DEVELOPMENT OF LATENT FINGERMARKS ON DIFFERENT SUBSTRATES USING POLYANILINE-BASED POWDER OBTAINED BY SIMPLE PRECIPITATING METHOD

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PURPOSE

Forensic trace analysis implies the utilization of methods and techniques with the aim to identify and compare specific types of trace materials that could be transferred during the commission of a crime, and afterward the identification of a perpetrator and/or victim (Mozayani & Noziglia, 2006; Sonne, 2006). These trace materials include human and/or animal hair, textile fibers and fabric, rope, soil, glass, building materials and biological materials (Champod, Lennard, Margot, & Stoilovic, 2004; Sonne, 2006). However, fingermarks or fingerprints, as the common traces, represent about 10% of all material traces that could be recovered from a crime scene (Durose, Burch, Walsh, & Tiry, 2016), being one of the main tools used for identification of individuals in modern forensic science, due to their uniqueness, immutability, ease of classification and transferability (Mozayani & Noziglia, 2006). Fingermarks often remain as random marks, when the fingertip comes in contact with the surface of an object. The papillary line traces are transferred by the sweat (eccrine) glands, which secrete sweat and other components through the sweat pores. Additionally, the other compounds, such as blood, oil, dye, etc. can often be found and transferred from finger surface to the substrate along with the fingerprint (Datta, Lee, Ramotowski, & Gaensslen, 2001; Champod, Lennard, Margot, & Stoilovic, 2004; Lennard, 2007). Therefore, fingermarks

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could be divided in two main groups – the visible and latent (invisible) fingermarks. Visible fingermarks are easier for manipulation (e.g. photographing and lifting) when compared to the latent marks, requiring the visualization prior to processing and, for that purpose, different chemical, physical and optical methods are employed (Mozayani & Noziglia, 2006; Bumbrah, Sharma, & Jasuja, 2016; Milašinović & Koturević, 2016).

However, due to the aggressiveness and limitations of certain physical and chemical methods (Champod, Lennard, Margot, & Stoilovic, 2004), researchers are constantly attempting to develop new investigation approaches in forensic analysis of (latent) fingermarks. Polymeric materials are broadly used in different fields, such as vehicle and air industry, nanotechnology, medicine and pharmacy (Milašinović, Kalagasidis Krušić, Knežević-Jugović, & Filipović, 2010; Milašinović, et al., 2016), but scientific public is almost unaware and barely recognizes the potential of these materials in forensic applications (Lee, et al., 2014; Milašinović, 2016; Sen, Mohite, & Kayande, 2019). The goal of our previous researches was the utilization of biopolymeric materials (chitosan and dextran) in visualizing latent fingermarks, deposited on different (common) substrates and kept in different storage conditions. These so far promising achievements drove the us to the idea to extend our research to the utilization of synthetic polymeric materials (Vučković, Dimitrijević, & Milašinović, 2020; Vučković, Glođović, Radovanović, Janaćković, & Milašinović, 2020) that possess somewhat specific properties allowing the application of the prepared systems to various (illusive) surfaces. Polyaniline (PANI), one of the earliest known synthetic polymers, refers to a large class of conducting polymers. Synthetized from the cheap aniline, PANI can be found in one of three oxidation states: leucoemeraldine (white/clear & colorless), emeraldine (green or blue) and pernigraniline (blue/violet) (Heeger, 2001; Ćirić-Marjanović, 2010). On the other hand, unique properties (due to its inherent electrical activity), the ease of synthesis and low cost make PANI attractive to various industries and it is currently widely exploited in electronics (electrochromic glasses, solar cells, sensors, etc.), medicine (neural interfaces, scaffolds, delivery systems, etc.) and in anticorrosion materials (Ćirić-Marjanović, 2010; Beygisangchin, Abdul Rashid, Shafie, Sadrolhosseini, & Lim, 2021). However, there are not many studies regarding PANI application in forensic science (forensic trace analysis) (Beresford & Hillman, 2010; Beresford, Brown, Hillman, & Bond, 2012; Yuan, Li, Wang, Cao, & Lin, 2021).

In this paper, the polyaniline-based polymer powder, obtained by simple precipitating method, was synthetized and characterized with the aim to develop latent fingermarks deposited onto different surfaces, often found at the crime scene, i.e. plywood, glass and paper. The results demonstrated the potential of prepared PANI-based powder in developing latent fingermarks, as well as the ability to complement or replace some of the routinely applied physical methods.

DESIGN/METHODS/APPROACH

Materials

Aniline was purchased from Lach-Ner (Czech Republic), itaconic acid (IA) from Sigma-Aldrich (USA) and ammonium persulfate ($(NH_4)S_2O_8$) from Centrohem (Serbia). Distilled water was used for the preparation of all solutions, as well for the rinsing process. All materials were used without further treatment or purification.

Preparation of PANI-based powder

The PANI-based polymer powder was prepared by modifying the experimental procedures described by Yilmaz (2007). Briefly, 0.054 mole of ammonium persulfate was dissolved in 70 ml of 0.5 M IA in a 150 ml beaker and 0.054 mole of aniline was dissolved in 75 ml of 0.5 M IA in another 150 ml beaker, for one hour, at room temperature and low speed (~400 rpm). Afterward, the solution of $(NH_4)S_2O_8$ was added slowly to the aniline solution and then mixed for additional one hour, at the same conditions as already stated above. The obtained precipitate ("cake") in the form of emeraldine salt was collected on a Büchner funnel and flask using a water aspirator. The precipitate "cake" was washed with plenty (~1 l) of distilled water until the filtrate became colorless. After washing, the precipitate remained under suction for ~10 minutes until significant cracking of the moist "cake" occurred. Afterwards, the partially dried "cake" was transferred to a drying chamber at 37°C for additional few hours (until complete drying). Finally, the obtained dry formulation was ground with pestle and mortar to fine powder and kept in desiccator until further use.

CHARACTERIZATION OF THE PREPARED PANI-BASED POWDER

ATR FT-IR Analyses

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

SEM Analysis

SEM analysis was performed using electron microscope Tescan Mira3 XMU (Cranberry Township, USA). Prepared powder formulation was recorded in dry state. Before the analysis, dry powder formulation was coated using gold/platinum alloy (15/85) under vacuum using Polaron SC502 sputter coater.

Optical microscopy

The obtained PANI-based powder formulation and BVDA Magnetic black powder (control powder) (BVDA, The Netherlands) 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 backlight. Prior to imaging under the microscope, latent fingerprints left on microscope glass slides were developed using prepared powder formulation and the control powder.

Development of latent fingermarks

In order to determine the performances of PANI-based powder formulation, three donors (two male and one female), using only a thumb of the right hand, deposited sebaceous/oily and dry fingermarks onto different non-porous, semi-porous and porous surfaces, i.e. plywood, glass and paper. The marks were then left under laboratory (humid) conditions for a short period of time. That period allowed the traces to dry and reduce the residues, until the latent fingerprints were developed with synthetized powder formulation and the control powder, using BVDA Squirrel hair brush and BVDA Magnetic brush (BVDA, The Netherlands), respectively (International Fingerprint Research Group, 2014).

Optical microscopy was used to determine performances of PANI-based polymer powder in visualizing latent fingermarks on glass surface, where the best results were obtained. Therefore, sebaceous fingerprints were randomly deposited onto the glass microscopic slides (properly labeled) and left for a few minutes. After this period, the fingermarks were separated into halves using a thin glass barrier and then the prepared powder formulation and BVDA Magnetic black powder were used for their visualization, according to the International Fingerprint Research Group (IFRG) (International Fingerprint Research Group, 2014).

FINDINGS

Prepared PANI-based polymer powder was used to develop latent fingermarks deposited onto three different substrates: plywood, glass and paper. Three different donors deposited fingermarks in accordance with the Guidelines proposed by the IFRG (International Fingerprint Research Group, 2014). Deposited sebaceous and dry fingermarks were halved with a thin slide barrier and two different powders were applied on the same fingermark – synthetized powder was applied to the left and BVDA Magnetic black powder (control powder) was applied to the right barrier side, using BVDA Squirrel hair brush and BVDA Magnetic brush, respectively. Both PANI-based powder and control powder were applied on mul-

tiple number of fingerprints on each substrate, to determine the performances and reproducibility of their application, i.e. to obtain satisfying results. The best results were obtained with the sebaceous fingerprints deposited onto the glass surface and developed with prepared PANI-based powder. On the other hand, development of dry fingerprints on each substrate with both powders was poor, without fingerprint basic form(s) and the papillary lines and therefore additional studies should be conducted, as it is already well known that visualization of dry marks is a challenging problem in forensic investigation of fingerprints (Cadd, Islam, Manson, & Bleay, 2015). The results obtained on plywood and paper substrate were satisfying, but with visible "overpowdering" of the fingerprints, since more powder particles retained in the porous (surface) structure of mentioned substrates. Additionally, disruption of papillary lines flow was observed on white paper surface, probably due to porous structure of the surface and the loss of fingerprint (sweat and lipid) residues.

Figure 1 shows sebaceous fingerprints of one donor, developed with prepared powder (left-half side of the images) and the control powder (right-half side of the images) on three different substrates, i.e. glass, paper and plywood. The prints were then photographed under visible light with a 12 MP camera (aperture $f/2.2$, pixel size 1.22 μm), using white background surface for appropriate contrast (for glass and paper substrate).

When comparing fingerprints developed with PANI-based powder and BVDA Magnetic black (control) powder on different surfaces, the best results were obtained on glass surface, with visible and clear fingerprint image, basic patterns, papillary lines and some minutiae points, as well (Figure 1, a)). PANI-based powder also showed satisfying results on paper and plywood surface, with obvious fingerprint pattern and basic characteristics, but with noticeable disruption of papillary lines flow (on paper surface) and "overpowdering" of fingermarks (on plywood surface) (Figure 1, b) and c)). On the other hand, by comparing PANI-based powder with control powder in developing latent fingermarks, it is evident that equally good and comparable results were obtain on each surface, showing satisfying and very promising result.

a) b) c)

Figure 1. *Sebaceous fingerprints developed on: a) glass, b) paper and c) plywood surface, using PANI-based powder (left-half side of the images) and BVDA Magnetic black powder (right-half side of the images), recorded under visible light using white background surface for appropriate contrast (for glass and paper substrate).*

ATR FT-IR Analyses

ATR FT-IR analyses were performed in order to determine interactions between components of synthetized formulation, i.e. interactions due to polymerization process. Figure 2 shows spectra of PANI-based polymer powder (emeraldine salt) and pure aniline. Both spectra contain some specific bands: very weak and broad band around 3400–3200 cm^{-1}, assigned to the N–H stretching vibrations (primary and secondary amines), and weak band near 3050–3030 cm^{-1} due to the C–H stretching (Habib & Maheswari, 1989; Kulkarni, Viswanath, & Khanna, 2006; Yilmaz, 2007). Peak shifting and decrease in intensity from 1599 cm^{-1} at spectrum of pure aniline to 1568 cm^{-1} at spectrum of prepared PANI-based powder can be related with mixed C=C and C–N stretching in a quinoid ring (Kulkarni, Viswanath, & Khanna, 2006; Yilmaz, 2007). Furthermore, the band around 1496 cm^{-1} at spectrum of pure aniline shifts to the band around 1488 cm^{-1} of a lower intensity at spectrum of PANI-based polymer powder and it is associated with C–C stretch in a benzenoid ring and C–H mixed vibrations (Quillard, Louarn, Lefrant, & Macdiarmid, 1994; Yilmaz, 2007). The peak around 1272 cm^{-1} at spectrum of pure aniline resulted in two new bands at spectrum of PANI-based powder, around 1294 and 1236 cm^{-1}, which can be assigned to C–N stretching (secondary aromatic amine) and C–H bending vibrations, due to bonding of aromatic rings in the polymerization process (Habib & Maheswari, 1989; Kulkarni, Viswanath, & Khanna, 2006; Yilmaz, 2007). Additionally, very weak peak around 1117 cm^{-1} at spectrum of pure aniline is slightly shifted to a broad band around 1120 cm^{-1} at spectrum of PANI-based powder, probably related with deformation of aromatic ring and C–H (in plane) bending (Quillard, Louarn, Lefrant, & Macdiarmid, 1994; Kulkarni, Viswanath, & Khanna, 2006; Yilmaz, 2007). Decrease in

intensity of peak around 880 cm^{-1} at spectrum of prepared powder is associated with plane vibration in para-disubstituted aromatic rings, indicating polymer formation (Quillard, Louarn, Lefrant, & Macdiarmid, 1994; Kulkarni, Viswanath, & Khanna, 2006). Strong peak near 499 cm^{-1} at spectrum of pure aniline resulted in a formation of a weak peak around 505 cm^{-1} at spectrum of prepared powder, which can be ascribed to deformation of C–H (out of plane) of 1–4 disubstituted aromatic ring (Kulkarni, Viswanath, & Khanna, 2006; Yilmaz, 2007).

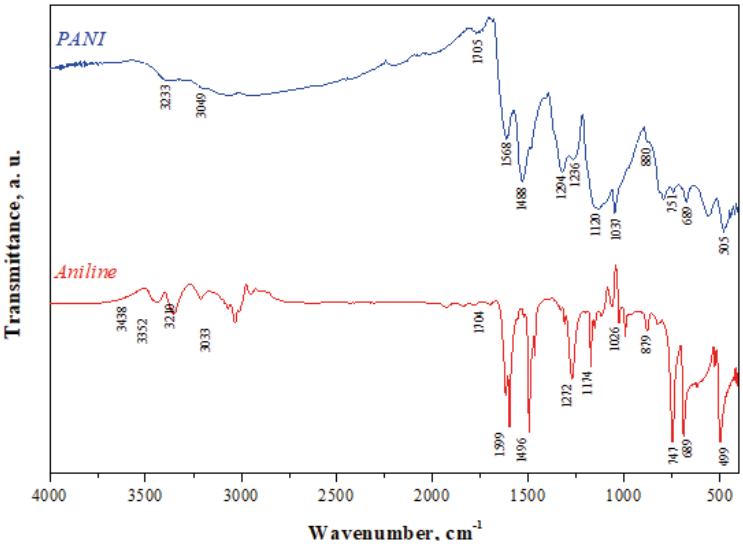


Figure 2. *ATR FT-IR spectra of prepared PANI-based powder (emeraldine salt) and pure aniline.*

The shifting of peaks from spectrum of pure aniline to spectrum of prepared powder was also confirmed in studies of aniline polymerization by Trchova et al. (2005) and aniline doping by Ilic et al. (2000). A broad band around 1150-1100 cm^{-1} was assigned, as stated by Quillard et al. (1994), to the "electronic like band" and is considered to be a measure of the degree of delocalization of electrons and therefore denoted as a characteristic peak of PANI conductivity. Additionally, in the region around 1300 cm^{-1}, the peaks are associated with the presence of aromatic amines in polyaniline (Yilmaz, 2007).

SEM Analysis

SEM analysis was performed in order to determine microparticle morphology and surface fineness, their size and uniformity. Figure 3 shows SEM micrographs of prepared PANI based-powder, at different magnifications.

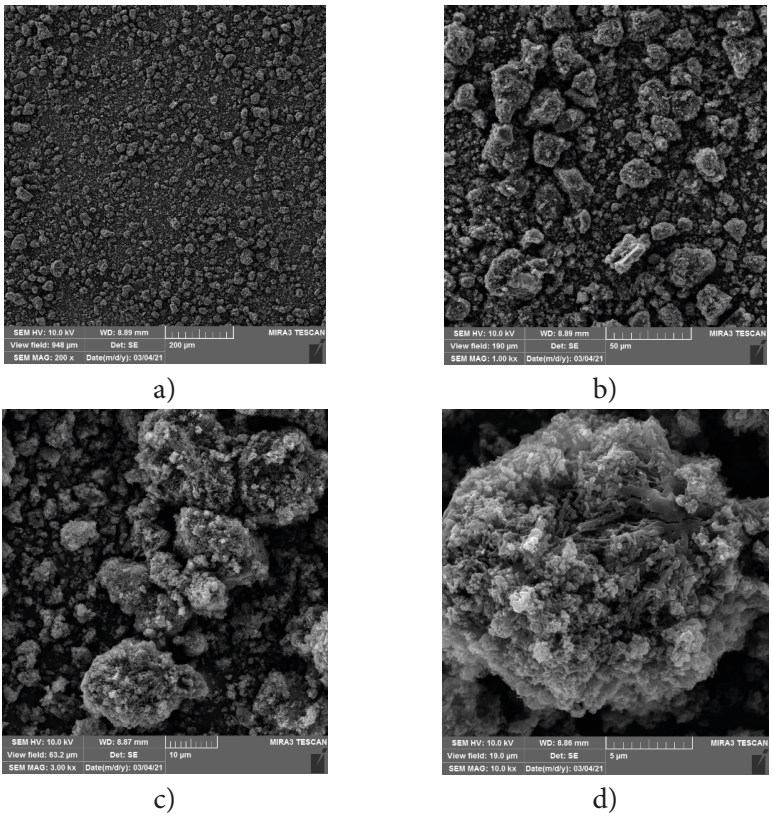


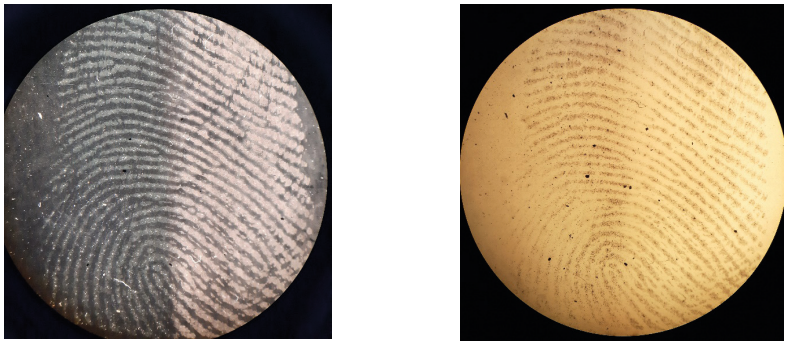
Figure 3. *SEM micrographs of PANI-based polymer powder, at different magnifications: a) ×200; b) ×1000; c) ×3000; d) ×10000.*

SEM analysis showed that the particles of prepared powder were somewhat fine and uniform in size, which is additionally complemented by their small diameter size. The diameter of particles of prepared powder (~ 20 µm) was even smaller than some commercial magnetic powders, described in research of Gürbüz and associates (2015). Therefore, good binding of these particles to the fingerprint sweat and lipid residues was observed. We assume that small diameter particles managed to bind to and visualize papillary line traces with their continuous flow (on glass surface) and did not retain in the interpapillary space, which was very satisfying result.

Optical microscopy

In order to confirm results obtained from initial testing of powders, as well as hypothesis from SEM analysis, PANI-based powder and BVDA Magnetic black powder were used for visualization of latent fingermarks on glass surface. Therefore, three donors deposited sebaceous fingermarks onto labeled glass microscop-

ic slides using technical scale (force applied to accommodate 100–150 g, per fingermark) and the fingermarks were left for a few minutes, which allowed the traces to dry and reduce the residues. After this period, the fingermarks were separated into halves using a thin glass barrier, and then two different powder samples were used for their visualization – synthetized PANI-based powder was applied to the left and BVDA Magnetic black powder (control powder) was applied to the right barrier side, using BVDA Squirrel hair brush and BVDA Magnetic brush, respectively. Afterwards, the samples of enhanced fingermarks were recorded under the optical microscope (magnification ×15), using dark-field (Figure 4, a)) and bright-field (Figure 4, b)) contrast techniques.



a) b)

Figure 4. *Sebaceous fingerprint deposited onto glass microscopic slide, left for a few minutes and developed using PANI-based powder (left-half side of the images) and BVDA Magnetic black powder (right-half side of the images), recorded with optical microscope (magnification ×15), using: a) dark-field and b) bright-field contrast techniques.*

When compared to the BVDA Magnetic black powder, PANI-based powder showed even better results in terms of visualizing latent fingerprints, by developing the papillary lines with their continuous flow and making perceptible some minutiae, as well. When observing the fingerprint developed with control powder, obtained fingerprint pattern was somewhat blurred and "overpowdered", but with visible papillary lines and some minutiae points. Additionally, when applied with a brush, prepared powder formulation bound with fingerprint residues and did not remain in the interpapillary spaces. Based on the obtained results, very promising visualization of sebaceous fingerprints was achieved using glass surface as a substrate, and with cheap PANI-based powder system.

ORIGINALITY/VALUE

In this research, PANI-based polymer powder, obtained by simple precipitating method, was prepared and characterized with the aim of evaluating its performance as a fingerprint powder. PANI-based powder was used due to low price and availability of aniline, as well as for easy synthesis process. Based on the obtained results, prepared powder formulation showed the best properties when developing sebaceous fingermarks deposited onto glass substrate, with good binding capacity to the fingermark residues and their clear development. Additionally, satisfying results were obtained on white paper and plywood substrate, with obvious fingerprint image, basic patterns and some minutiae points, as well. However, there were some drawbacks: disruption of papillary lines flow was observed on white paper substrate; the "overpowdering" of the fingermarks was present when developing marks on plywood substrate; dry fingermarks were not visualized on any substrate, with neither powder, so additional studies need to be performed to overcome these problems. On the other hand, as many commercial fingerprint powders, this formulation requires no prior knowledge, the method itself is non-destructive and prepared powder is easily applicable with standard/commercial fingerprint brushes. Finally, additional researches could include doping of prepared formulation, with the aim of expanding utilization of this PANI-based powder system on other (conductive) substrates and potentially supplementing or replacing some of the routinely used physical methods (fingerprint powders) in visualizing latent fingermarks.

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