Perspex and metal sheets.

Among the three sampling procedures, tape-lifting the trace residues was found to be easy to carry out and did not require any solvent, as in the swabbing procedure, or destruction of the surface material, as in the scraping procedure. By swabbing, the amount of trace residues recovered was found to be lower, and therefore, providing relatively lower detection. This could be due to the adherence of the particles on the swab without being transferred into the acid solution during the sample preparation step. In relation to the scraping procedure, removing the trace residues from a malleable surface was found not to be adequately effective. On the contrary, the adhesive behaviour of the tape could allow for the physical transfer of such residue onto it and be subjected to analysis.

Comparison of the concentration of copper recovered from bullet holes by sampling procedures

The effect of sampling procedures and the surface materials with impact by projectile was tested with two-way ANOVA based on the concentration level of Cu detected by AAS. Assumptions for the statistical test were fulfilled with no significant outliers, normal distribution of dependent variables (Shapiro-Wilk test with $p > 0.05$), and the homogeneity of variances within the dataset (Levene's test with $p > 0.05$). The statistical output is demonstrated in **Table 3**.

Table 3. Adjusted mean and 95% confidence interval of the main effects of sampling procedure and target surfaces on the detection of copper

Factors		**Adjusted Mean (95% CI)**	**F-Statistics (df)**	***p*-value**
Sampling procedures	Swabbing	0.211 (0.202, 0.220)	22.815 (2)	< 0.001*
	Tape-lifting	0.240 (0.231, 0.249)		
	Scraping	0.196 (0.187, 0.205)		
Surfaces	Perspex	0.291 (0.278, 0.304)	168.633 (5)	< 0.001**
	Plywood	0.165 (0.152, 0.178)		
	Formica board	0.151 (0.138, 0.164)		
	Gypsum board	0.106 (0.093, 0.119)		
	Thin metal sheet	0.316 (0.303, 0.329)		
	Thick metal sheet	0.263 (0.250, 0.276)		

Post-hoc* test: **Swabbing vs Tape-lifting, *p* <0.001; **Swabbing vs Scraping, *p*= 0.027**; **Tape-lifting vs Scraping, *p* <0.001**.
***Post-hoc* test: **Perspex vs Formica board, *p* <0.001**; **Perspex vs Plywood, *p* <0.001**; **Perspex vs Gypsum board, *p* <0.001**; Perspex vs Thin metal sheet, *p*= 0.084; **Perspex vs Thick metal sheet, *p*= 0.043**; Formica board vs Plywood, *p*= 0.636; **Formica board vs Gypsum board, *p* <0.001**; **Formica board vs Thin metal sheet, *p* <0.001**; **Formica board vs Thick metal sheet, *p* <0.001**; **Plywood vs Gypsum board, *p* <0.001**; **Plywood vs Thin metal sheet, *p* <0.001**; **Plywood vs Thick metal sheet, *p* < 0.001**; **Gypsum board vs Thin metal sheet, *p* <0.001**; **Gypsum board vs Thick metal sheet, *p* <0.001**; **Thin metal sheet vs Thick metal sheet, *p* <0.001**.

There was a statistically significant difference between the sampling procedures used to recover Cu as determined by ANOVA. The statistical result suggests that adhesive collection was more effective by up to 13.7% in removing the trace residues from the bullet hole compared to solvent-assisted swabbing or mechanical scraping. Similar to the findings regarding the concentration of Pb, among the surface materials with impact holes for Cu recovery, relatively malleable materials were found to contain higher amounts of the target trace element to be recovered and detected by AAS. The statistical test demonstrated a significant interaction between the effects of sampling procedures and target surfaces on the detected concentration levels of Cu [$F(10, 108) = 10.253$, $p < 0.001$]. **Table 4** shows the statistical output for a separate one-way ANOVA test in investigating the effect of sampling procedure for Cu from each surface material, while the *post-hoc* test determines which pairwise comparison of means contributed to the overall significant difference.

Sampling procedure to be taken to sample the trace residue from the bullet holes formed on plywood ($p = 0.476$) and Formica board ($p = 0.218$) was found not to provide a significant difference in the detected concentration of Cu. From the Perspex surface with bullet holes, the swabbing procedure only recovered a relatively smaller amount of trace copper that could be successfully detected by AAS. In the case of gypsum board, tape-lifting was reported to be a better choice for recovery compared to scraping ($p = 0.040$), while no significant difference was noticed for the other two combinations. From metal sheets, regardless of their thickness, either the swabbing or the tape-lifting procedure should be done to recover the trace Cu. Scraping the materials from these surfaces was not adequately effective in successfully recovering a relatively lower amount of trace elements from malleable surfaces, especially metal sheets.

Comparison of the concentration of zinc recovered from bullet holes by sampling procedures

Compared to Pb and Cu, the presence of Zn was detected at a very low level, frequently less than 0.05 µg/mL. The three sampling procedures were compared and investigated using two-way ANOVA, with the surface materials serving as another independent variable (**Table 5**).

Table 4. Means and standard deviations on the concentration of copper recovered by the different sampling procedures.

Surfaces	Sampling Procedures	Mean	SD	F-Statistics (df)	*p*-value
Perspex	Swabbing	0.237	0.0465	11.810 (2)	0.001
	Tape-lifting	0.313	0.0133		
	Scraping	0.323	0.0405		
	Post-hoc test: **Swabbing vs Tape-lifting, *p*= 0.003; Swabbing vs Scraping, *p*= 0.001**; Tape-lifting vs Scraping, *p*= 0.864				
Plywood	Swabbing	0.154	0.0238	0.774 (2)	0.476
	Tape-lifting	0.174	0.0190		
	Scraping	0.168	0.0423		
Formica board	Swabbing	0.145	0.0280	1.661 (2)	0.218
	Tape-lifting	0.165	0.0286		
	Scraping	0.143	0.0190		
Gypsum board	Swabbing	0.099	0.0194	4.307 (2)	0.030
	Tape-lifting	0.123	0.0218		
	Scraping	0.097	0.0119		
	Post-hoc test: Swabbing vs Tape-lifting, *p*= 0.068; Swabbing vs Scraping, *p*= 0.962; **Tape-lifting vs Scraping, *p*= 0.040**				
Thin metal sheet	Swabbing	0.339	0.0289	13.831 (2)	<0.001
	Tape-lifting	0.352	0.0212		
	Scraping	0.257	0.0524		
	Post-hoc test: Swabbing vs Tape-lifting, *p*= 0.784**; Swabbing vs Scraping, *p*= 0.002; Tape-lifting vs Scraping, *p* <0.001**				
Thick metal sheet	Swabbing	0.290	0.0376	32.815 (2)	<0.001
	Tape-lifting	0.311	0.0222		
	Scraping	0.188	0.0293		
	Post-hoc test: Swabbing vs Tape-lifting, *p*= 0.418; **Swabbing vs Scraping, *p* <0.001; Tape-lifting vs Scraping, *p* <0.001**				

Table 5. Adjusted mean and 95% confidence interval of the main effects of sampling procedure and target surfaces on the detection of zinc

Factors		Adjusted Mean (95% CI)	F-Statistics (df)	*p*-value
Sampling procedures	Swabbing	0.086 (0.082, 0.092)	20.402 (2)	< 0.001*
	Tape-lifting	0.088 (0.081, 0.099)		
	Scraping	0.071 (0.068, 0.075)		
Surfaces	Perspex	0.132 (0.126, 0.138)	93.898 (5)	< 0.001**
	Plywood	0.062 (0.056, 0.068)		
	Formica board	0.060 (0.054, 0.066)		
	Gypsum board	0.058 (0.053, 0.064)		
	Thin metal sheet	0.087 (0.081, 0.093)		
	Thick metal sheet	0.091 (0.085, 0.097)		

Post-hoc test: Swabbing vs Tape-lifting, *p*= 0.852; **Swabbing vs Scraping, *p* <0.001**; **Tape-lifting vs Scraping, *p* <0.001**.
Post-hoc* test: **Perspex vs Formica board, *p* <0.001; **Perspex vs Plywood, *p* <0.001**; **Perspex vs Gypsum board, *p* <0.001**; **Perspex vs Thin metal sheet, *p* <0.001**; **Perspex vs Thick metal sheet, *p* <0.001**; Formica board vs Plywood, *p*= 0.997; Formica board vs Gypsum board, *p*= 0.998; **Formica board vs Thin metal sheet, *p* <0.001**; **Formica board vs Thick metal sheet, *p* <0.001**; Plywood vs Gypsum board, *p*= 0.946; Plywood vs Thin metal sheet, *p* <0.001; **Plywood vs Thick metal sheet, *p* < 0.001**; **Gypsum board vs Thin metal sheet, *p* <0.001**; **Gypsum board vs Thick metal sheet, *p* <0.001**; Thin metal sheet vs Thick metal sheet, *p*= 0.011.

Significant differences were evident between the sampling procedures and between the surface materials for the recovery of Zn from the bullet holes. Based on the *post-hoc* tests, both tape-lifting and swabbing significantly allowed for better recovery of Zn up to 21.1% than scraping procedure. Regarding the surface materials, the Perspex surface recovered a greater amount of Zn for detection, which was significantly higher than the other five surfaces tested in this study. On the other hand, the amount of Zn successfully sampled from plywood, Formica board, and gypsum board was of a lower concentration upon detection by AAS.

Statistically significant interaction was evident between the effects of sampling procedures and target surfaces on the detected concentration levels of Zn [F(10, 108) = 4.219, p <0.001]. Subsequent one-way ANOVA statistical tests investigated the effect of sampling procedure for Zn from each surface material, separately (**Table 6**). Note that a *post-hoc* test was only conducted when a statistically significant result was reported.

Table 6. Means and standard deviations on the concentration of zinc recovered by the different sampling procedures

Surfaces	Sampling Procedures	Mean	SD	F-Statistics (df)	*p*-value
Perspex	Swabbing	0.151	0.0493	2.459 (2)	0.114
	Tape-lifting	0.130	0.0174		
	Scraping	0.115	0.0115		
Plywood	Swabbing	0.068	0.0121	3.580 (2)	0.059
	Tape-lifting	0.060	0.0015		
	Scraping	0.059	0.0026		
Formica board	Swabbing	0.060	0.0013	2.461 (2)	0.114
	Tape-lifting	0.061	0.0024		
	Scraping	0.059	0.0018		
Gypsum board	Swabbing	0.057	0.0017	55.96 (2)	<0.001
	Tape-lifting	0.065	0.0029		
	Scraping	0.053	0.0013		
	Post-hoc test: **Swabbing vs Tape-lifting, *p* <0.001**; **Swabbing vs Scraping, *p*= 0.020**; **Tape-lifting vs Scraping, *p* <0.001**				
Thin metal sheet	Swabbing	0.085	0.0054	70.846 (2)	<0.001
	Tape-lifting	0.106	0.0076		
	Scraping	0.070	0.0031		
	Post-hoc test: **Swabbing vs Tape-lifting, *p* <0.001; Swabbing vs Scraping, *p*= 0.002; Tape-lifting vs Scraping, *p* <0.001**				
Thick metal sheet	Swabbing	0.087	0.0073	68.170 (2)	<0.001
	Tape-lifting	0.096	0.0070		
	Scraping	0.070	0.0021		
	Post-hoc test: **Swabbing vs Tape-lifting, *p*= 0.047**; **Swabbing vs Scraping, *p* <0.001; Tape-lifting vs Scraping, *p* <0.001**				

Statistical tests showed that the sampling procedures affected the recovery and detection of Zn from three types of surface materials, namely gypsum board and metal sheet, each reporting a p-value less than 0.001. From these surfaces, all three sampling procedures were found to be significantly different from each other. However, the tape-lifting procedure gave the highest recovery and detection of Zn, suggesting its effectiveness in maximising the amount of trace residue to be detected. On the other hand, no significant differences in terms of the sampling procedures were found in experiments involving Perspex (p = 0.114), plywood (p = 0.059), and Formica board (p = 0.114).

Table 7 demonstrates the selected sampling procedure of Pb, Cu, and Zn across all materials in this study. There were instances in which no significance difference in the recovered concentration of residue by the sampling procedure, particularly from bullet holes shot on plywood. From the experimental outcomes, tape-lifting the internal circumference of a bullet hole demonstrated comparatively higher recovery and detection of target elements, regardless of Pb, Cu, or

Zn. The adhesive tape used for tape-lifting allowed the transfer of the trace residues from the bullet holes onto the tape, and subsequently digested by the acids, followed by the elemental detection by AAS. In other words, the residue transferred from the projectile onto the circumference of bullet holes might have physically adhered to and could be effectively removed onto the tape.

Table 7. Selection of sampling procedure with better recoveries of Pb, Cu and Zn

Surfaces	Sampling Procedure								
	Pb			Cu			Zn		
	Swabbing	Taping-lifting	Scraping	Swabbing	Taping-lifting	Scraping	Swabbing	Taping-lifting	Scraping
Perspex		✓	✓		✓	✓	✓	✓	✓
Plywood	✓	✓	✓	✓	✓	✓	✓	✓	✓
Formica board		✓		✓	✓	✓	✓	✓	✓
Gypsum board	✓	✓	✓	✓	✓			✓	
Thin metal sheet		✓		✓	✓			✓	
Thick metal sheet		✓		✓	✓			✓	

✓ indicates better recovery among the three sampling procedures

Previous studies reported on the successful application of tape lifts to recover trace samples from various surfaces, including the hands and peripheral areas near the impact hole in GSR testing. Utilising tape lift, half of their surface could be subjected to organic GSR analysis, and the other to inorganic GSR detection [20]. A stub with tape attached could cover a larger area during the sampling procedure from hands or other surfaces with the intention of collecting as many trace residues as possible to detect the presence of GSR particles. However, such a stub might not be suitable for collecting samples from the inner circumference of a bullet hole, as the tape could not reach the target area for sample collection, as seen in this study. Therefore, the tape-lifting procedure was modified to capture the trace residues from the bullet hole. The adhesive tape used for GSR sampling was used and it was attached to a stick. Subsequently, the prepared tape-lifter was then gently applied to the internal circumference of the bullet hole (**Figure 2**). The tape-lifting procedure proposed in this study was similar to the modified stub created by Chavez Reyes et al. [21] with the intention of collecting GSR particles from the nose hair.

Swabbing was previously proposed for GSR sampling using an appropriate solvent [3]. It was suggested previously that such a method could have carried less efficiency compared to stubbing [7,22], although comparable results were evident when it was applied to hair [11]. In this study, swabbing using methanol also showed its effectiveness; but it involved the use of solvent, restricting its application if a bullet hole was found at a crime scene and hardly brought to the forensic laboratory. In most instances, a solvent might not be available unless being prepared in the crime scene kit. On the other hand, scraping showed also good recovery of elemental residues from impact holes; however, better results were demonstrated with softer surface materials as they could be easily removed and subjected to digestion. With malleable materials such as metal sheets, the scraping procedure was relatively difficult to perform in which the impact hole might be destroyed by the sampling procedure. Furthermore, the amount of trace residues successfully through scraping procedure was reported to be relatively lower.

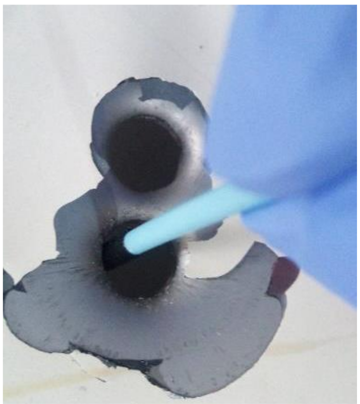

Figure 2. Tape-lifting procedure applied on the bullet hole

The present findings align with previous studies demonstrating the variability of residue recovery depending on sampling technique. Dalby et al. [23] reported higher GSR particle counts using adhesive stubs compared to alcohol-swabbed samples, consistent with the superior recovery of Pb, Cu, and Zn observed for the tape-lifting method in this study.

Minzière et al. [3] emphasised the importance of standardised recovery procedures for inter-laboratory comparability and evidential reliability, a concern also reflected in the current results. Nonetheless, unlike earlier research primarily focused on residues from hands or clothing, this study examined residues from bullet hole circumferences, providing new information into elemental transfer mechanisms during the impact of projectile.

Tape lifting yielded the highest recovery of trace elements compared to swabbing and scraping. From a forensic perspective, this suggests that tape lifting might be more reliable for collecting trace elemental residues at shooting scenes, particularly when non-destructive and quick sampling is required. These findings provide empirical evidence to guide forensic practitioners in selecting appropriate residue recovery methods depending on the substrate and context. It was noted that only one ammunition type and shooting distance were investigated in this study. Variations in bullet composition, surface material, or environmental conditions could affect residue deposition and should be explored in subsequent studies. Although this study successfully compared the efficiency of swabbing, tape lifting, and scraping methods for recovering elemental residues from bullet holes, the sampling parameters were not further optimised under the controlled conditions. Factors such as solvent volume, swabbing duration, surface texture, and sample storage conditions may influence recovery efficiency. Future research shall therefore include systematic optimisation of these variables to establish standardised and reproducible protocols for routine forensic applications.

Conclusion

This study serves as an attempt to investigate the effectiveness of the sampling procedure on bullet holes for elemental detection. Comparing the three sampling procedures, the tape-lifting procedure was found to have successfully recovered greater quantities of trace elements from the bullet holes than the swabbing and scraping procedures. Furthermore, it was easier to carry out without the requirement of any solvent and less tendency to destroy the bullet holes, which could be further examined by the forensic investigators. Tape-lifting procedures involving the gently tabbing and circulation of an adhesive tape attached to a stick onto the impact hole shall be practiced in collecting and recovering trace residues, at least to prove that a hole was previously made by a projectile. Beyond identifying the most efficient technique, these findings emphasise the broader forensic importance of developing and validating standardised sampling procedures. Reliable residue recovery is fundamental to reconstructing shooting events and ensuring that analytical results are admissible and comparable across laboratories. Therefore, the present study contributes to the foundation for standardising residue collection protocols, promoting both scientific robustness and evidentiary in forensic firearm investigations. Future work should expand validation across ammunition types, target materials, and field conditions to establish comprehensive best-practice guidelines for routine forensic application.

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