Article

Water-Soaked Porous Evidence: A Comparison of Processing Methods

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Abstract: This study compared the U.K. Home Office formulation for physical developer (PD) against Oil Red O (ORO) and a modified formulation of physical developer (MPD) that uses Tween 20 instead of Synperonic-N for enhancing fingermarks. Three different donors deposited fingermarks on porous surfaces (white paper, leaflets, and cardboard), with aging periods varying from 7 to 28 days. None of the techniques that were tested provided enhancement of latent fingermarks on leaflets, whereas poor-quality enhancement was observed on cardboard. In contrast, all techniques were more successful on white paper surfaces. The results obtained on white paper suggested that PD and MPD performed similarly, with PD detecting 82.3% of the deposited fingermarks and MPD detecting 86.5% of the deposited fingermarks. PD yielded a higher percentage (38.5%) of fingermarks with fine ridge detail (i.e., those with grade 2 or above) than MPD (35.4%). ORO, however, yielded poor results, enhancing only 4.5% of latent fingermarks, but showed no ridge detail in any of the enhancements (i.e., only showed grade 1 enhancements.)

Introduction

Approximately 99% of a latent fingermark is water [1]. This is rapidly lost through evaporation, meaning that over time, methods that rely on water being present lose their effectiveness. These methods also lose their effectiveness if the item has been exposed to water or high humidity. On aged latent finger-

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marks, or those that have been exposed to water or humidity, it is therefore necessary to target the portions of the residue that are less likely to have been lost. Latent fingermark residues can be divided into two portions: water soluble and water insoluble [2]. The water-soluble portion contains amino acids and salts that can be targeted by methods such as ninhydrin and DFO. However, on contact with water, the water-soluble portion is lost and what remains is mostly the water-insoluble portion. This water-insoluble portion can be subdivided into a labile fraction and robust fraction [3]. The labile fraction contains components likely to undergo changes in the short term such as saturated or unsaturated fatty acids and triglycerides. These materials oxidize quickly when exposed to air but not when exposed to water. The robust fraction consists of large water-insoluble proteins and lipoproteins, which can bind strongly to the cellulose in paper via hydrogen bonding, allowing such residues to remain on paper for long periods of time. It has been reported that Oil Red O (ORO) interacts with the labile fraction whereas physical developer (PD) interacts with the robust fraction [4].

ORO, also known as solvent red 27, is a lipophilic dye from the diazo family similar to Sudan black (solvent black 3)[2]. ORO received its first reported forensic application in 2002 for the use of visualizing lip prints [5] and was later used by Beaudoin in 2004 as a staining solution for the enhancement of latent fingermarks on porous surfaces [6]. Fingermarks enhanced by ORO show red ridge detail on a light pink stained background. The study aimed to determine whether ORO could enhance latent fingermarks on wetted papers as effectively as PD. Although at the time no direct comparison was attempted, subsequent studies have been carried out by Rawji and Beaudoin [7], Salama et al. [4], and Wood and James [8], who have suggested that, in certain scenarios, ORO can match or outperform PD. Other studies investigated modifications of the original ORO methodology by removing the filtration step and removing and altering the buffer used [4]. However, despite many alterations, the original formulation [6] was found to work the best. Because ORO targets the labile fraction of latent residues, the performance of ORO decreases over time whereas the performance of PD increases. Fingermarks that are known to be older than 28 days are recommended to be treated with PD rather than ORO [2]. Nonetheless, ORO has successfully been used to enhance a 21-year-old fingermark on paper recovered from an attempted fire [9]. The ORO staining process has fewer processing stages than PD and may be perceived as faster and simpler; however,

overall it may take longer [10]. Furthermore, sequential treatment of latent fingermarks with DFO–ninhydrin–ORO may not be beneficial because solvents used in ninhydrin and DFO may remove some of the constituents targeted by ORO [11]. This effect appears to be limited when HFE-7100 is used as a solvent for the preparation of ninhydrin and DFO solutions rather than petroleum spirits [11]. Additional research by McMullen and Beaudoin [12] demonstrated that the sequential process of DFO–ninhydrin–ORO did not prevent the development of useful fingerprints and that ORO developed fingerprints that had been undetected with the DFO–ninhydrin sequence.

PD is based on a redox reaction that produces metallic silver particles that interact with the latent fingermark residues that are present in the robust fraction such as triglycerides, wax esters, hydrocarbons, monounsaturated lipids, and EDTA [13]. PD reagent consists of iron (III) nitrate, ammonium iron (II) sulphate, silver nitrate, citric acid, and surfactants [14]. The iron (III) nitrate and ammonium iron (II) sulphate provide the Fe^{3+} and Fe^{2+} ions required for the redox reaction. Silver nitrate is the source of colloidal silver particles that interact with the latent fingermark residue. Citric acid is used to adjust the pH and change the charge of the silver particles from positive to negative to prevent aggregation. Prior to chemical processing with PD, papers must be given a prewash in maleic acid (or any other acid that does not contain chlorine) to neutralize calcium carbonate that is present which is often used as an inexpensive filler in paper. If this is not done, the calcium carbonate can act as a nucleation site, causing the surface of the paper to turn black. Thicker papers may also require extra soaking before being treated with PD working solutions. The surfactant solution is commonly prepared using n-dodecylamine and Synperonic-N, which assist in keeping the cationic surfactant soluble and can also help further stabilize the developing colloidal particle [13].

Synperonic-N is a 25 to 40% (v/v) solution of a nonylphenol ethoxylate nonionic surfactant (typically Synperonic-NP8), and degradation of this compound causes the mixture to become less soluble and biodegradation becomes more difficult. EU directive 82/242/EEC seeks to ban chemicals where the degradation products are considered more harmful than their precursor, which is the case for nonylphenol ethoxylates [15]. Consequently, because of environmental concerns, other EU directives banned the sale of nonylphenol ethoxylates for industrial use, leading to the consideration of other surfactants for the PD process. The United States Secret Service (USSS) compared different

surfactants for the PD process: Tween 20, Tween 80, and NonIdet P40 (also known as Triton-X 100 and Tergitol NP 9), and results from this study indicated that Tween 20 was the most suitable replacement [14]. Similarly, the U.K. Home Office Centre for Applied Science and Technology (CAST, previously HOSDB) evaluated Tween 20, Tween 80, Synperonic 91/5, Synperonic 91/6, Synperonic 13/6.5, and caflon-N as potential substitutes for Synperonic-N. Tween 20 appeared to be the most suitable alternative; however, none of the replacements were deemed to be as effective as Synperonic-N. Therefore, in the United Kingdom, no recommendation to replace Synperonic-N was made [16]. The large branched structure of Tween 20 provides superior protection for the silver colloid, preventing agglomeration and improving stability. Working solutions prepared using Synperonic-N have a shelf life of approximately 7 to 14 days; however, those prepared with Tween 20 have a reported shelf life of up to 2 months [17–19]. It has also been noted that PD solutions prepared with Tween 20 may provide superior enhancement if allowed to rest for two to three weeks prior to use [13].

A number of studies [4, 7] have demonstrated that ORO is superior to PD; however, such studies used "loaded" fingermarks. In contrast, other studies [8] using "ungroomed" fingermarks found that ORO did not outperform PD for the detection of latent fingermarks up to seven days old. This current study expanded the aging period time to a maximum of 28 days to assess how the effectiveness of each technique changed over time. Furthermore, this study aims to compare the effectiveness of PD as recommended by the U.K. Home Office against a modified PD (MPD) with an alternative surfactant (Tween 20) and ORO because previous studies compared only two techniques directly: ORO against PD [4, 7, 8] and PD against MPD [18–20].

Materials and Method

The methodology in this study follows guidelines recommended by CAST [21].

Donors

The study used three donors: two male and one female between the ages of 23 and 40. Donors were asked to wash their hands one hour before depositing any fingermarks and to not apply any cosmetics or purposely rub their hands on their head or face but to otherwise continue their daily tasks as normal. Before deposition, donors were asked to rub their hands together

thoroughly to ensure even distribution of residues across the fingertips.

Substrates and Aging Periods

Fingermarks were deposited on three surfaces:

- Plain white copier paper–75 gsm
- Glossy, colored leaflets
- Brown corrugated cardboard

Before fingermark deposition, surfaces were cut into strips approximately 21 cm x 5 cm long. A center line and box edges were drawn in pencil to aid donors with placement when depositing the fingermarks. Six fingermarks were deposited along the center line of each strip in a depletion series, with no new residues being applied to the finger after each deposition. Each donor did this twice for each method to be tested on each surface because of differences in pressure that may occur on one side of a fingermark compared to the other [21]. After fingermarks had been deposited, strips were left to age on the laboratory bench top away from direct sunlight for the required time. Four aging periods were tested; 7, 14, 21, and 28 days. After aging, the strips were submerged in a clean Pyrex dish containing tap water for one hour before being removed and placed on chemical-free paper towels and left to dry overnight. Before chemical processing, the strips were halved, with each half to be treated with a different method to give a split series. This was repeated, but the methods were reversed to account for pressure differences from the individual making the fingermarks. Each strip was labeled with the donors' identification, the date the print was deposited, the aging period, and which treatments were being compared–with the method used on that strip being underlined.

Chemical Formulations

All working solutions used in this study were made fresh. The PD working solution was prepared as per the CAST guidelines [22]. MPD was also made up following these guidelines, with Tween 20 replacing Synperonic-N on a 1:1 ratio [10]. The ORO solution was prepared following Beaudoin's original recipe [6]. The PD and MPD working solutions were made and used on the day required. The ORO staining solution was made fresh for processing the 7- and 14-day aged fingermarks. After processing, the remaining ORO staining solution was stored in a brown glass bottle and stored away from sunlight and used again for processing the 21- and 28-day aged fingermarks because it

has a reported shelf life of eight months [2]. The ORO buffer solution was made fresh as required. Water used for making PD and MPD was tested for appropriate purity using a test devised by Hardwick [23] where a few crystals of silver nitrate are placed into a small beaker containing the water to be used. If the water remains clear, then the water has sufficient purity; if the water becomes cloudy, it does not have sufficient purity. Pyrex dishes that were used in processing the strips were thoroughly cleaned before use, firstly by cleaning under running tap water using clean chemical-free paper towels for 10 minutes and then with three rinses of distilled water.

PD Reagent Preparation and Application [22]

Redox solution: 900 mL of extra pure deionized water was measured out and placed in a 2 L beaker. 30 g of iron (III) nitrate, 80 g of ammonium iron (II) sulphate, and 20 g of citric acid were added in the order given and as quickly as possible while stirring with a magnetic stirrer. This was stirred until all the solid had dissolved, and for a further five minutes.

PD stock detergent solution: Stock detergent consisting of 1 L of extra pure deionized water, 2.8 g of n-dodecylamine acetate, and 2.8 g of Synperonic-N was used.

Silver nitrate solution: 50 mL of extra pure deionized water was measured out and placed in a 100 mL beaker. 10 g of silver nitrate was added and mixed using a magnetic stirrer for approximately one minute, until a colorless solution was given. This was made as needed and added immediately to make the working solution.

Acid prewash solution: 1 L of extra pure deionized water was measured out and placed in a 2 L beaker. 25 g of maleic acid was then added and stirred using a magnetic stirrer until completely dissolved.

PD working solution: 900 mL of the redox solution was measured out and placed in a 2 L beaker. 40 mL of the PD stock detergent solution was added and stirred using a magnetic stirrer for two minutes. 50 mL of the silver nitrate solution was then added and stirred for a further two minutes. (The working solution may vary in color from a clear yellow to a dark brown.)

Application: Strips containing latent fingermarks were first placed in a dish containing maleic acid solution for 10 minutes or until bubbles had stopped appearing, whichever was the longer, to neutralize the calcium carbonate often used as filler

in paper. This prevents the background from darkening too quickly in the working solution. The strips were then moved, using nonserrated plastic forceps, into the dish containing the PD working solution and rocked occasionally for approximately 20 minutes or until the background had begun to darken, affecting the contrast between the fingermarks and the background. The strips were then moved, using nonserrated plastic forceps, into a dish containing distilled water and were rinsed for approximately five minutes. This was repeated two more times in separate dishes, with the water being replaced when yellow coloration occurred. Once rinsed, the strips were removed from the dish and placed on chemical-free paper towel and left to dry overnight before evaluation.

MPD Reagent Preparation and Application [18]

The preparation and application of MPD was performed as described for PD using Tween 20 as the surfactant.

Tween 20 detergent solution: 1 L of extra pure deionized water was measured out and placed into a 2 L beaker. 2.8 g of n-dodecylamine acetate was then added followed by 2.8 g of Tween 20. The solution was stirred using a magnetic stirrer for approximately 30 minutes.

ORO Reagent Preparation and Application [6]

Staining solution: 1.54 g of ORO was added to 770 mL of methanol and stirred using a magnetic stirrer until dissolved. 9.2 g of sodium hydroxide was added separately to 230 mL of deionized water and stirred using a magnetic stirrer until dissolved. The water containing the sodium hydroxide was then added to the methanol containing the ORO and mixed. Once mixed, this was filtered before being stored in a brown bottle, away from light.

Buffer solution: 26.5 g of sodium carbonate was added to 2 L of deionized water and stirred using a magnetic stirrer until dissolved. 18.3 mL of concentrated nitric acid was slowly added while stirring. The volume was then increased to 2.5 L, using deionized water.

Application: Strips containing latent fingermarks were placed into a Pyrex dish containing the staining solution. The dish was then covered with Clingfilm and placed on a plate shaker and agitated for at least 60 minutes but less than 90 minutes. Strips were then moved, using nonserrated plastic forceps, to a dish containing the buffer solution, and left for approximately

five minutes. The strips were then rinsed in a dish containing distilled water before being placed on chemical-free paper towel and left to dry overnight before evaluation.

Controls

Before processing the strips with the donor fingermarks, all reagents were tested using positive and negative controls. Positive controls consisted of fresh, loaded fingermarks on white paper strips. Negative controls were the tested substrates with no fingermarks deposited on them.

Grading

When dry, the fingermarks were graded using the grading scheme recommended by CAST in Table 1 [21]. This allowed the number of detected fingermarks and the quality of the enhancement to be recorded.

For the purpose of this study, a positive enhancement was considered to be any fingermark that showed a grade 1 enhancement or higher. This included fingermarks where there was evidence of contact but not necessarily any ridge detail present. Fingermarks with usable ridge detail were those that showed grade 2 or higher enhancements.

Grade	Level of Detail
0	No evidence of print
1	Some evidence of contact but no ridge detail present
2	Less than 1/3 of print showing clear ridge detail
3	Between 1/3 and 2/3 of print showing clear ridge detail
4	Over 2/3 of print showing clear ridge detail

Table 1

Grading scheme for assessment of developed fingermarks.

Results

Controls

All controls provided enhancement of latent fingermarks on positive control samples but no enhancement on negative control samples.

Comparison of Techniques

Table 2 shows the total number and percentage of positive enhancements for each technique on all surfaces, illustrated graphically in Figure 1. The initial number of 7- and 14-day aged fingermarks totaled 144 for each technique. All surfaces with 7- and 14-day aged fingermarks were processed.

Corrugated cardboard showed some grade 1 enhancements, but none of the fingermarks showed usable ridge detail. Leaflets suffered from heavy background staining from all methods and no usable fingermarks were detected. Because of the poor number and quality of fingermark enhancements on corrugated cardboard and leaflets, these were discontinued. A further 144 fingermarks were tested that had been aged for 21 and 28 days on white paper only. The data and tables presented are based on results obtained from white paper alone. Table 3 shows the results obtained from white paper. This information is also presented graphically in Figure 2. These results show the similarities in the effectiveness of PD and MPD, however demonstrate that ORO performed poorly in comparison.

Surface	Total No. of Fingermarks	Number of Positive Enhancements (grades 1–4)			% of Positive Enhancements		
		PD	MPD	ORO	PD	MPD	ORO
Corrugated Cardboard	144	14	6	2	9.7	4.2	1.4
Leaflets	144	0	0	0	0	0	0
White Paper	288	237	249	13	82.3	86.5	4.5

Table 2

Number and percentage of positive enhancements by each technique and surface.

Technique	Total No. of Fingermarks	Grade 0	Grade 1	Grade 2	Grade 3	Grade 4
PD	288	51 (17.7%)	126 (43.8%)	48 (16.7%)	39 (13.5%)	24 (8.3%)
MPD	288	39 (13.5%)	147 (51.0%)	61 (21.2%)	24 (8.3%)	17 (5.9%)
ORO	288	275 (95.5%)	13 (4.5%)	0 (0%)	0 (0%)	0 (0%)

Table 3

Number and percentage of grades for the enhancement of fingermarks on white paper by each technique.

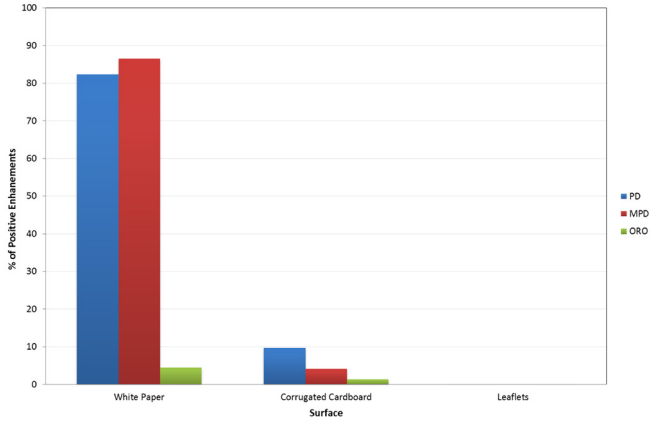

Figure 1

Percentage of positive enhancements on each surface by technique.

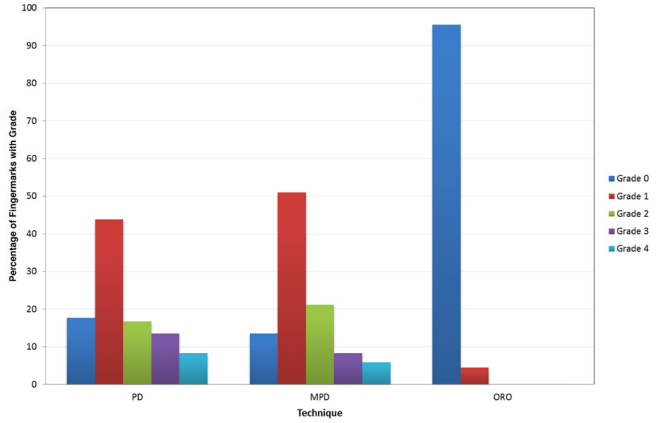

Figure 2

Percentage of fingermarks on white paper per technique for each grade.

Discussion

In general, the results and enhancement that were observed did not appear to be affected by the different donors. None of the enhancement techniques in this study gave fingermarks with ridge details on the leaflets because of heavy background staining. Firstly with ORO, background staining was so intense that if any fingermarks had been enhanced, these were likely lost in the background coloration. With PD and MPD, despite the leaflets having been submerged in maleic acid for 10 minutes, blackening of the background occurred almost immediately when the leaflets were submerged in the working solution. The maleic acid is used as a prewash to remove excess calcium carbonate that is often present in paper and is not specifically designed for leaflets. Therefore, it may be necessary to use a different pre-treatment to prevent background darkening occurring so quickly. Other techniques, such as Sudan black, may be more suitable for use on leaflets, although background staining is still likely to occur.

Enhancement of latent fingermarks on cardboard also exhibited signs of staining with ORO, causing a red background that may have masked any latent fingermarks that were enhanced. The poor performance of ORO on cardboard reflects the findings from other studies [12]. Cardboard gave some grade 1 fingermark enhancements when using PD and MPD, but no fingermarks with usable ridge detail were observed. This had also previously been observed in a study by Wood and James [8] where it was noted that neither PD nor ORO gave decent fingermarks on card surfaces when using normal fingermarks as opposed to loaded fingermarks.

On white paper surfaces, both PD and MPD outperformed ORO in terms of the number of fingermarks detected and quality of enhancement. This may be explained by the fact that ORO interacts with the labile fraction of the water-insoluble portion of a latent print. Because of the time difference between the deposition of the fingermarks and the processing of the paper, the labile fraction may degrade upon exposure to air, meaning that ORO is less useful for fingermarks that have been aged. Nonetheless, fresh fingermarks or fingermarks that have been submerged in water shortly after deposition may provide suitable enhancement, and further research in this area is required.

Figure 3 illustrates 28-day aged fingermarks on a white paper strip that was treated with ORO (left) and PD (right). The strip was halved and treated with the separate methods before being recombined once dry. The image shows the first three fingermarks deposited in the depletion series. This demonstrates the effectiveness of PD, even on fingermarks that have been aged 28 days, because they still show usable ridge detail even when residues have been depleted by multiple depositions. In contrast, the half of the strip that was treated with ORO shows no evidence of contact and no usable ridge detail. Figure 4 demonstrates the first fingermark deposited in the depletion series on a white paper strip. The fingermarks on the strip were aged 28 days before being exposed to water for one hour. Each half was then treated using MPD (left) and PD (right). The image demonstrates the comparability in the effectiveness of PD and MPD because both halves reveal fingermarks with usable ridge detail.

Figure 5 shows the first fingermark in the depletion series deposited on white paper that was aged 21 days and submerged in water for one hour before being halved and being treated with ORO (left) and MPD (right). It can be seen on the 21-day aged print that MPD was still able to give usable ridge detail on enhancement whereas ORO did not provide any enhancement. This again demonstrates that ORO may be less suitable on fingermarks that have been aged for any length of time, negatively affecting its ability to enhance fingermarks because of changes in the labile fraction.

Figure 6 shows the first fingermark deposited in the depletion series on a white paper strip. This fingermark was aged seven days before being submerged in water for one hour. Then the halves were treated with ORO (left) and PD (right). PD shows enhancement with usable ridge detail. However, ORO, despite the fingermark only being aged seven days, did not manage to provide any suitable enhancement.

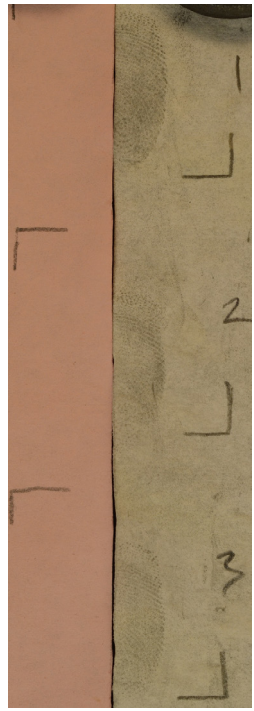

Figure 3

Enhancement of 28-day aged fingermarks on white paper: ORO (left) v PD (right).

Figure 4

Enhancement of a 28-day aged fingermark on white paper: MPD (left) v PD (right).

Figure 5

Enhancement of a 21-day aged fingermark on white paper: ORO (left) v MPD (right).

Figure 6

Enhancement of a 7-day aged fingermark on white paper: ORO (left) v PD (right).

Conclusion

When comparing the results that were obtained using PD and MPD, the two techniques yielded similar results in terms of overall quality. MPD showed grade 1 or higher enhancements on a slightly higher percentage of the latent fingermarks (86.5%) compared to PD (82.3%); however, PD showed a higher percentage of enhancements with usable ridge detail (grade 2 or above) compared to MPD (38.5% to 35.4%, respectively). The study demonstrates that the use of MPD may be a suitable replacement for PD. Future work aims to assess the impact on the enhancement methods when the article under examination is submerged in different types of water (e.g., river water, sea water, tap water).

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