FROM WASTE TO LATENT TRACES VISUALIZATION: THE APPLICATION OF POLY(ETHYLENE TEREPHTHALATE)-BASED POWDERS IN DEVELOPMENT OF LATENT FINGERPRINTS

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PURPOSE

Polymers are natural or synthetic, organic or inorganic compounds that possess numerous specific properties. They are among the most utilized materials, due to their versatile and cost-benefit properties, particularly their high durability and strength (Young & Lovell, 2011). Molecular structure, low density of repeating units (monomers), strong (covalent) bonding, structural organization and additives/reinforcements together provide specific properties of polymers, suitable for application in different industries (Carraher Jr, 2017). Polymers consist of long chains of monomers, typically lightweight compared to their volume, which contribute to the low specific gravity of these materials. Besides being lightweight, the strong covalent bonds formed between the monomer units contribute to the high strength-to-weight ratio of polymers. These properties have influenced the extensive usage of polymers in many fields, although their lifespan often exceeds the needs of the application (Mikitaev, Ligidov, & Zaikov, 2006). They can also be combined with other compounds to obtain a desired material for a specific application (van Krevelen & te Nijenhuis, 2009).

Based on their chemical structure and response to heat, polymers are classified as thermosetting and thermoplastic (Massy, 2017). Thermosetting polymers or thermosets undergo a chemical reaction when heated, creating a three-dimensional network of bonded molecules (Pascault & Williams, 2013). This process is irreversible, meaning that once thermosets have been set, they cannot be melted or reshaped. Therefore, these materials can be recycled only through chemical degradation of their three-dimensional structures, where mono-oligomers and/or fillers are recovered at the end of the cycle (Morici & Dintcheva, 2022). Thermosets are typically hard, strong, and have excellent resistance to heat and chemicals, due to strong chemical bonds and crosslinked chains formed during the curing process (Pascault & Williams, 2013). Some common thermosets include melamine, epoxy resins, polyester resins, silicone, urea-formaldehyde resins, polyurethane, polyvinylidene fluoride, etc. (Mamunya & Iurzhenko, 2012; Pascault & Williams, 2013; Massy, 2017). On the other hand, thermoplastic polymers or thermoplastics are solids at room temperature that have linear molecular chains. Upon heating, they become soft but hardened when cooled since there are no chemical bonds involved. These polymers could be poured into moulds to cool and solidify into desired shapes (Van de Velde &

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Kiekens, 2001). Additionally, they can be reheated, recycled, and remoulded without any risk to their material properties. Polycarbonate, polyethylene, polystyrene, poly(vinyl chloride), polypropylene (PP), poly(ethylene terephthalate) (PET), and polytetrafluoroethylene are typical thermoplastic polymers. These materials have been used for plastic injection moulding, extrusion, and thermoforming processes, and generally tend to resist shrinking while offering good strength and elasticity (Van de Velde & Kiekens, 2001; Heckele & Schomburg, 2003; Bîrcă, Gherasim, Grumezescu, & Grumezescu, 2019; Dhinakaran, Surendar, Hasunfur Riyaz, & Ravichandran, 2020).

Polymeric materials have been used for many years in various industries, including packaging, agriculture, silviculture, fishing, water and waste treatment, electronics, automotive, medicine, dentistry, and pharmacy (Milašinović, Knežević-Jugović, Milosavljević, Filipović, & Kalagasidis Krušić, 2012; Namazi, 2017; Vallejos, et al., 2022; Khah & Soleimani, 2023; Vučković, et al., 2024). For example, nylon, aramid, polyester (PET), and poly(vinyl alcohol) have been widely used in synthetic fibre manufacture for textiles. Various countries have moved away from paper currency and manufacture money from polymer films (generally PP), due to longer lifespan and cleanliness. Polymers have also been used for personal protective equipment, car parts, paints, glues, household items, lubricants, packaging, containers, etc. (Namazi, 2017; Al-Ghraibah, Al-Qudah, & Al-Oqla, 2020; Bhagyaraj, Oluwafemi, & Krupa, 2020; Kumar, Verma, & Khare, 2021; Vallejos, et al., 2022; Khah & Soleimani, 2023).

However, the massive production and consumption of polymeric materials have led to the generation of huge amounts of polymeric (plastic) waste. The annual growth in global polymer production has surpassed global gross domestic product and population growth (Coates & Getzler, 2020). As a consequence, polymeric materials can be found everywhere – in air, soil, water and/or biological organisms. High durability and strength, which are beneficial for their exploitation, represent huge flaws upon cessation of their application, as these materials are being thrown into landfills, thus polluting the environment (Da Silva & Wiebeck, 2020; Valerio, Muthuraj, & Codou, 2020; Prlainović, Milovanović, Milašinović, Bezbradica, & Mijin, 2024). Therefore, environmental scientists and other researchers are attempting to develop sustainable recycling technologies for polymeric waste, but these still remains a significant challenge.

There are several methods/approaches used for the mechanical recycling of thermoplastic polymers. Primary mechanical recycling involves direct reuse of uncontaminated discarded polymer as a new product, with initial/unchanged properties. In most cases, primary recycling is conducted by the manufacturer itself for post-industrial waste, often referred to as closed-loop recycling (Al-Salem, Lettieri, & Baeyens, 2010; Baillie, Matovic, Thamae, & Vaja, 2011). Since exact content and purity grade of end-of-life and post-consumer streams are usually unknown, polymers are processed through secondary mechanical recycling, which includes separation and purification. The polymer is not changed during the secondary recycling, but its molecular weight decreases due to chain scissions in the presence of water and small volumes of acids, which can cause the weakening of mechanical properties (Achilias, 2012). Feedstock or tertiary recycling involves the conversion of polymeric chains to smaller molecules through chemical processes, such as hydrolysis, pyrolysis, hydrocracking, and gasification. Typical conversion products are liquids and gasses, which can be used as feedstock for the production of new polymers, fuels, chemicals, etc. (Patel, von Thienen, Jochem, & Worrell, 2000; Brems, Baeyens, & Dewil, 2012). Biological degradation could be used as an improved form of the tertiary recycling, as considered by some research groups (Hopewell, Dvorak, & Kosior, 2009; Li, et al., 2013). Some polymers can be degraded in the presence of air and water (Mülhaupt, 2012) into smaller molecules by bacteria (Cho, Moon, Kim, Nam, & Kim, 2011), fungi (Schnürer & Schnürer, 2006), and other microorganisms that biosynthesize relevant enzymes (Gross & Kalra, 2002). This form of degradation clearly maintains the intrinsic value of waste materials and returns them into the biological cycle (Ignatyev,

Thielemans, & Vander Beke, 2014). Finally, incineration, or quaternary recycling is especially used for processing mixed and heavily contaminated wastes that cannot be easily and/or economically recycled by other methods. Burning high-energy waste can create heat, electricity, or other forms of energy, which can be used directly in technological processes or for heating buildings. This method reduces waste volume to roughly 1% of the initial volume and decomposes toxic and contagious waste (Al-Salem, Lettieri, & Baeyens, 2010). Therefore, incineration is suitable for recycling medical applications and packaging of hazardous materials, while inorganic constituents converted to inert slag through incineration can be used for road construction (Brems, Baeyens, & Dewil, 2012; Grigore, 2017).

Biodegradable polymers represent a class of "green polymers", implying that they do not exhibit detrimental effects on human health and the environment (Vučković, Glođović, Radovanović, Janaćković, & Milašinović, 2020). However, the degradation of such polymers in landfills typically leads to losing the value of these materials, while their decomposition in the oceans could potentially create environmental imbalance (Hong & Chen, 2017). On the other hand, industrial mechanical recycling usually results in a loss of quality. One of the best sustainable solutions is to develop chemically recyclable polymers that not only solve the end-of-life issue of polymers, but also provide a direct approach to establish a circular economy (Shanmugam, et al., 2020; Valerio, Muthuraj, & Codou, 2020). Revalorization of PET packaging material not only addresses a major pollution issue but also fosters a more sustainable and eco-friendly practice. Such a recycling strategy includes either a depolymerization or a repurposing process. Polymer wastes are depolymerized under controlled conditions back to their starting feedstocks that are purified and then subsequently repolymerized to yield high-quality polymeric materials. In a repurposing process, polymer wastes are converted to "building blocks" for new and value-added polymeric materials (Hong & Chen, 2017; Sikorska, Musioł, Zawidlak-Węgrzyńska, & Rydz, 2021; von Vacano, et al., 2023).

In this research, we investigated the depolymerization of PET packaging material (bottles) to prepare powders for developing latent fingerprints. PET is a thermoplastic and one of the most widely used and versatile polymers, but it is also one of the major environmental pollutants (Dhaka, et al., 2022). Although some research groups proposed different approaches of PET recycling and further application of recycled material (Welle, 2011; Pudack, Stepanski, & Fässler, 2020; Jang, Sadeghi, & Seo, 2022; Smith, Takkellapati, & Riegerix, 2022; Li, et al., 2023; Bian, Xia, Xin, Jiang, & Ma, 2024), none have specifically addressed the recycling of PET for the preparation of PET-based powders used for the visualization of latent fingerprints in forensic examination. In this study, powder formulations were obtained by PET glycolysis reaction, using ethylene glycol as a solvent, and zinc acetate as a catalyst. Three different PET packaging materials (bottles with different additives) were used to prepare powders with green, white and blue appearance. The PET-based powders were then used to visualize latent fingerprints deposited onto non-porous (glass) surface. The interactions between components of the prepared powders and potential presence of additives were investigated by Fourier-transform infrared spectroscopy (FT-IR) analyses, equipped with a diamond attenuated total reflectance (ATR) smart accessory. Optical microscopy revealed that the prepared powders consisted of round-shaped particles uniform in size, which enabled their easy adhesion to the sweat and lipid fingerprint residues. We compared the structure and properties of the prepared powders, and assessed the quality of visualization against the commercial BVDA Magnetic Silver powder, routinely used in forensic practice. The results demonstrated that powders prepared from depolymerized PET material could potentially supplement routinely used fingerprinting powders, particularly due to their low price, as they are derived from waste raw material.

DESIGN/METHODS/APPROACH

Materials

Three different PET packaging materials, i.e. green, white and blue bottles with different additives, were procured from local grocery stores in Belgrade, Serbia. Ethylene glycol and zinc acetate were procured from Centrohem (Serbia). Distilled water was used for washing/purification of final products. Except PET, all materials were used without further treatment.

Preparation of PET-Based Powders

The powders were prepared in accordance with the procedure described by Chan and Zinchenko (Chan & Zinchenko, 2022). The PET glycolysis reaction was performed using three different PET packaging materials: of green, white and blue appearance. Ethylene glycol was used as the solvent, and zinc acetate as the catalyst. The ratio of 1:3 (w/w) PET/ethylene glycol was used for preparation of all powders. Specifically, 1 g of green/white/blue PET chips, 3 mL of ethylene glycol, and 0.01 g of zinc acetate were added into a 250 mL round bottom flask equipped with a thermometer and a reflux condenser. The glycolysis reaction was conducted using a heat mantle at the temperature of 150 ± 5°C until all PET chips were dissolved (~ 4 h). The obtained solution was allowed to cool to room temperature, after which the precipitate was collected and resuspended in distilled water. The washed precipitate was then filtrated using a Büchner funnel, transferred to a Petri dish, and subsequently frozen at –19°C. The sample underwent lyophilized for approximately 24 h, at a pressure of 0.1 Pa, with a sample temperature of –49°C and chamber temperature of –68°C. The obtained clumps were ground into fine powders using pestle and mortar and were stored in a desiccator until further application.

CHARACTERIZATION OF PET-BASED POWDER FORMULATIONS

ATR-FTIR Analyses

The ATR-FTIR analyses of obtained PET-based powders were performed using Nicolet iS10 FTIR spectrometer with ATR smart accessory (Thermo Fisher Scientific, USA), in the range of 4000-400 cm^{-1}, at a resolution of 2 cm^{-1}, and at 25°C.

Optical Microscopy

The obtained powders were recorded with optical microscope *Leica FS C Comparison Macroscope*, equipped with the *Leica IM Matrox Meteor II Driver Software Module*. Powders were recorded in dry state, with and without backlighting. Prior to imaging under the microscope, prepared powders were spread over the glass microscopic slides.

Development of Latent Fingerprints

In order to examine the performances of the prepared PET-based powders, fingerprints were collected from one female and two male donors. Each donor used only their right-hand index finger to deposit sebaceous/oily fingerprints onto a glass surface (properly labelled glass microscopic slides). The finger-

prints were applied using a technical scale (force applied to accommodate 100–150 g, per fingerprint). The prints were then stored under laboratory (humid) conditions and developed at different time intervals: immediately after deposition (day 0), after 1 day, 7 days and 30 days. These intervals allowed the traces to dry and reduce the quantity of residues before developing the latent fingerprints. The prepared PET-based powders were applied using a BVDA Squirrel hair brush (BVDA, The Netherlands), and BVDA Magnetic Silver powder (control sample) using BVDA Magnetic brush (BVDA, The Netherlands) (Almog, Cantu, Champod, Kent, & Lennard, 2014). Subsequently, a magnifying glass and optical microscopy were used to determine the ability of binding of the prepared PET-based powders to fingerprint residues and to evaluate the quality of developed fingerprints on the glass surface.

FINDINGS

Obtained formulations were labelled as PET-1, PET-2, and PET-3, derived from depolymerized green, white, and blue PET bottles, respectively. These powders were used to visualize latent fingerprints deposited onto non-porous (glass) surface. Multiple fingerprints were developed on glass substrates to assess the quality and reproducibility of each powder. The formulation PET-2 yielded the best results in visualizing sebaceous fingerprints deposited onto the glass surface.

After preparation, the initial examination of PET-based powders was performed by visualizing latent fingerprints deposited onto glass surface. Figure 1 shows sebaceous fingerprints from one male donor, deposited onto glass surface, left for a few of minutes, and developed using formulations PET-1 (Figure 1, a)), PET-2 (Figure 1, b)), and PET-3 (Figure 1, c)). The prints were photographed with a magnifying glass (×5) under visible light, using a 50 MP camera (aperture $f/1.8$, pixel size 0.64 μm). Fingerprints were recorded with assistance of black background surface for appropriate contrast.

a) b) c)

Figure 1. *Sebaceous Fingerprints of One Male Donor Developed on Glass Surface, Using Prepared Formulations: a) PET-1, b) PET-2, and c) PET-3, Recorded with a Magnifying Glass (×5) Under Visible Light. Fingerprints Were Recorded with Assistance of Black Background Surface*

When observing fingerprints in Figure 1, it is evident that fine and smooth structure of glass surface enabled the development of complete fingerprint images. The results showed that all obtained formulations demonstrated satisfying detection and enhancement of latent fingerprints, by visualizing complete fingerprint images. The powders developed the prints with continuous papillary line flow, probably due to small particles, enabling good contrast and visualization of fingerprint level 1 and 2

features. On the other hand, slight retention of particles in the interpapillary space caused "overpowdering" of fingerprints, probably due to non-uniform distribution of powders' particles, but without significant impact on fingerprint quality and features.

ATR-FTIR Analyses

ATR-FTIR analyses were performed to determine the structure of the prepared PET-based powders, as well as to identify the presence of additives. The aim of analyses was also to investigate the structural differences and/or similarities between the initial PET packaging material and the corresponding powder, with a particular focus on the residues of components used in the glycolysis reaction. PET exhibits some specific bands, which are observed in the spectra of all PET packaging materials and the corresponding PET-based powders, as shown in Figures 2–4, a) and d). A strong peak around 1715 cm^{-1} corresponds to C=O stretching in ketones, while sharp and broad peaks around 1245 cm^{-1} and 1100 cm^{-1} are associated with C–O stretching of aromatic and aliphatic ethers, respectively (Chen, Hay, & Jenkins, 2012; Mecozzi & Nisini, 2019). Peaks around 870 cm^{-1} and 720 cm^{-1} are related to the bending of C–H aromatic and aliphatic bond, respectively (Ruvolo-Filho & Curti, 2008; Ioakeimidis, et al., 2016). Zinc acetate, used as a catalyst, also contains characteristic bands and peaks (Figures 2–4, b)). The broad band at 3057 cm^{-1} is associated with O–H stretching vibrations from intermolecular hydrogen bonding in acetate salt and/or water adsorption, while two weak bands at 2159 cm^{-1} and 1985 cm^{-1} can be related to C–H stretching vibrations of methyl groups (Schlur, Carton, & Pourroy, 2015; Phoohinkong, Foophow, & Pecharapa, 2017). Two bands at 1548 cm^{-1} and 1417 cm^{-1} are attributed to the antisymmetric and symmetric stretching vibration modes of carboxylate anion, respectively. A weak peak positioned at 1056 cm^{-1} can be related to the vibration of C–CH_3 (Zhang, Zhu, Zhang, & Xia, 2008; Wang, Li, Zhou, Zu, & Deng, 2011). The band at 688 cm^{-1} corresponds to the acetate anion scissoring, while band at 618 cm^{-1} is associated with acetate anion twisting mode (Duan, et al., 2008; Phoohinkong, Foophow, & Pecharapa, 2017). Additionally, in the spectra of ethylene glycol (Figures 2–4, c)) some specific peaks and bands can be observed. The strong and broad band at 3355 cm^{-1} is a result of the stretching vibrations of the O–H groups, while two strong bands at 2945 cm^{-1} and 2880 cm^{-1} are related to the antisymmetric and symmetric stretching of C–H groups, respectively (Lin, Ren, Tian, Zhou, & Sun, 2013; Çabuk, Yılmaz, & Yıldız, 2019). A peak at 1460 cm^{-1} corresponds to CH_2 bending, while the peak at 1400 cm^{-1} is associated with the bending of C–O–H (Haoue, Derdar, Belbachir, & Harrane, 2020). A weak band at 1205 cm^{-1} is associated with CH_2 wagging, while two very strong and sharp bands at 1085 cm^{-1} and 1042 cm^{-1} are attributed to C–O stretching vibration. Furthermore, a strong band at 882 cm^{-1} is related to CH_2 rocking, while weak band at 522 cm^{-1} results from C–C–O bending vibrations (Kononova, Rodnikova, Solonina, & Sirotkin, 2018; Çabuk, Yılmaz, & Yıldız, 2019).

Figure 2 shows spectra of PET packaging material of green appearance, pure zinc acetate, pure ethylene glycol, and formulation PET-1.

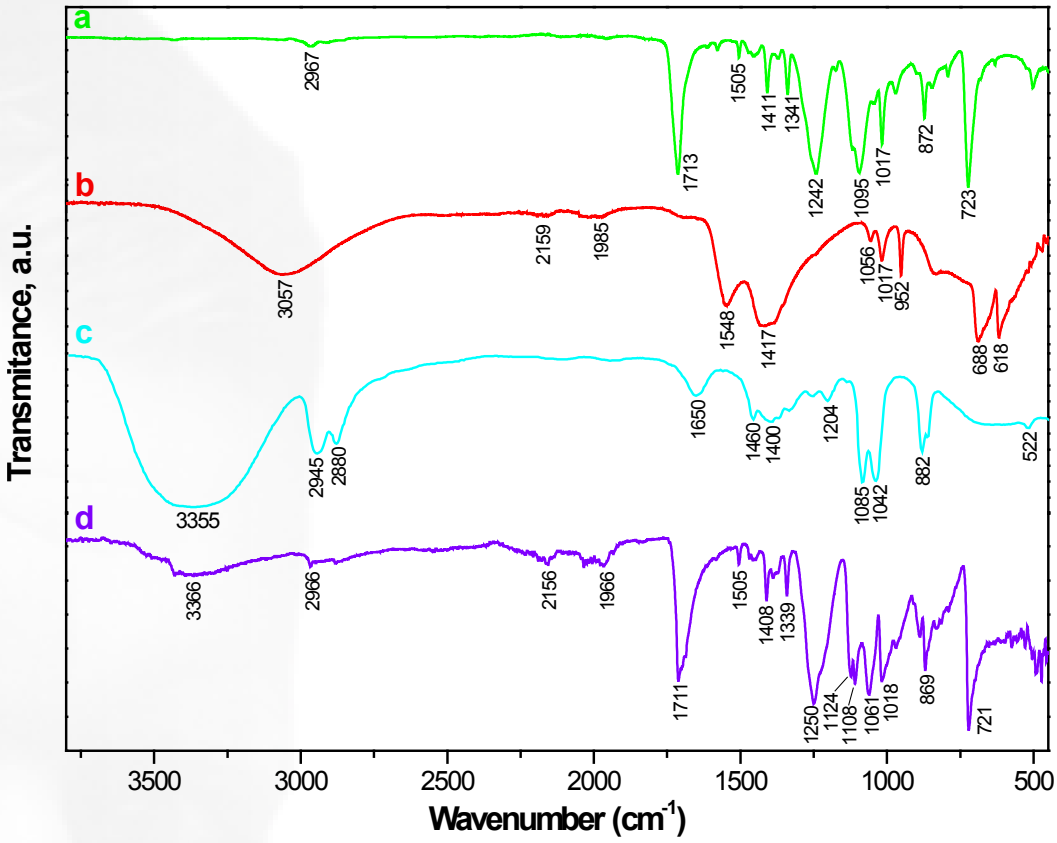

Figure 2. *ATR-FTIR Spectra of: a) PET Packaging Material of Green Appearance, b) Pure Zinc Acetate, c) Pure Ethylene Glycol, and D) Formulation PET-1*

In the spectrum of formulation PET-1 (Figure 2, d)), a broad and weak band appears at 3366 cm^{-1}, which was not observed in the spectrum of initial PET packaging material (Figure 2, a)). This band could be attributed to the presence of ethylene glycol and its hydroxyl groups. Additionally, two weak bands appear at 2156 cm^{-1} and 1966 cm^{-1} in spectrum of PET-1, which can be attributed to stretching vibrations of methyl groups from zinc acetate. Furthermore, two very weak peaks at 1124 cm^{-1} and 1108 cm^{-1} are present in the spectrum of PET-1 but are absent in spectrum of initial PET packaging material. These peaks could be associated with increased number of C–O stretching vibrations due to the presence of ethylene glycol.

Figure 3 shows the spectra of PET packaging material with white appearance, pure zinc acetate, pure ethylene glycol, and formulation PET-2, while Figure 4 shows the spectra of PET packaging material with blue appearance, pure zinc acetate, pure ethylene glycol, and formulation PET-3.

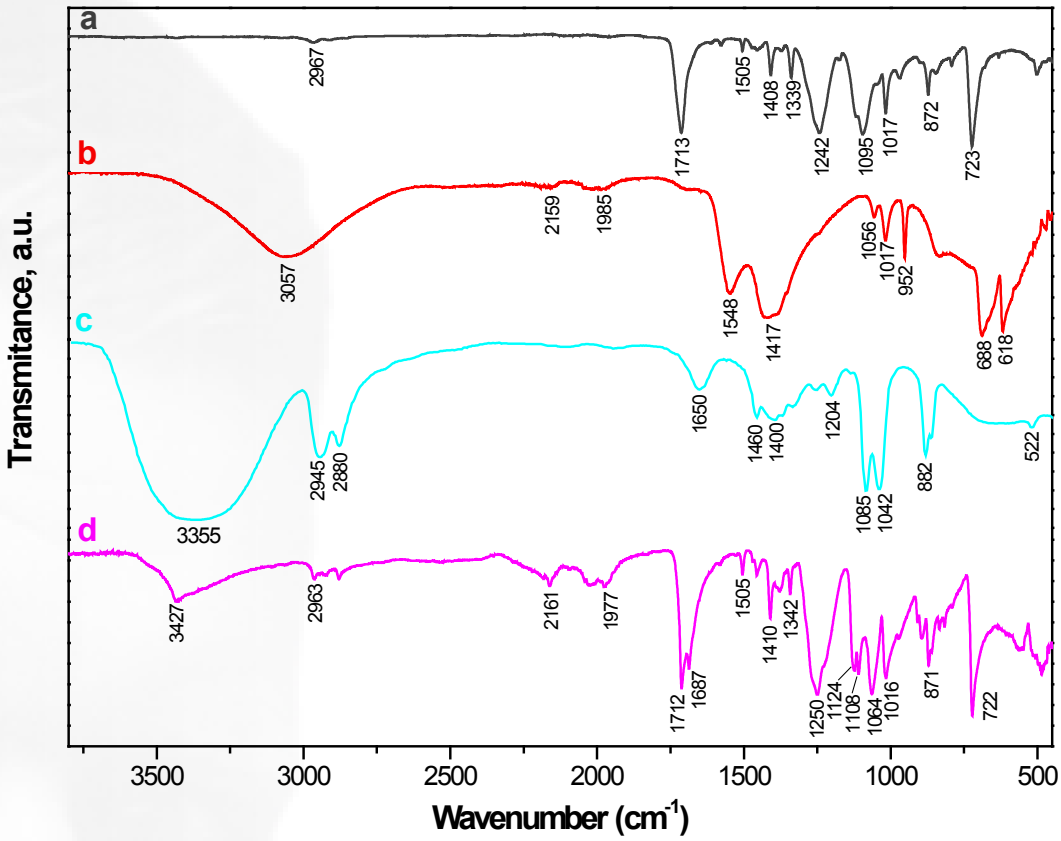

Figure 3. *ATR-FTIR Spectra of: A) Pet Packaging Material with White Appearance, B) Pure Zinc Acetate, C) Pure Ethylene Glycol, and D) Formulation PET-2*

In the spectra of formulations PET-2 (Figure 3, d)) and PET-3 (Figure 4, d)), similar patterns were observed to those in the spectrum of formulation PET-1 (Figure 2, d), including shifts and changes in the intensity of bands at 3427 cm^{-1}, 2161 cm^{-1}, 1977 cm^{-1}, 1124 cm^{-1}, and 1108 cm^{-1}. These similarities suggest that ethylene glycol and zinc acetate did not become covalently incorporated into polymer structure. Instead, it is more likely that they established electrostatic interaction with the polymer. Another possibility is that the clumps obtained after glycolysis reaction were not sufficiently rinsed, leading to residual contamination in the powders. Additionally, some unspecified bands in the spectra of all PET packaging materials and their corresponding PET-based powders (Figures 2–4, a) and d)), such as those around 1505 cm^{-1}, 1410 cm^{-1}, and 1340 cm^{-1}, could be attributed to additives present in the original polymer. These bands may reflect the presence of residual additives or impurities that were not fully removed during the glycolysis and purification processes.

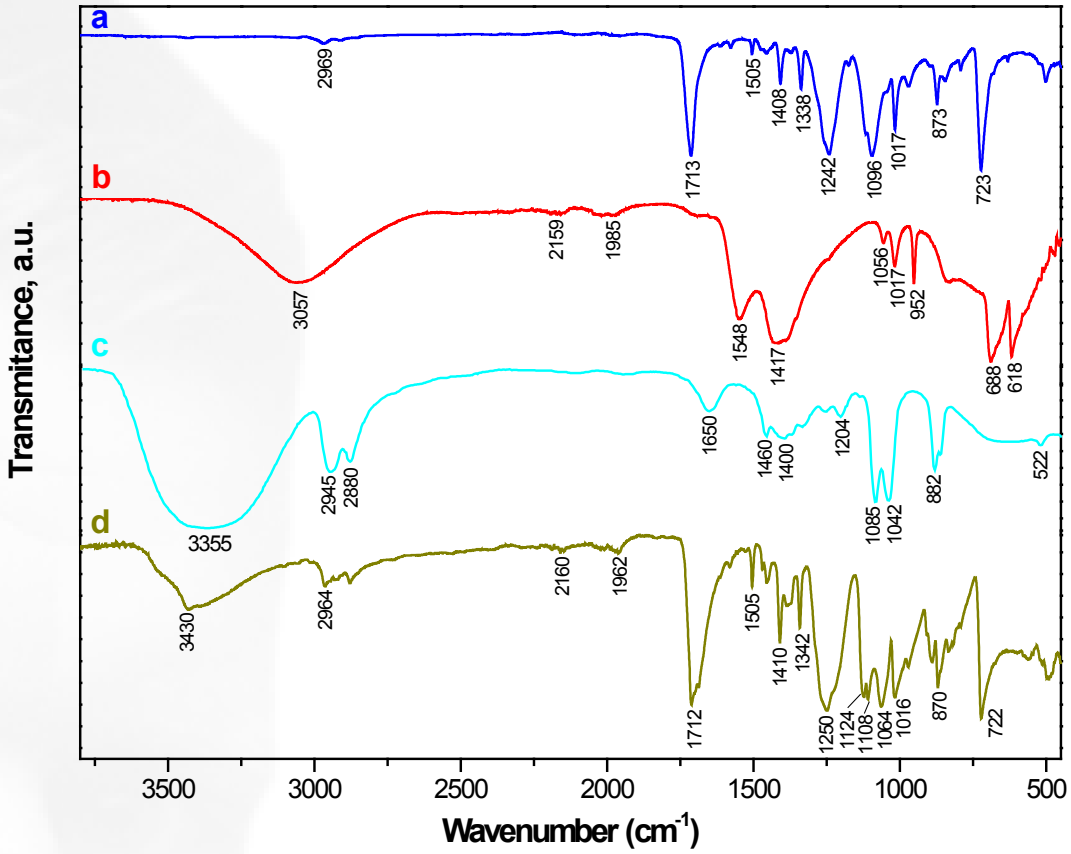

Figure 4. *ATR-FTIR Spectra of: A) Pet Packaging Material with Blue Appearance, B) Pure Zinc Acetate, C) Pure Ethylene Glycol, and D) Formulation PET-3*

OPTICAL MICROSCOPY

The Appearance of PET-Based Powders

Optical microscopy was used to estimate the size and uniformity of the prepared powder formulations. Figure 5 shows images of these powders, recorded with the *Leica FS C Comparison Macroscope*, equipped with the *Leica IM Matrox Meteor II Driver Software Module*. Images were taken at ×10 magnification using both dark-field (Figure 5, *I*) and bright-field (Figure 5, *II*) contrast techniques. Different backlighting modes were employed to enhance contrast, with the aim to observe and compare both amassed and thin layers of powders. Therefore, powder samples were spread over microscopic glass slides to approximate the size and compare the uniformity of their particles. Figure 5, *III*, represents the structure and appearance of the prepared powders as photographed under visible light with a magnifying glass (magnification ×5) using a 50 MP camera (aperture *f*/1.8, pixel size 0.64 μm).

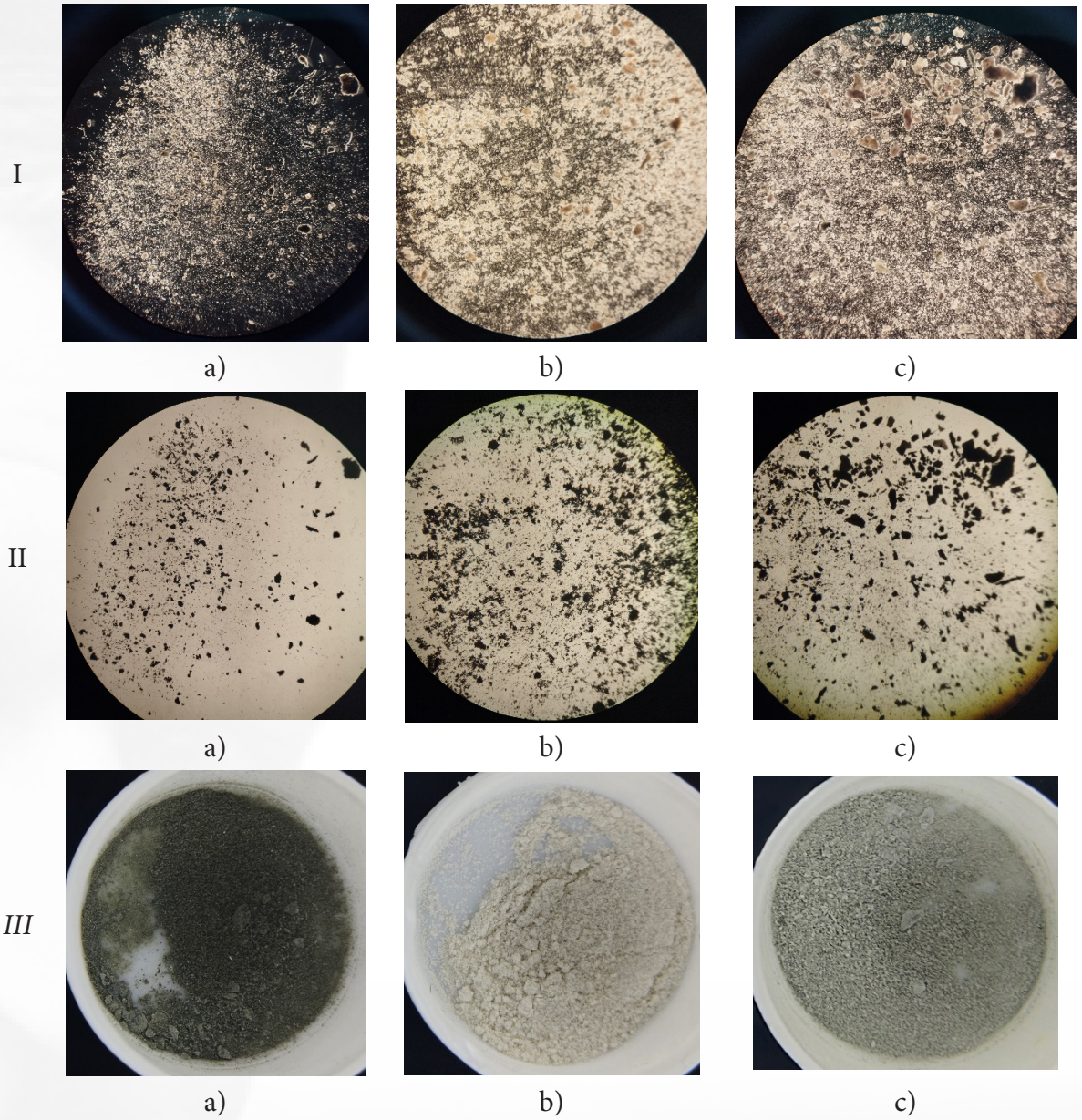

Figure 5. *Microscopic Images of Prepared Powders: a) PET-1, b) PET-2 and c) PET-3, Recorded with: I – Dark-Field Contrast Technique, and Ii – Bright-Field Contrast Technique, Using Optical Microscope with Magnification × 10; Iii – a Magnifying Glass (Magnification × 5) Under Visible Light, Using a 50 Mp Camera (Aperture ƒ/1.8, Pixel Size 0.64 μm)*

When comparing the prepared powders recorded under different backlighting modes, it is evident that all powders have small, round-shaped particles, but with noticeable deviation in particle size, likely due to imperfections of grinding or dusting process. As shown in Figure 5, b, formulation PET-2 had the most uniform particles evenly distributed over the glass substrate. In contrast, PET-1 and PET-3 had smaller particles that noticeably adhered to the glass surface (Figure 5, a and c)). Such properties of the powders' particles could potentially cause smudging or blurring of developed fingerprints, due to a larger dimeter of particles and increased adhesion to the substrate (Gürbüz, Özmen Monkul, İp-eksaç, Gürtekin Seden, & Erol, 2015). On the other hand, as shown in Figure 5, *III*, prepared powder formulations retained the appearance of waste raw materials used for their preparation, i.e. PET-1 with green, PET-2 with white, and PET-3 with blue appearance. This indicates that chemical structure of the original PET material was preserved throughout the process.

Testing of PET-Based Powders and Comparison with Commercial Powder

The prepared powder formulations were used to develop latent fingerprints on glass substrates to validate the results of the initial examination. Following the Guidelines proposed by the International Fingerprint Research Group (Almog, Cantu, Champod, Kent, & Lennard, 2014), one female and two male donors, using only a right-hand index finger, deposited sebaceous fingerprints onto microscopic glass slides using a technical scale (force applied to accommodate 100–150 g). The prints were then stored under laboratory (humid) conditions and developed at different time intervals: immediately after deposition (day 0), after 1 day, 7 days and 30 days. After the specified intervals, fingerprints were divided into two halves using a thin glass barrier, and then two different powders were used for their visualization – prepared PET-based powder formulations were applied with BVDA Squirrel hair brush to the left-half, and BVDA Magnetic Silver powder was applied with BVDA Magnetic brush to the right-half barrier side. The developed fingerprints were then recorded using an optical microscope *Leica FS C Comparison Macroscope*, equipped with the *Leica IM Matrox Meteor II Driver Software Module*, with dark-field (Figure 6, *I*) and bright-field contrast technique (Figure 6, *II*), at ×10 magnification. Different backlighting modes were used to achieve appropriate contrast and observe both level I and II fingerprint features. For practical reasons, only the fingerprints developed immediately (day 0) from one female donor are shown in the Manuscript, while detailed results are available in the Supplementary Material.

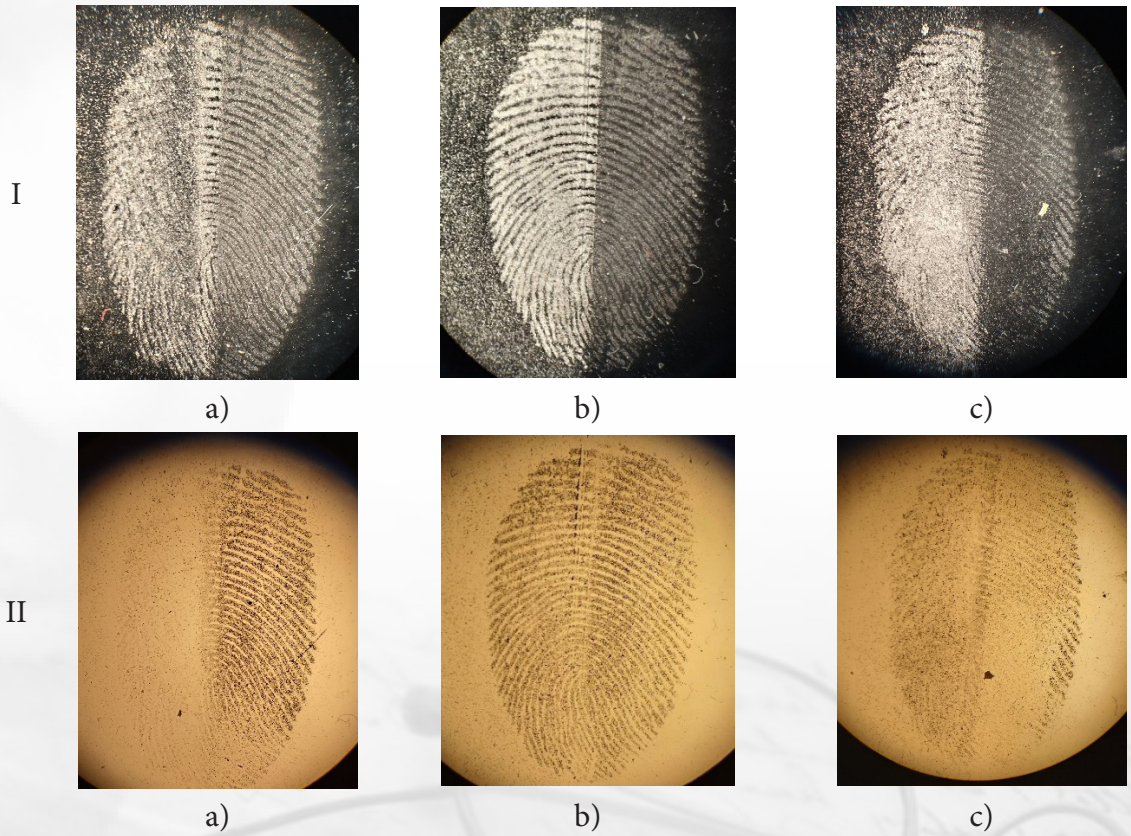

Figure 6. *Sebaceous Fingerprints Deposited onto Glass Substrate, Left for a Few Minutes, and Developed with: a) PET-1, b) PET-2, and c) PET-3 on the Left-Half Side of the Images, and a)–c) BVDA Magnetic Silver Powder on the Right-Half Side of the Images. Fingerprints Were Recorded with Optical Microscope (Magnification × 10), Using: I – Dark-Field Contrast Technique, and Ii – Bright-Field Contrast Technique*

The results obtained using optical microscopy confirmed the initial observations on glass substrate (Figure 1), and highlighted the superior performance of formulation PET-2 in visualizing latent fingerprints (Figure 6, b), left-half side of the fingerprint images). It was determined that formulation PET-2 enabled the clear development of basic fingerprint patterns and papillary lines, including minutiae points as well (i.e. level I and II features of fingerprints). Such results were noticeable when comparing PET-2 with formulations PET-1 and PET-3, and especially with commercial powder. Both, PET-2 and BVDA Magnetic Silver powder developed high-quality and complete fingerprint images, with all level I and II features. On the other hand, formulations PET-1 and PET-3 resulted in "over-powdering" of the interpapillary space and blurring of the fingerprints (Figure 6, a) and c), left-half side of the fingerprint images), which disrupted the flow of papillary lines. The issue is likely due to the larger particle size and their non-uniform distribution (Gürbüz, Özmen Monkul, İpeksaç, Gürtekin Seden, & Erol, 2015; Vučković, Glođović, Radovanović, Janaćković, & Milašinović, 2020).

Further analysis, Figures S2–S5 presented in Supplementary Material, assessed the impact of moisture on fingerprint residues and the quality of visualization over time. The results indicated that moisture and the flat glass surface probably altered the structure and quantity of fingerprint residues (Barnett & Berger, 1976; Jones, Downham, & Sears, 2010), leading to a decrease in fingerprint quality as storage time increase. Additionally, variations in developed fingerprint quality among donors were also observed, likely to uneven pressure applied to the glass surface. This resulted in different quantity of sweat and lipid residues, even though donors deposited traces in accordance with the guidelines. Therefore, the performance of the PET-based powders should be enhanced through modifications of the depolymerization process or the addition of specific additives (e.g. fluorescent compounds), which could improve their effectiveness and expand their applicability across different substrates and under various conditions (thoroughly presented in Supplementary Material).

ORIGINALITY/VALUE

In this paper, PET-based powders were prepared by depolymerizing PET packaging material (bottles) through a glycolysis reaction, using ethylene glycol as a solvent and zinc acetate as a catalyst. The prepared powders were used to develop latent fingerprints on glass substrates, and their efficiency was compared with the commercially available BVDA Magnetic Silver powder. This approach presents a potential solution for managing PET waste, a significant environmental pollutant. ATR-FTIR analyses confirmed the structure of prepared PET-based powders, and identified the presence of additives and components from the glycolysis reaction. The results of initial examination of prepared powder formulations demonstrated satisfying visualization of sebaceous fingerprints deposited onto the glass surface, for all prepared powders. These results were further investigated by optical microscopy, suggesting that formulation PET-2 contained the most uniform particles, which provided the best results in developing latent fingerprints. Additionally, formulation PET-2 demonstrated performances comparable to the commercial BVDA Magnetic Silver powder.

However, results obtained from fingerprints stored over various time indicated that humid environment (moisture), along with a flat glass surface, contributed to changes in the structure and reduction of fingerprint residues (Barnett & Berger, 1976; Jones, Downham, & Sears, 2010). This effect led to a decrease in print quality over time. Variations in developed fingerprints quality among donors were also observed. Such result may be attributed to uneven pressure applied on the glass surface, resulting in more or less sweat and lipid residues, despite adherence to guidelines. Besides substrate properties (type, colour, structure, etc.), environmental conditions and touch pressure, the quality of fingerprints is also affected by sex, age, ethnicity, metabolism of the donors and their daily routine (nutrition,

smoking, medication, cosmetics, etc.) (Archer, Charles, Elliott, & Jickells, 2005; Trapecar & Balazic, 2007; Croxton, Baron, Butler, Kent, & Sears, 2010; Hazarika & Russell, 2012; Cadd, Islam, Manson, & Bleay, 2015). Nevertheless, the consistent method of depositing and visualizing latent fingerprints ensured that the results were valid and reliable. On the other hand, formulation PET-2 showed better performances than formulations PET-1 and PET-3, by maintaining continuous papillary line flow and developing complete fingerprint images, with level I and II features, without retaining in the interpapillary space.

The prepared PET-based powders are cost-effective, easy to apply, and durable during testing, offering a sustainable solution through recycling PET packaging material. By integrating circular economy principles, this approach significantly contributes to environmental protection by revalorizing PET waste, thereby reducing landfill accumulation and resource depletion. Based on all presented results, PET-based powders could complement existing fingerprinting methods used on non-porous substrates in forensic practice. Further research should focus on expanding the application of these powders to unconventional surfaces (e.g., multi-coloured substrates, firearms, skin, etc.), to enhance selectivity, sensitivity and overall quality of developed fingerprints.

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SUPPLEMENTARY MATERIAL

Examination of the functionality and performances of prepared PET-based powders in visualizing latent fingerprints was conducted by following the guidelines proposed by the International Fingerprint Research Group. Experimental procedures were set to carry out Phase 1 (*Pilot Studies*) of recommended guidelines, which briefly includes:

- 3–5 donors;
- 1–3 clean, low interference substrates;
- Donors briefed on how to deposit fingerprints, with assistance provided as required;
- Natural prints preferred, with groomed prints avoided where possible;
- Fingerprints should normally be allowed to "age" for a minimum of 24 hours prior to development and the actual age prior to treatment should be recorded and reported;
- The study should include at least a preliminary performance comparison against relevant routine detection methods (for example, the treatment of split prints can be used to provide an initial indication of relative performance);
- Qualitative or holistic scales should be employed for assessing the quality of fingerprint development; and

Reports must clearly indicate limitations and conclusions must be conservative (Almog, Cantu, Champod, Kent, & Lennard, 2014).

Therefore, one female and two male donors, using only a right-hand index finger, deposited their sebaceous fingerprints onto non-porous (glass) surface using technical scale (force accommodated to 100–150 g, per fingerprint), in order to simulate manipulation with objects under real conditions, and fingerprints were left under humid (laboratory) conditions for specified time intervals – a few minutes (day 0; immediate development of latent fingerprints), 1 day, 7 days and 30 days. After the mentioned periods, three prepared powder formulations, as well as BVDA Magnetic silver powder (control powder) were used to evaluate performances in developing latent fingerprints. Afterwards, all fingerprints were recorded with *Leica FS C Comparison Macroscope*, equipped with the *Leica IM1000 Matrox Meteor II Driver Software Module*, using magnification ×10 and two contrast techniques (backlighting).

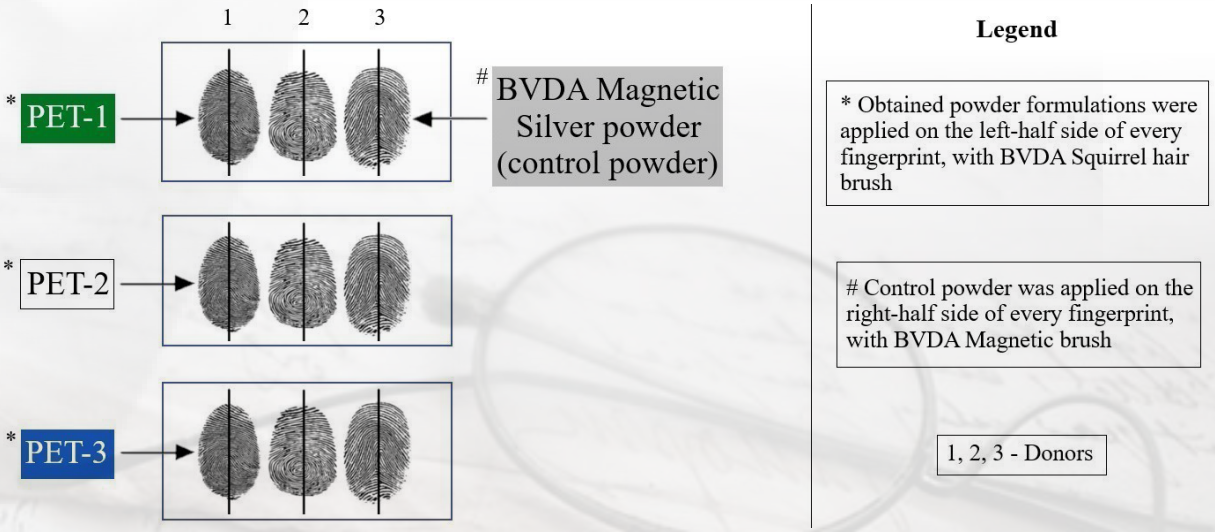
Initial testing of obtained powders was performed prior to thorough investigation by optical microscopy, with the aim of evaluating the properties of the prepared powders. The results of initial examination are shown in Figure S1, which represent sebaceous fingerprints of one male donor, developed immediately after deposition on the glass surface, using the prepared powder formulations, PET-1 (Figure S1, a)), PET-2 (Figure S1, b)), and PET-3 (Figure S1, c)). The prints were then photographed with a magnifying glass (×5) under visible light, using a 50 MP camera (aperture $f/1.8$, pixel size 0.64 μm). The fingerprints were photographed with assistance of black background surface for adequate contrast.

a) b) c)

Figure S1. *Sebaceous Fingerprints of One Male Donor Developed on Glass Surface, Using Prepared Formulations: a) PET-1, b) PET-2, and c) PET-3, Recorded with a Magnifying Glass (×5) Under Visible Light. The Fingerprints Were Recorded with Assistance of Black Background Surface.*

Based on results in Figure S1, it is evident that the sebaceous fingerprints were successfully developed on the glass surface with all applied powder formulations. The results showed that all obtained formulations demonstrated satisfying development of latent fingerprints, by visualizing complete fingerprint images. The powders developed the prints with continuous papillary line flow, probably due to small particles, enabling good contrast and visualization of fingerprint level 1 and 2 features. On the other hand, slight retention of particles in the interpapillary space caused "overpowdering" of the fingerprints, probably due to non-uniform distribution of powders' particles, but without significant impact on fingerprint quality and features.

Subsequently, in different time intervals, the fingerprints deposited onto the glass surface were separated into halves with a thin glass barrier, and specified powders were applied, as represented in the scheme bellow:



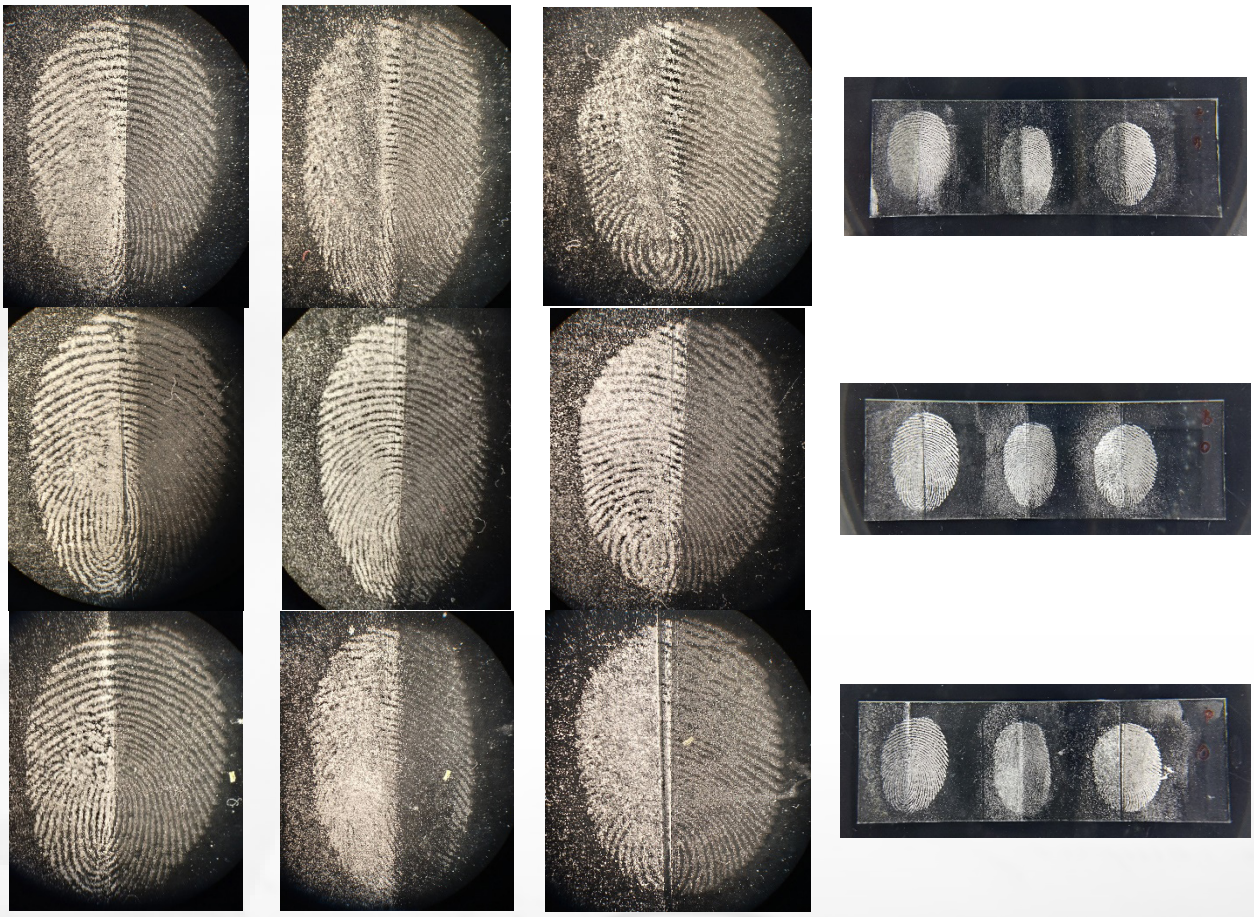
Afterwards, the fingerprints developed after different time intervals, were recorded with the mentioned optical microscope, using magnification ×10, and dark-field and bright-field contrast techniques.

Immediate Development of Latent Fingerprints – April 15, 2024

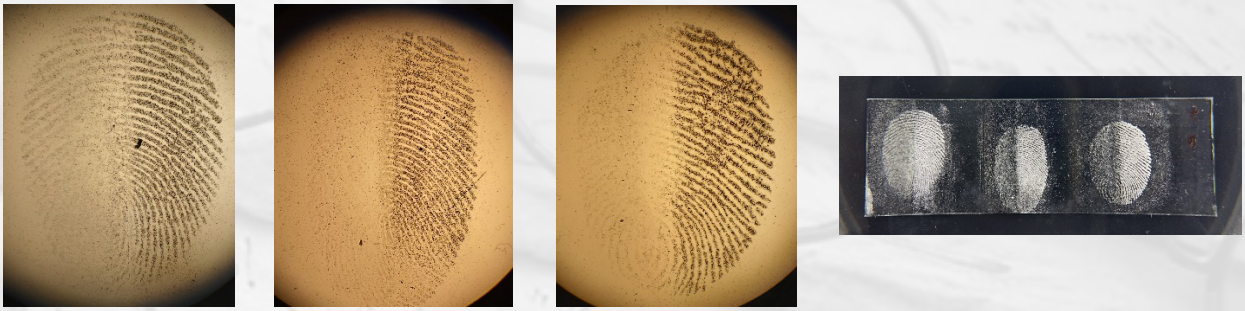
The sebaceous fingerprints of one female and two male donors were developed immediately after deposition (day 0), with formulations PET-1, PET-2, PET-3 and BVDA Magnetic silver powder (control powder). Figure S2 represents fingerprints developed on the glass surface, recorded using dark-field (Figure S2, *I*) and bright-field (Figure S2, *II*) contrast techniques. In the far-right column, all developed fingerprints photographed with a magnifying glass (magnification ×5) using a 50 MP camera (aperture *f*/1.8, pixel size 0.64 μm) were shown.

The fingerprints were developed on the glass surface and recorded with the optical microscope, using:

I – Dark-field contrast technique



II – Bright-field contrast technique



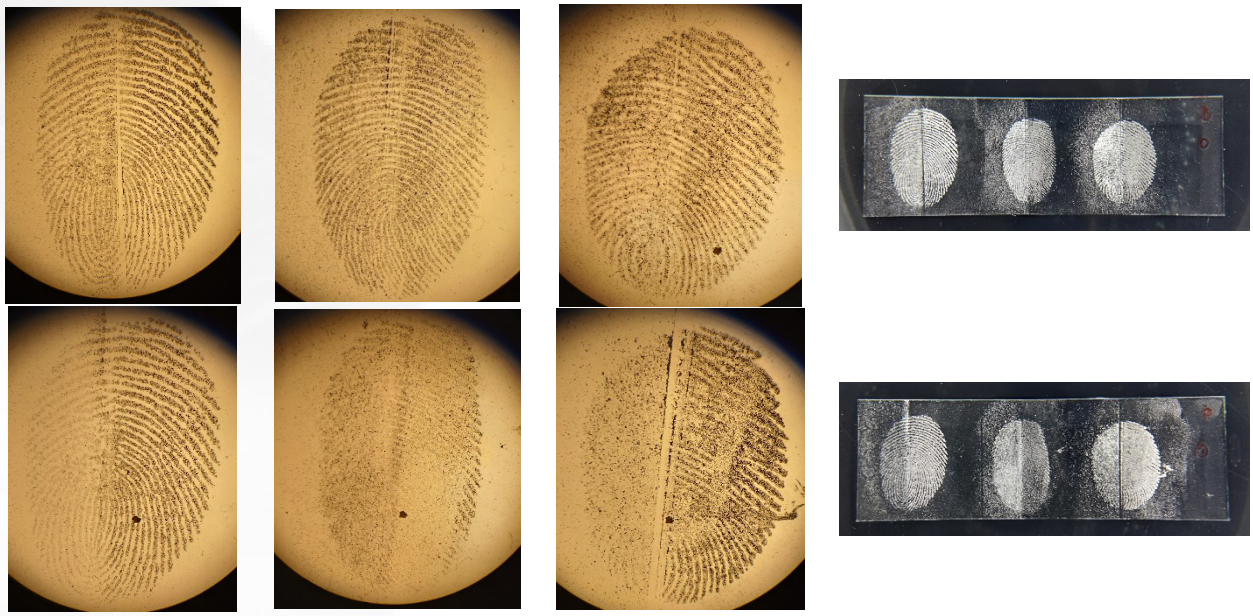


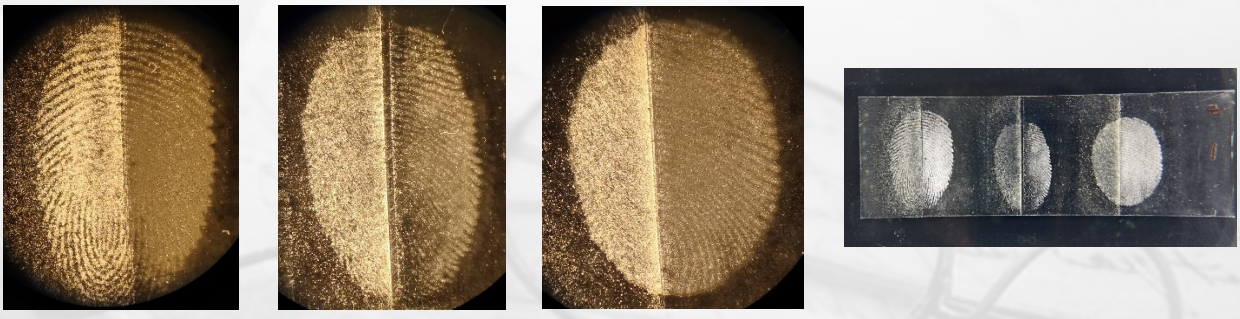
Figure S2. *The Sebaceous Fingerprints Developed Immediately After Deposition (Day 0) on the Glass Surface with the Aforementioned Powders, Recorded Using: I – Dark-Field, and II – Bright-Field Contrast Techniques. In the Far-Right Column Are Shown All Developed Fingerprints Photographed Under Visible Light with a Magnifying Glass (Magnification ×5).*

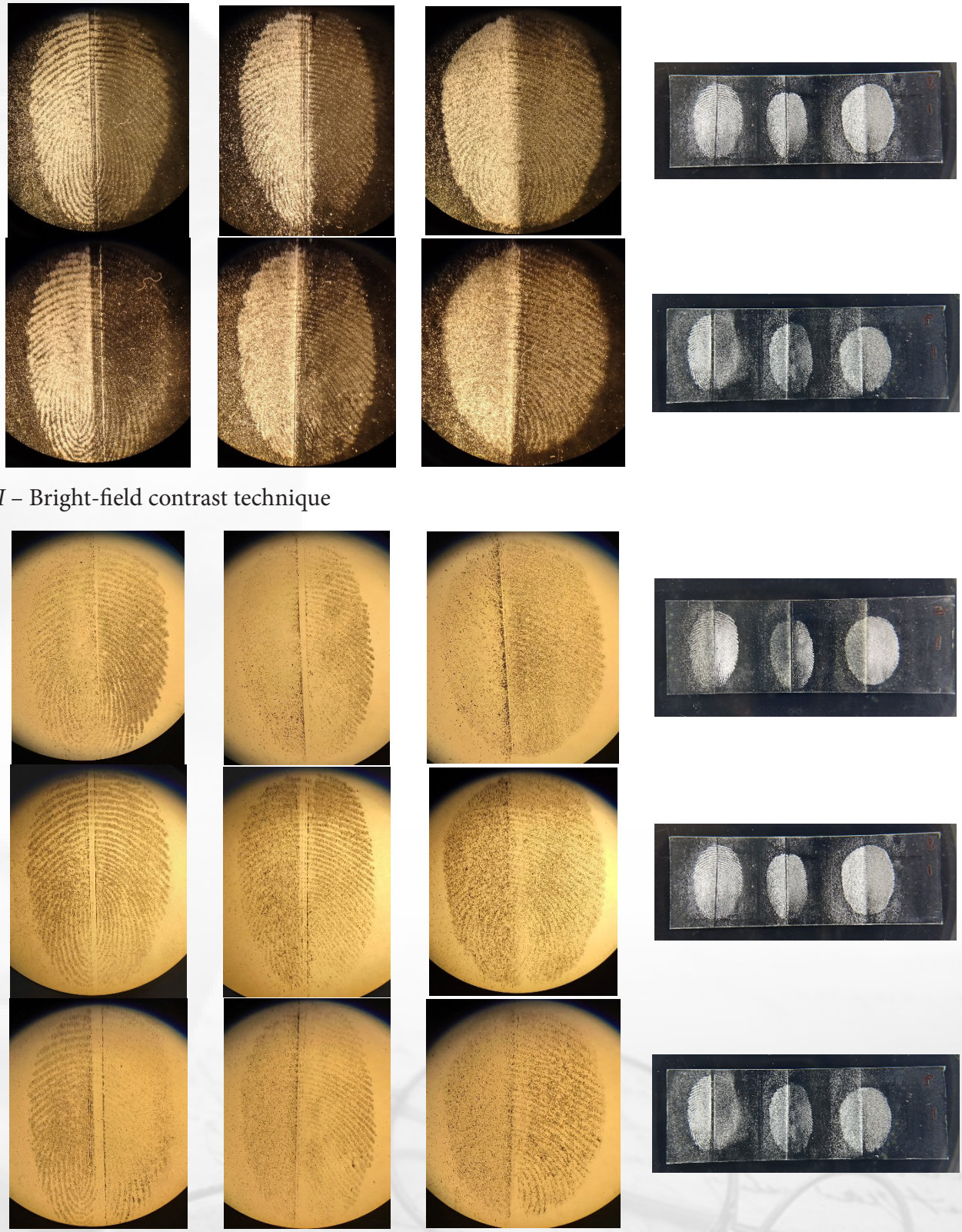
Development of Latent Fingerprints 1 Day After Deposition – April 16, 2024

The sebaceous fingerprints of one female and two male donors were developed 1 day after deposition, with formulations PET-1, PET-2, PET-3 and BVDA Magnetic silver powder (control powder). Figure S3 represents the fingerprints developed on the glass surface, recorded using dark-field (Figure S3, *I*) and bright-field (Figure S3, *II*) contrast techniques. In the far-right column, all developed fingerprints photographed with a magnifying glass (magnification ×5) using a 50 MP camera (aperture *ƒ*/1.8, pixel size 0.64 μm) were shown.

The fingerprints were developed on the glass surface and recorded with the optical microscope, using:

I – Dark-field contrast technique





II – Bright-field contrast technique

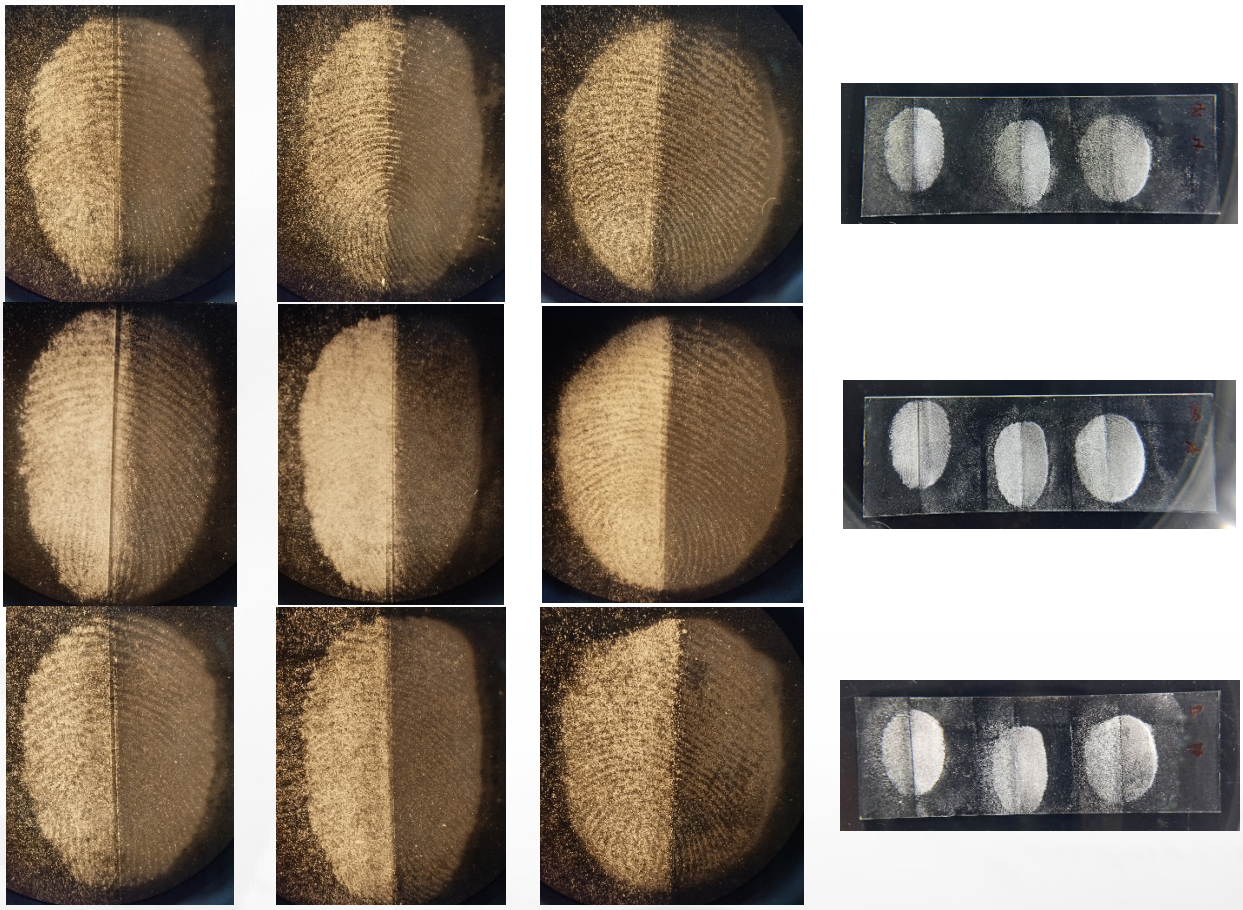
Figure S3. *The Sebaceous Fingerprints Developed 1 Day After Deposition on the Glass Surface with the Aforementioned Powders, Recorded Using: I – Dark-Field, and II – Bright-Field Contrast Techniques. In the Far-Right Column Are Shown All Developed Fingerprints Photographed Under Visible Light with a Magnifying Glass (Magnification ×5).*

Development of Latent Fingerprints 7 Days After Deposition – April 22, 2024

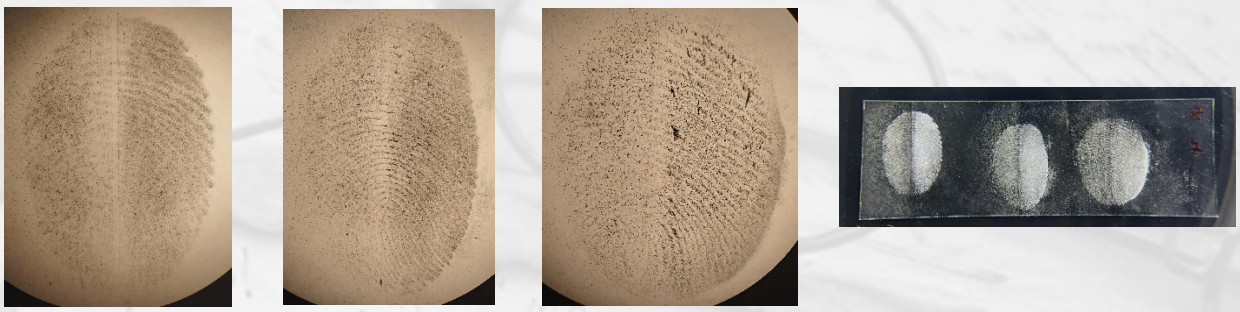
The sebaceous fingerprints of one female and two male donors were developed 7 days after deposition, with formulations PET-1, PET-2, PET-3 and BVDA Magnetic silver powder (control powder). Figure S4 represents the fingerprints developed on the glass surface, recorded using dark-field (Figure S4, *I*) and bright-field (Figure S4, *II*) contrast techniques. In the far-right column, all developed fingerprints photographed with a magnifying glass (magnification ×5) using a 50 MP camera (aperture *ƒ*/1.8, pixel size 0.64 μm) were shown.

The fingerprints were developed on the glass surface and recorded with the optical microscope, using:

I – Dark-field contrast technique



II – Bright-field contrast technique



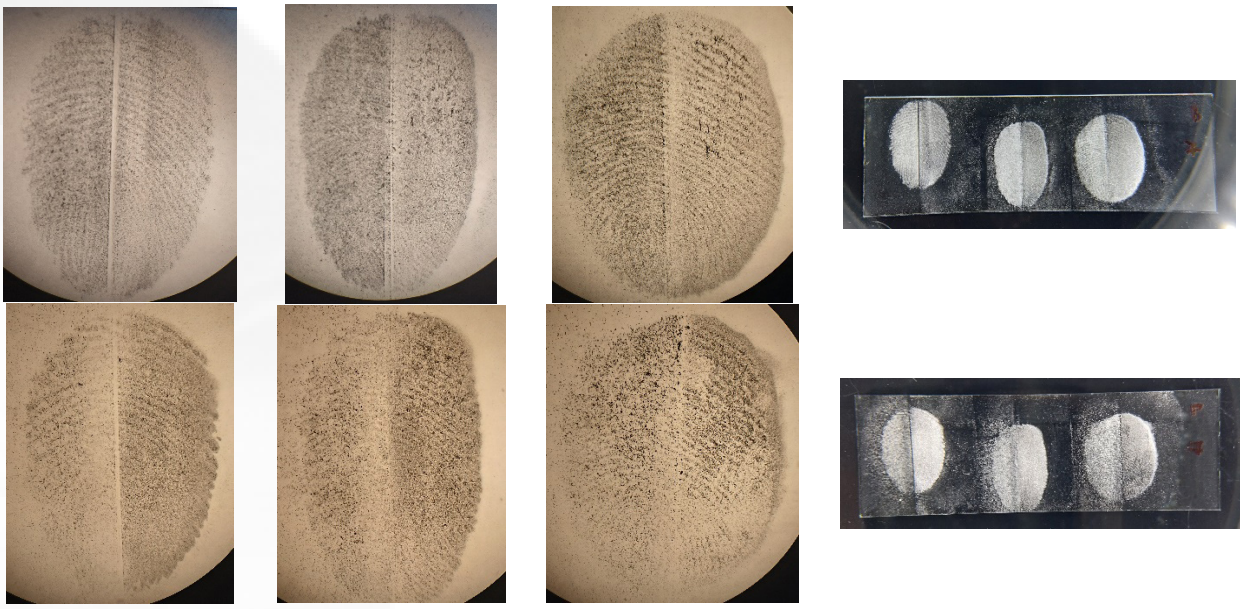


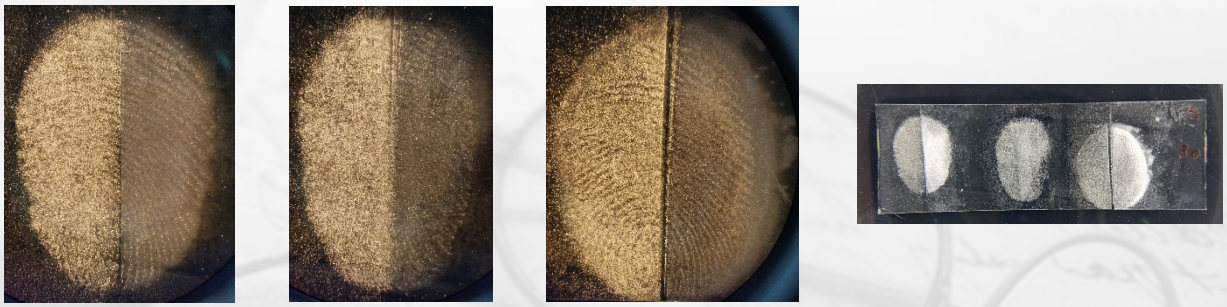
Figure S4. *The Sebaceous Fingerprints Developed 7 Days After Deposition on the Glass Surface with the Aforementioned Powders, Recorded Using: I – Dark-Field, and II – Bright-Field Contrast Techniques. In the Far-Right Column Are Shown All Developed Fingerprints Photographed Under Visible Light with a Magnifying Glass (Magnification ×5).*

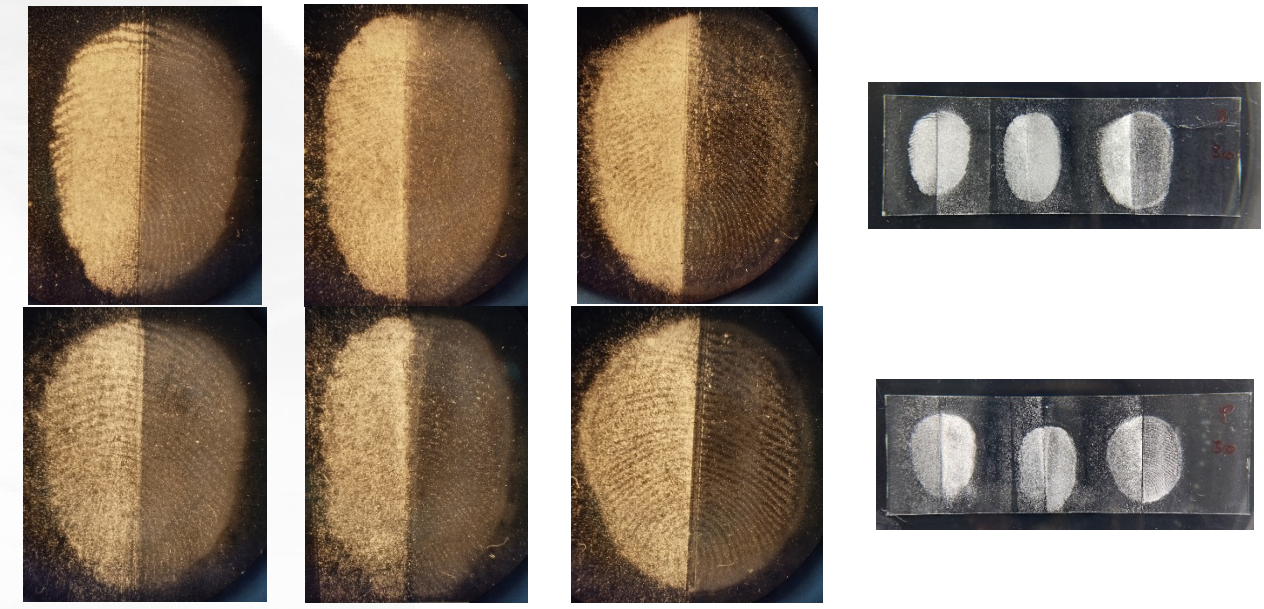
Development of Latent Fingerprints 30 Days After Deposition – May 15, 2024

The sebaceous fingerprints of one female and two male donors were developed 30 days after deposition, with formulations PET-1, PET-2, PET-3 and BVDA Magnetic silver powder (control powder). Figure S5 represents the fingerprints developed on the glass surface, recorded using dark-field (Figure S5, *I*) and bright-field (Figure S5, *II*) contrast techniques. In the far-right column, all developed fingerprints photographed with a magnifying glass (magnification ×5) using a 50 MP camera (aperture $f/1.8$, pixel size 0.64 μm) were shown.

The fingerprints were developed on the glass surface and recorded with the optical microscope, using:

I – Dark-field contrast technique





II – Bright-field contrast technique

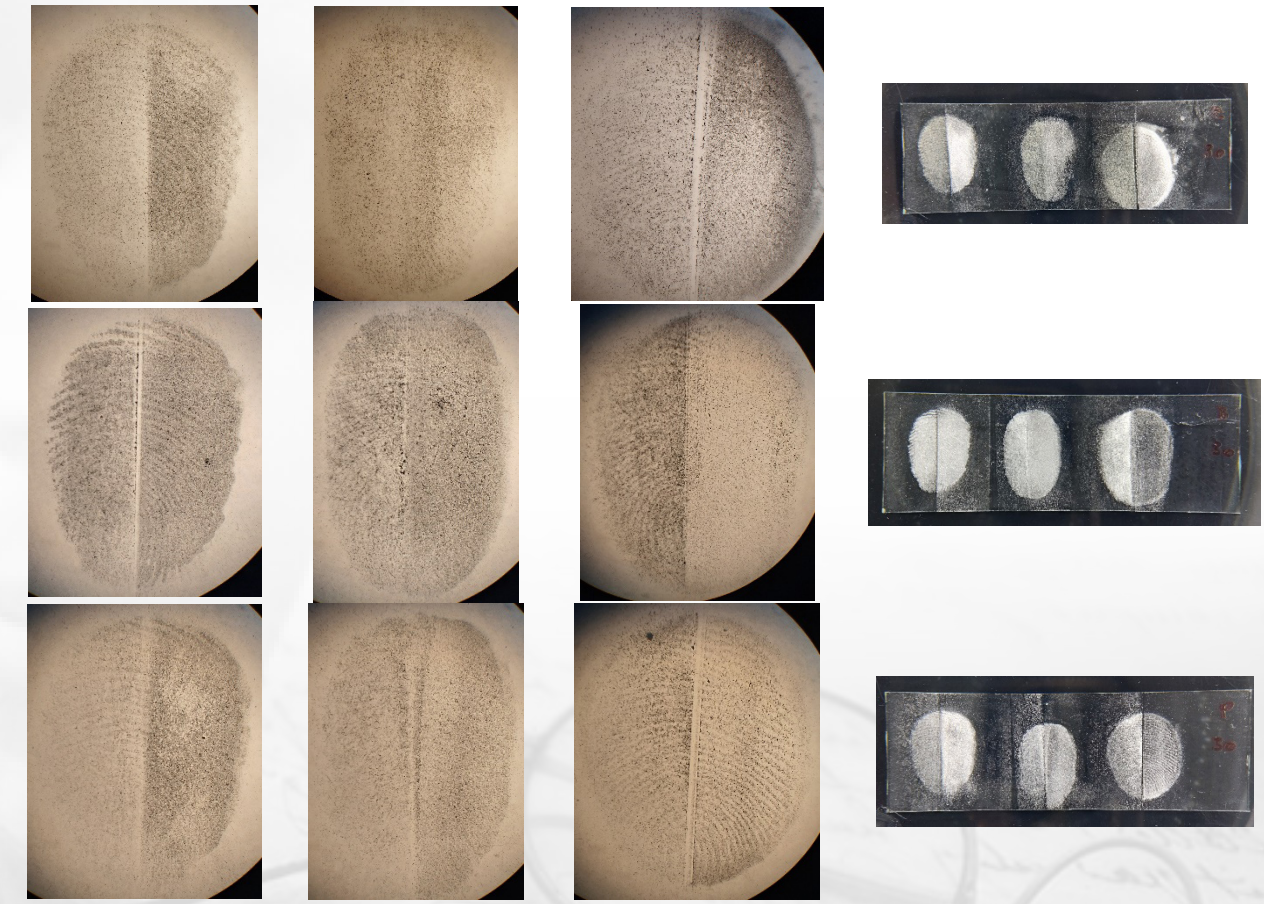


Figure S5. *The Sebaceous Fingerprints Developed 30 Days After Deposition on the Glass Surface with the Aforementioned Powders, Recorded Using: I – Dark-Field, and II – Bright-Field Contrast Techniques. In the Far-Right Column Are Shown All Developed Fingerprints Photographed Under Visible Light with a Magnifying Glass (Magnification ×5).*

CONCLUSIONS

The results of initial examination showed that all obtained formulations demonstrated satisfying development of latent fingerprints, by visualizing complete fingerprint images. However, when comparing visualization of the latent fingerprints kept in different time intervals, it is evident that the quality of prints kept for a shorter period of time (Figures S2 and S3) was far better than those stored for longer periods (Figures S4 and S5). Humid environment and a flat glass surface probably contributed to the change in the structure and reduction of sweat and lipid fingerprint residues, respectively (Barnett & Berger, 1976; Jones, Downham, & Sears, Effect of substrate surface topography on forensic development of latent fingerprints with iron oxide powder suspension, 2010). Therefore, the powders' particles had a smaller quantity of residues available for interaction (demonstrating unselective binding to prints), with increased sticking to the substrate due to adhesion. Variations in quality of visualized fingerprints between donors could also be observed. Such results may be related to uneven pressure on the glass surface, resulting in more or less sweat and lipid residues, even though donors deposited traces in accordance with the guidelines. Besides substrate properties (type, colour, structure, etc.), environmental conditions and touch pressure, the fingerprint quality is also affected by sex, age, ethnicity, metabolism of the donors and their daily routine (nutrition, smoking, medication, cosmetics, etc.) (Archer N. E., Charles, Elliott, & Jickells, 2005; Trapecar & Balazic, 2007; Croxton R. S., Baron, Butler, Kent, & Sears, 2010; Hazarika & Russell, 2012; Cadd S. , Islam, Manson, & Bleay, 2015). Nevertheless, the consistent method of depositing and visualizing the latent fingerprints ensured that the results were valid and reliable.

Additionally, when comparing the prepared powder formulations, the best results were obtained with PET-2, due to selective binding and visualization of papillary lines with their continuous flow, without retaining in the interpapillary space. Formulations PET-2 successfully developed level 1 and 2 features, which could be related to small particles of powder, more uniform in size, when compared to PET-1 and PET-3 (confirmed by optical microscopy). Smaller particles better adhere and bind to sweat and lipid fingerprint residues (Gürbüz, Özmen Monkul, İpeksaç, Gürtekin Seden, & Erol, 2015), and these results are particularly visible in Figures S2 and S3. Moreover, when compared to BVDA Magnetic Silver powder, formulation PET-2 showed comparable and equally good results of visualization. It is important to emphasize that commercial powder also developed lower-quality fingerprints previously kept for 7 days and 30 days when compared to prints kept for a few minutes and 1 day, as well as prepared formulations. Therefore, the presented results showed very satisfying visualization of the sebaceous fingerprints on the glass surface, with cheap PET-based powders obtained through recycling of waste raw material.

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