ZERO WASTE LATENT FINGERMARK DEVELOPING NANOPOWDER FROM EGGSHELLS

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Abstract. The powder dusting technique is the most versatile latent fingermark development method, enhanced through nanotechnology. This research synthesised nanoparticle-based high-definition fingerprint developing powder from agriculture waste. Eggshells produced from local food outlets were subjected to stepwise thermal treatments, carbonisation and calcination at 80 °C (2 hours) and 900 °C (3 hours), respectively. The powder's efficiency for latent fingermark development was tested in two phases; multiple surfaces and multiple donor studies. The first phase tested the efficiency of powder compared to the commercial white powder (SIRCHIE) across varied non-porous surfaces. The second phase was carried out using 150 random fingermark donors. The fingermark was graded based on the ridges' clarity, and scores were analysed. Fine white calcium oxide powder was produced from thermal treatment. Upon application on latent fingermarks, the synthesised powder exhibited high selectivity and sensitivity to fingermark residue, resulting in clear ridge details. Independent t-test analysis of fingermark grades in Phase 1 ($t_{plastic} = -2.366$, p-value = 0.031; $t_{metal} = -0.849$, p-value = 0.409; $t_{glass} = -0.918$, p-value = 0.372) revealed significant difference in development on plastic surface alone attributed to the adhesive property of the SIRCHIE. Meanwhile, phase 2 data ($t_{male} = -3.809$, p-value <0.001; $t_{female} = -3.145$, p-value = 0.002) showed that there was significant improvement in the clarity and contrast of the fingermarks developed using calcium oxide powder. Findings of this research may contribute to increased fingermark recovery in the crime scenes and provide a safer and cost-effective alternative to the commercial white powder.

Keywords: Eggshells, fingermark, powder dusting, calcium oxide, waste upcycling

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1. INTRODUCTION

The soles and palms have distinguished parallel ridge patterns to friction during walking and holding things. Fingers or footprints are the impressions formed by these parallel ridges on another surface and are unique to each individual. The entirely coincidental formation of these parallel ridges during conception makes the patterns unique. The fingermarks ridge pattern unconsciously deposited on any surface during contact is a significant piece of evidence often left behind at the crime scene. Law enforcement officers attempt to visualise, recover and identify these impressions for various purposes. Dactylogists match the fingermarks to known fingerprints by analysing the flow of the parallel ridges. Although, for the most part, the ridges move parallel to each other, they also tend to form endings, bifurcation (splitting of one ridge into two), lake, eyelet and so on. These patterns are called the minutiae (secondary level details), and matching them can produce identification [1].

The finger ridge impressions left at a crime scene may vary from visible to invisible marks, depending on the surface and colourants that came into contact with the ridges before deposition. Accordingly, three distinct types of fingermarks can be found at a crime scene, plastic, patent and latent marks. Plastic marks are often left on a soft solid surface like clay or putty, while patent marks contain colourants such as blood or paint to give the naked eye immediate visibility of the ridges. Nevertheless, most of the marks left at a crime scene are invisible to the human eye and require a development process [2]. The development techniques of latent fingermarks can vary from surface to surface. Surfaces can be porous, non-porous or semi-porous, depending on the void space between substrate molecules. The powder dusting technique is one of the forefronts of fingermark development techniques applied in-situ during a crime scene investigation. Although the powder dusting technique is most suitable for non-porous surfaces, applicability over a wide range of surfaces and the time and cost-saving factors contribute to favourability. Mobile or packable evidence is treated accordingly with other techniques, for example, cyanoacrylate or ninhydrin fuming, amido black, oil red O, and leuco crystal violet solution [1].

Various powders with different properties and colours are available for latent fingermark development on varied surfaces. Granular, metallic flakes, magnetic, and fluorescent powder are among the variety of powders available, and the use depends on the contrast required for the particular mark. A bristled brush carries the non-magnetic powder to the surface in one-directional strokes until the ridges obtain good visibility. The powder particles leave the bristle and mechanically adhere to the sticky residue left behind during contact [3]. The composition of latent fingermark residue may vary according to the individual and the deposited residue's age. Although the difference in residue composition due to individual variations is difficult to account for, an approximate degradation pathway can be theorised. Freshly deposited latent marks contain moisture, sebaceous and eccrine secretions. Water will evaporate first, followed by water-soluble components due to the residue's constant contact with humid air. Generally, the sebaceous secretions persist more extended than the other parts of fingermark residue. Nevertheless, this pathway of fingermark residue degradation is limited in a dry and moderately humid environment. Due to the dynamic nature of environmental conditions, the approximation of the fingermark degradation process does not apply to other weather conditions [4].

Successful recovery of the ridge pattern by powder dusting is directly related to the fingermark residue's quality. In almost all cases, environmental factors and the fingermark residue composition cannot be controlled; therefore, improvising the reagents' efficiency for the latent fingermark development and recovery is imperative. This has become the driving force behind the plethora of research in improving and enhancing the fingermark development techniques. Ergo, copious research aims to improve the fingerprint powder's quality to optimise the powder formulation's sensitivity and selectivity to work on a broader fingermark recovery condition. Accordingly, plenty of researchers investigated the efficacy of industrially manufactured metals, metal oxides, and colorants from minerals as powdering agents [5]. Yet, ingredients added to increase adhesion, such as resins and kaolin, often compromises the powder's selectivity. These extraneous substances promote non-selective adherence of the powder particles to the surface bearing the fingermark, lowering the contrast.

Recently, greener methods include naturally occurring pigments such as turmeric, chilli powder, spice powders, food colourings, gambir, and limestone powder have been explored as alternative powders for fingermark development [1]. Some of these powders worked well as a developing agent, while some produced fragmented ridges and uneven powder distribution. On the other hand, nanomaterial fabrication advancements have prompted many researchers to incorporate nanoparticles with enhanced properties and functionalities into fingermark development [6]. Specific tuning of nanoparticles' size, shape and surface properties allows focused targeting of the fingermark residues' components and versatility in developing fingermarks on varied types of substrates. Multiple nanoparticle-based techniques, such as multimetal and single metal deposition, exhibited breakthroughs in developing too faint and aged fingermarks. Despite the narrow window of development conditions for the methods to work optimally, these methods have been useful for recovering fingermarks where all other methods failed due to the enhanced sensitivity and selectivity [7]. Alternatively, metal oxide nanoparticle-based techniques using titanium dioxide, aluminium oxide and zinc oxide have been reported to elucidate identifiable fingermark ridges as well. Quantum dots, which are extensively studied due to their unique photo-optical properties that can elicit better sensitivity towards fingermark residue, often fail to accommodate surface functionalisation while retaining optical properties [8].

Silica nanoparticles considered an underdog in the arena of fingermark development, recently began garnering more research interest as researchers started to observe silica's versatility. The shape, optical properties and easy surface functionalisation make silica a favourable alternative for latent fingermark development. Silica particles of various sizes doped or functionalised with dyes has been reported to be efficient for latent fingermark development. However, research optimising the size, shape, aggregation and surface functionalisation is still being undertaken [9]. An alternative advance green technology approach in fingermark research is synthesising highly functional materials using low-cost resources such as agricultural and food wastes. Past research has reported the successful derivation of silica nanoparticles with the size and shapes tuned for optimal fingermark development by utilising eco-friendly resources. Tandem synthesis of carbon and silica nanoparticles from thermochemical treatment of rice husks produced black and white fingermark powder (ECO^{fp}). The regular formulations of these powders have been proved to develop high definition fingermarks on several non-porous and semi-porous surfaces [10].

Dye doping the silica nanoparticles with curcumin pigment extracted from turmeric produced a fluorescent variant that enhanced contrast on colourful surfaces [11]. The powder's selectiveness owing to the small-sized, spherical, and minimally clustered particles sets the powder apart from other researches involving fingermark development using silica.

Recently Sankhla et. al, 2024 reported the synthesis of eggshell nanosheets with size ranging from 30 to 90 nm for the development of latent fingermark on various surfaces. Authors reported that the powder performed well on all surfaces [12].

This research focused along the similar lines of reutilising waste for fingerprint powder development. The municipal solid waste principally consists of food waste generated in Malaysia, has reached about 10 million tonnes in 2020. Unlike recyclable items, agricultural and food wastes rarely get recycled or composted. Many studies have reported low-cost material synthesis from eggshell wastes. One such use is the preparation of hydroxyapatite nanoparticles from eggshell wastes for antibiotic removal from water [13]. Other applications of eggshells as a catalytic agent, membrane template, fertilisers, food additives, and bio-nanocomposites makes it a versatile natural resource [14]. We have attempted to re-use chicken eggshells for latent fingermark development in the present research. High mineral content in the chicken eggshells (Calcium 38.2%, Carbonate 44.3% with Sodium, Phosphate, Sulfate, Potassium, Strontium and Fluoride present in less than 0.5%) [15] makes them the right candidate for fingerprint powder production with minimal treatment.

The objective of the current work was to reduce food waste generation and simultaneously generate an upscaled product that could be a source of revenue. Hence, we employed calcium oxide nanoparticles (CO_{NP}) synthesised by heat-treating eggshells as a latent fingermark developing agent. A simple calcination process converted the eggshells into fine white nanoparticle powder. We expected the calcium oxide nanopowder to exhibit enhanced sensitivity to latent fingermarks residue due to the small and rounded particles' nature.

2. MATERIALS AND METHODS

2.1 Synthesis and Characterisation of CO_{NP} from Eggshells

Eggshells procured from local eateries were washed and dried in an oven at 90 ℃ for five hours. The inner membrane was removed from the dried shells before being ground into powder using a regular counter-top blender. The powder was further sieved to remove large particles. Thermal treatment of the powdered eggshells at 80 ℃ for 4 hours on hotplate produced semi-charred coarse powder. Subsequently, the powder was calcined in the furnace at 900 ℃ for five hours to convert the char into a fine white powder used for fingermark development without any further modifications. The surface texture and particle agglomeration were visualised using LEO 1525 Field Emission Scanning Electron Microscopy (FESEM) (LEO Electron Microscopy Inc. Thornwood, NY). The sample was mounted on a carbon tape coated aluminium stub and sputter-coated with gold before viewing at a working distance of 2mm using a 15.00kV electron beam to observe the morphology and size of the particles. The sample's elemental and chemical composition was determined using Energy Dispersive X-Ray (Oxford Insruments INCAx-act, 51-ADD0011) and Fourier-Transform Infrared Spectroscopic (Nicolet Summit, Thermo Fisher Scientific) techniques.

2.2 Natural Fingermark Collection, Development and Grading

Donors were requested to wash their hands using tap water and resume routine 30 minutes before fingermark deposition. The fingermark deposition was performed without charging the residue by touching other body parts to obtain natural fingermark constituents. Two pieces of the same surface were kept side by side, and the donors were assisted in depositing their fingermark to acquire one half on each side of the substrate, splitting the fingermark into equal halves (longitudinal). Assisted fingermark deposition was performed on the assigned surfaces to ensure constant deposition force and contact time. The fingermarks were aged for an hour before development. One half of the fingermark was developed using the Indestructible White SIRCHIE powder using SIRCHIE squirrel hairbrush, both purchased from SIRCHIE Inc., Youngsville, NC, USA. The other half was developed using the synthesised CO_{NP} powder using the SIRCHIE squirrel hairbrush. The developed fingermarks were photographed using iPhone X with 12 megapixels, a six-element lens, quad-LED. The fingermark developed was displayed on the laptop screen and evaluated according to CAST (Centre for Applied Science & Technology) holistic grading scheme [16] (Table 1).

Table 1. CAST grading scheme for the assessment of developed fingermark

Grade	Detail Visualised
0	No evidence of a fingermark
1	Some evidence of a fingermark
2	Less than 1/3 clear ridge detail
3	Between 1/3 and 2/3 clear ridge detail
4	Over 2/3 clear ridge detail

2.3 Powder Efficiency Testing

The synthesised powder's efficiency compared to the existing powder in the market (SIRCHIE Indestructible White) was conducted in two phases, each phase to determine the powder's effectiveness across varied substrates and donors, respectively. In Phase 1, three subjects (robust, medium and weak donors) and three substrates (metal, glass and acrylic PVC) were studied. The donor type was established by developing several natural fingermark samples at different intervals. Robust donors would form clear and thick ridges, and medium donors contribute to thin and continuous ridges while the fingermark from weak donors will be fragmented, discontinuous or incomplete. Donor type can be easily determined by developing several natural fingermarks on any non-porous surface using SIRCHIE black powder. The black powder was used in this instance to avoid biases.

Each donor was requested to deposit one fingermark on each surface, the test done in triplicates. Following Phase 1, one optimal substrate with clear fingermark development was selected for Phase 2. A total of 150 donors comprising of 75 males and 75 females were recruited for this phase. Each donor deposited one fingermark on the selected surface (acrylic PVC). We appropriated the split fingermark methodology to maintain the consistency between both halves of the tested fingermarks. Both halves developed using the Squirrel-hair brush by SIRCHIE by the same person but using control (SIRCHIE Indestructible White) and CO_{NP} powders for each half, respectively. Each half was graded accordingly, and the mean difference between both gradings was determined using an independent t-test or Mann-Whitney U-test following the normality analysis.

3. RESULTS AND DISCUSSION

Synthesis of CO_{NP} was achieved through a simple thermal assisted decomposition resulting in zero waste. CO_{NP} powder applied using powder dusting technique brought forth continuous ridgelines with minimal undesirable powdering over the fingermark valleys. Preliminary efficiency studies revealed that CO_{NP} developed fingermark with sufficient details for identification purposes regardless of the donor and surface variation.

3.1 Morphological and Structural Composition of CO_{NP}

The calcination of the powdered eggshells produced a lightweight white powder (Figure 1). A clear transition from the block like structure with sharp edges in the dimensions of 20-50 µm (Figure 2(a)) into finely dispersed particles (Figure 2(b)) can be observed after calcination of the eggshells. However, higher magnification images of the eggshells both before and after heat treatment displayed highly agglomerated particles in the dimension of 10 to 20 nm, as illustrated in Figures 2(c) and (d).

Table 2 reveals the elemental metamorphoses owing to the heat treatment, whereby the sulphur was removed, and a reduction in carbon content was evident. There was an apparent increase in the element's oxygen, magnesium, and calcium percentage composition. Magnesium is a naturally occurring metallic element in the eggshells, and the heat treatment was not successful in removing it.

The structural analysis performed using infrared wavelengths showed no observable differences between the spectrum of raw eggshells and CO_{NP} powder (Figure 3). A similar study characterising the calcium oxide from eggshell waste reported that with increasing calcination temperature at an interval of 100 ˚C from 700 ˚C the sharp peak at 29.48˚ slowly disappears and they recorded the appearance of new peak at 34.11˚ with calcination temperature above 900 ˚C. The shift in the peak indicate the transformation from calcium carbonate to calcium oxide [13].

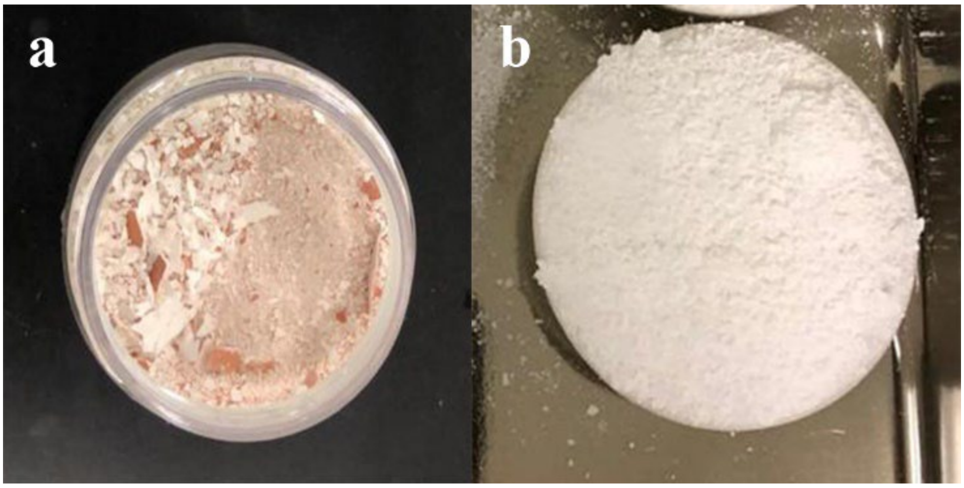

Figure 1: Eggshell powder a) before and b) after calcination

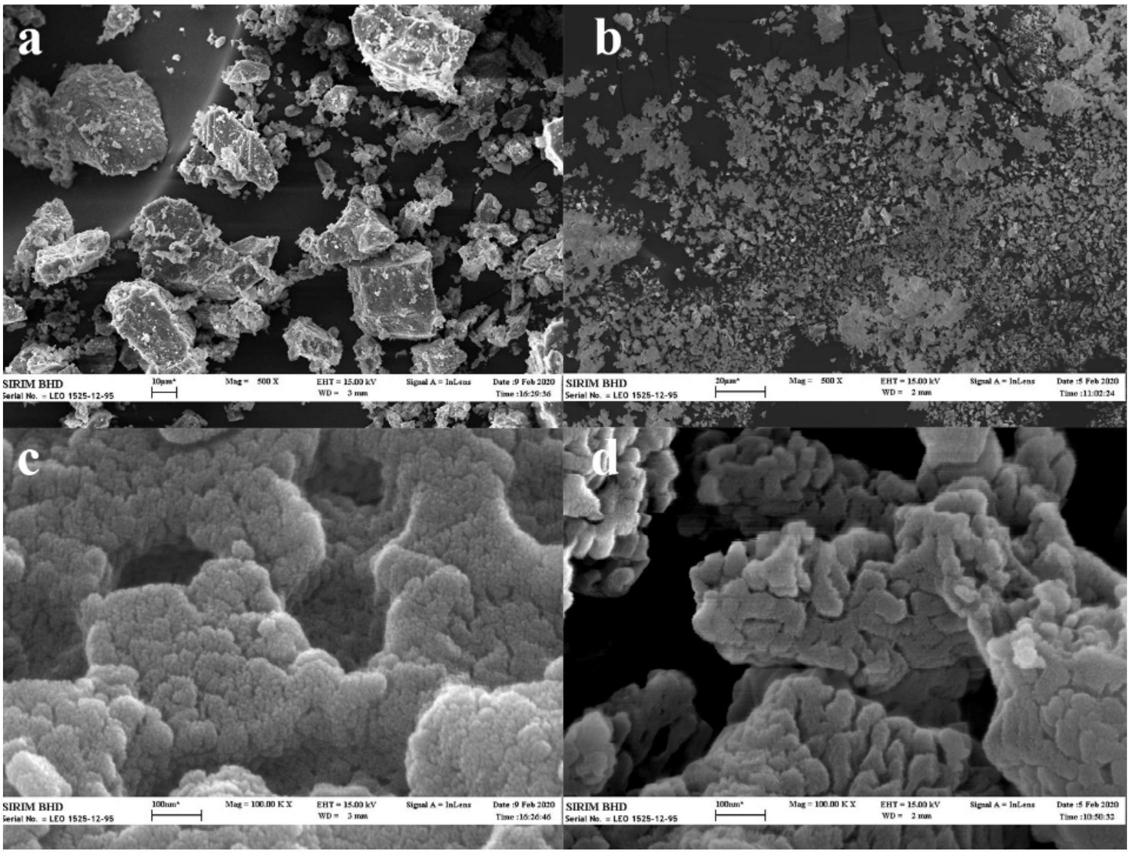

Figure 2: FESEM micrographs of (a) calcium carbonate (untreated eggshell, 500x), (b) CO_{NP} (after calcination treatment, 500x) (c) calcium carbonate (untreated eggshell, 100 000x) and (d) CO_{NP} (after calcination treatment, 100 000x)

Table 2: Elemental composition of eggshell powder before and after heat treatment at 900 °C for five hours

Elements	Before calcination (Atomic %)	After calcination (Atomic %)
Carbon, C	19.03	10.30
Oxygen, O	61.41	67.64
Magnesium, Mg	0.69	0.85
Sulphur, S	1.56	-
Calcium, Ca	17.31	21.22

The major peaks observed in the eggshell powder spectrum specifically at 1405.99 cm^{-1}, 872.60 cm^{-1}, and 712.02 cm^{-1}, are characteristic of the C-O bond. The presence of carbonate minerals within the eggshell matrix can be significantly linked to the sharp peak of the eggshell particle detected at 1405.99 cm^{-1}. Additionally, there are two discernible peaks at 712 and 876 cm^{-1}, corresponding to in-plane and out-of-plane deformation, respectively, suggesting the presence of calcium carbonate ($CaCO_3$) [13]. On the other hand, the major peaks observed in the calcium oxide powder spectrum specifically at 1443.99, 1065.08, 874.38 and 719.04 cm^{-1}, are characteristic of the C-O bond (ether group). Furthermore, the peak at 3640.14 cm^{-1} corresponds to the OH^- group, indicating the presence of hydroxide within the calcium oxide powder. The overall observed peaks suggest a possible mixture of carbonate and hydroxide components in the sample.

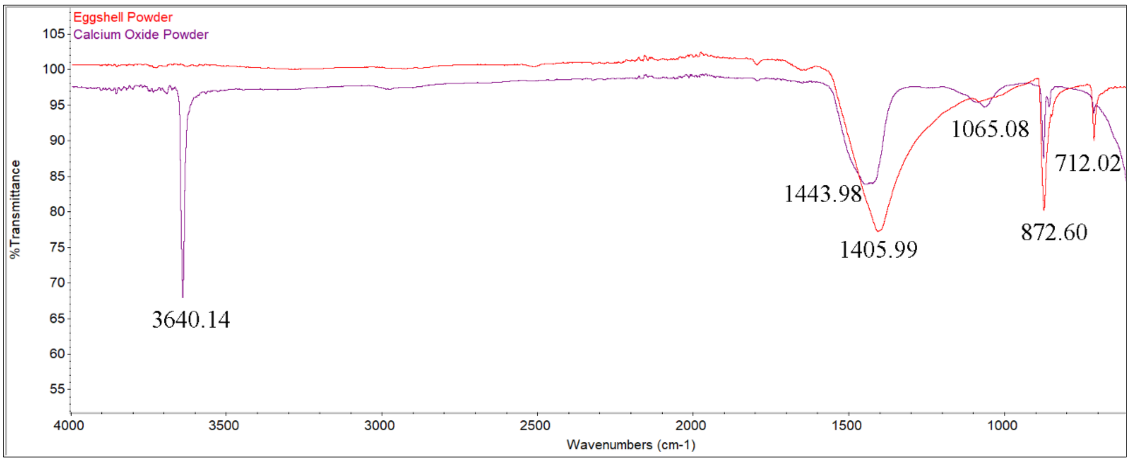

Figure 3: FTIR spectra of raw eggshell and CO_{NP} powders studied under the absorbance range of 400 cm^{-1} to 4000 cm^{-1}

3.2 Fingermark Ridge Clarity and Contrast

Ridge clarity and contrast is essential component of fingerprint identification. Higher ridge clarity and contrast enables the identification of minutiae that can lead to the successful identification of an individual. However, latent fingerprint identification can become cumbersome when developed latent fingermarks exhibit poor ridge details. Therefore, ridge development is a rather essential and critical process before extracting the features [17].

The latent fingermarks developed using CO_{NP} and SIRCHIE powder on varied non-porous surfaces revealed good fingermark ridge definitions. Notably, the CO_{NP} powder was more regio-selective to the ridges showing minimal background powdering. The excessive powdering resulting from SIRCHIE powder dusted on acrylic plastic surfaces containing fingermarks was more evident. The currently experimented CO_{NP} powder does not contain any additives or resin that will confer adhesive property to the powder particles. The particles were small-sized and rounded. The physical attributes of the CO_{NP} powder allowed for unassisted uniform adhesion on the ridges, forming precise ridge details and minimising unspecific adhesion of powder particles. These traits resulted in lower background interaction, as observed in Figures 4 and 5. Size of developing particles are crucial in latent fingermark development to confer higher resolution of the ridges details. Larger particles will obscure the valleys between the fingermarks and lead to loss of minutiae identification.

Figure 4 shows that the CO_{NP} elucidated clear but faint ridge details on the acrylic surface compared to glass and metal surfaces, and less background powdering on all three surfaces than SIRCHIE powder. On the other hand, in Figure 5 CO_{NP} display ridge-selective particle adhesion, minimising the ridge blurring.

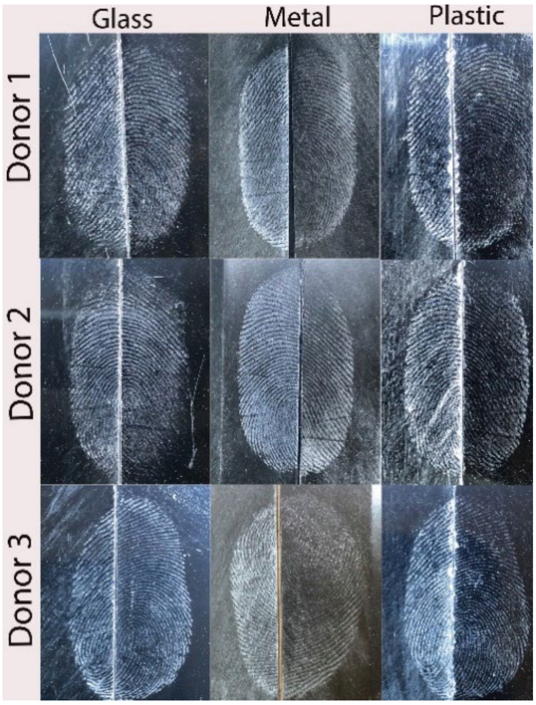

Figure 4: Latent fingermarks from donor 1 to 3 developed using CO_{NP} (right half) and SIRCHIE white powder (left half) on glass, metal and plastic surface

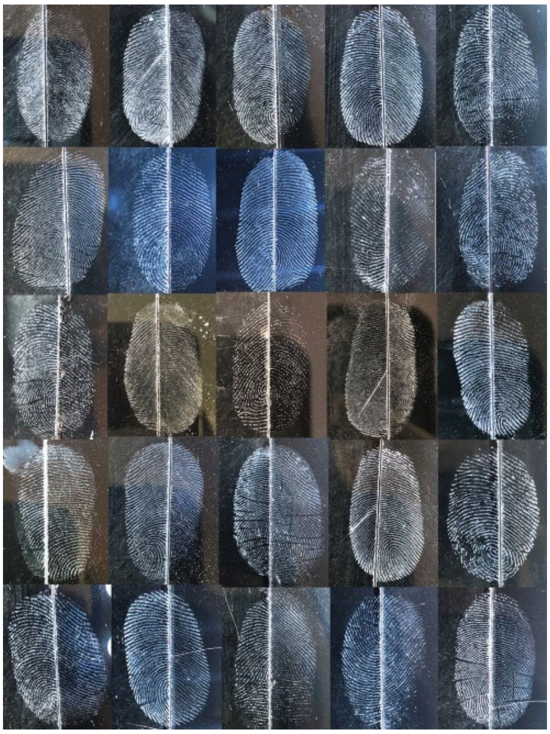

Figure 5: A sample of latent fingermarks from 25 donors (female), developed using CO_{NP} (right half) and SIRCHIE white powder (left half)

Most conventional fingerprint powders consist of an additive such as aluminium and silicon. Meanwhile, SIRCHIE white powder consists of a resinous adhesive polymer (starch, rosin, silica gel, kaolin, etc.) and a contrast colourant (aluminium flake) to enhance the fingermark developed by improving contrast. These adhesive components cling to the sweat residue's moisture and oil components by the pressure deficit mechanism, acting as a primary adsorption layer. The colourant behaves as a secondary particle attached to the adhesives imparting colour to the developed ridges. This entire process results in ridges' visualisation, as observed by the unaided eye [18]. However, the addition causes the product to clump after short term storage especially in a highly humid environment. CO_{NP} remained stable and free of clumps even after long term storage making the powder an attractive alternative to the commercial formulation. The pure white colour of the CO_{NP} also performs comparably to the SIRCHIE Indestructible White powder in generating sufficient contrast between the ridges and the background.

3.3 Surface Interactions

Fingermark residue is primarily made up of the eccrine sweat, excreted through pores present along the ridges. These secretions typically contain trace amounts of fatty acids, proteins, amino acids and inorganic salts. Powder selectivity is highly dependent on the nature of the surface and its ability to absorb fingermark residues [18]. The non-porous surface repels and does not absorb moisture. Therefore, the fingermark residues deposited remain on the surface when transferred, resulting in higher mechanical adhesion between residue and fingerprint powder. This phenomenon depicts why the powder dusting method for latent fingermark development works well on non-porous surfaces.

On the other hand, the porous surface absorbs the moisture from fingermark residue resulting in low mechanical adhesion of fingerprint powder, resulting in poor fingermark development after a certain period. Although plastic surfaces are classified as non-porous surfaces, there are few exceptions to this category. Some plastics can absorb moisture from the surrounding atmosphere. The plastic used in this experiment was derived from a polymer called Poly-(methyl methacrylate) (PMMA), a transparent thermoplastic known as acrylic. PMMA is quite hygroscopic and, given its water insolubility, it can absorb about two per cent weight of moisture. The hygroscopic property also results in serious changes in PMMA materials' dimensions. These materials are known as "hygroscopic plastics" [1].

Acrylic PVC plastic polymer used as a substrate exhibits a strong affinity to absorb moisture, resulting in increased penetration of latent fingermark components into the surface over time. After some time, the fingermark residue's surface diffusion creates a "halo" like appearance around the fingermark. Consequently, the development of fingermarks on this surface results in a high background interaction, decreasing the ridge clarity. The "halo" development is more evident in the SIRCHIE white powder due to adhesive components initially intended to increase particle adsorption to ridges. The fingermarks developed on metallic surfaces were of lower quality for both powders, attributed to the micro or sub-micron scale scratches on the surface, leading to uneven fingermark residue transference.

An overall evaluation of both powders' performance using independent t-test analysis on both powders' grading scores revealed a significant difference on the acrylic surface (Figure 6). The CO_{NP} powder exhibited a higher level of ridge clarity on the acrylic surface with a mean score of 3.22 compared to SIRCHIE white powder with a mean score of 2.44. Multiple donor studies also showed that CO_{NP} powder worked significantly better in developing fingermark ridges of higher quality than SIRCHIE powder on acrylic surfaces for both genders (p-value < .005). The findings of study also indicates that further research should be delved into the sub-division of non-porous surfaces into smaller categories and enhance the powder dusting efficiency by using powders tailored to each type of non-porous surface.

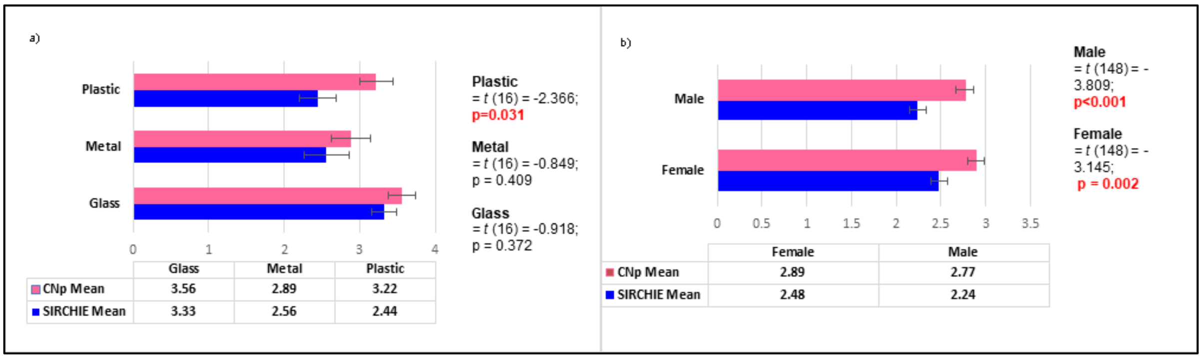

Figure 6: Mean comparison analysis of the fingermark grades developed using SIRCHIE white, and CO_{NP} powder performed using independent t-test (a) multiple surface studies and (b) multiple donor studies

Nevertheless, proper personal protective equipment should be donned during the application of the CO_{NP}. The CO_{NP} may react with moisture in the air exothermically, which may release large amounts of heat. Therefore, inhaling a large amount of CO_{NP} or direct skin exposure could cause caustic effects. However, there is no report of carcinogenicity effect from CO_{NP} exposure, which can be said to be true in the case of titanium dioxide (primary colourant of SIRCHIE white powder). Particulate calcium oxide (CaO) may be harmful to user in terms of acute health effects such as respiratory passage inflammation, nasal septum ulceration and perforation, and pneumonia which are related to CaO dust inhalation [19]. This is due to the alkalinity of CaO that may irritates the skin and the upper respiratory tract. Therefore, the use of personal protective equipment is required to prevent acute health effects.

4. CONCLUSIONS

All in all, this naturally occurring, cheap, and environmentally friendly method of synthesising fingerprint powder derived from eggshell is capable of developing good quality latent fingermark based on the clarity of the ridges on dry, dark non-porous surface. The findings of this research revealed that while relatively CO_{NP} white powder was more suitable for surfaces like glass, it was as well proved to be good in visualising latent fingerprints on metal and plastic surfaces. On top of that, the overall fingermark development performance of CO_{NP} white powder on dark non-porous surface evaluated following the International Fingerprint Research Group (IFRG) guidelines and protocols indicated that CO_{NP} powder may provide a good and safer alternative powder for fingermark identification in comparison to commercially available powders. Future work may be conducted to further test the performance of the powder by applying on many more surfaces, followed by phase three (validation via Pseudo-operational trials) and phase four (operational evaluation and casework trials) based on IFRG guidelines and protocol. Nevertheless, this research has shown the potential to be used in a crime scene.

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Author Contributions

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Sofia Haniza Mohd Zabit and Nik Fakhuruddin Nik Hassan. The first draft of the manuscript was written by Revathi Rajan and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Disclosure of Conflict of Interest

The authors have no disclosures to declare

Compliance with Ethical Standards

The human ethical clearance was approved by Human Research Ethics Committee, Universiti Sains Malaysia (reference number: USM/JEPeM/20120615.

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