

Article

Application of *K*-Carrageenan for One-Pot Synthesis of Hybrids of Natural Curcumin with Iron and Copper: Stability Analysis and Application in Papilloscopy

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Abstract: In this study, hybrid materials were synthesized incorporating curcumin, Cu²⁺ or Fe³⁺, and *Kappa*-carrageenan as a reducing agent to improve stability, considering that curcumin has low thermal and solution stability, which limits its applications. Colorimetric analysis showed color changes in the hybrids, ultraviolet–visible spectroscopy revealed band shifts in the hybrids, and infrared analysis indicated shifts in wavenumbers, suggesting changes in the vibrational state of curcumin after bonding with metal ions. These techniques confirmed the formation of hybrid materials. Thermogravimetric and chromatographic analyses demonstrated greater thermal and solution stability for the hybrids compared to curcumin. Additionally, the hybrid composites effectively developed natural and sebaceous latent fingerprints with good clarity and contrast on glass surfaces. Both composites performed similarly to commercial Gold[®] powder. When applied to surfaces representative of forensic scenarios, the composites were versatile, revealing sufficient fingerprint details for human identification on both porous and non-porous surfaces. Scanning electron microscopy images showed greater clarity in sebaceous and natural fingerprints developed with the Fe composite compared to the Cu composite.

Keywords: curcumin; hybrid materials; stability in solution; thermal stability; papilloscopy



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1. Introduction

Papilloscopy refers to the science dedicated to studying fingerprints, which vary between individuals and even in the same person. This uniqueness has made fingerprints a reliable method of human identification for over a century [1]. In forensic sciences, fingerprint analysis is based on Locard's exchange principle, which establishes that every contact

between two objects results in a transfer of traces between them. Consequently, the act of touching a surface transfers material from the ridges of the fingers onto a surface [2]. In criminal investigations, fingerprints are considered physical evidence, as they not only capture the pattern ridges but can also retain exogenous compounds such as drugs, explosives, and chemicals [3]. Latent fingerprints (LFPs) found at crime scenes are invisible to the naked eye and require specific methods to be visualized, which include optical (ultraviolet laser), chemical (cyanoacrylate fuming), and physical (powder dusting and deposition) methods [4,5]. One of the oldest techniques, still widely used by forensic experts, is the powder technique, especially when LFPs are deposited on smooth surfaces such as glass, metal, and plastic. The technique is simple, involving the application of powder with a specific brush onto the surface where the LFP is found, making the ridge patterns and their minutiae visible to the naked eye [6]. An ideal compound for developing fingerprint should provide high contrast, good adhesion properties, and desirable selectivity [1]. In this context, the search for a selective, high-contrast, low-cost, and reliable powder for developing LFPs has been relentless. Most available LFP powders use hazardous chemicals in their synthesis, and continuous use can cause side effects. Therefore, the use of natural products as a raw material source for the development of new LFPs is of great interest, as their use may result in a final product with little to no toxicity [7].

Based on this, curcumin, a natural dye extracted from the rhizomes of *Curcuma longa*, has emerged as a promising candidate [8]. Its structure comprises a β -diketone fraction, methoxy groups, phenolic hydroxyls, doubly conjugated bonds, and keto-enol tautomerism [9]. Studies report that the properties found in turmeric are mainly attributed to curcumin, making the isolated compound of great interest [10]. Interesting properties have been reported in isolated curcumin, including high antimicrobial activity [11], antibacterial properties [12], and antioxidant features [13], among others. However, isolated curcumin exhibits rapid photodegradation when exposed to light [14] and air [15], which limits its stability and effectiveness. Furthermore, the curcumin molecule is not stable in solution due to the β -diketone fraction of curcumin, which is a hydrolytic center. This hydrolytic center is involved in the elimination of the hydroxyl radical and in redox reactions, leading to the formation of degradation products, that is, minor phenolics [14], such as ferulic acid, vanillic acid, and vanillin [16]. To overcome these limitations, hybrid materials can be designed from various components in order to achieve original properties in a single material [17]. The β -diketone groups enable curcumin to chelate metal ions, leading to the formation of hybrid materials, which comprise a combination of two or more phases, whether organic or inorganic, with unique properties [18]. However, the properties of a hybrid material are not merely a sum of the individual contributions of its constituents but depend on the chemical nature of the organic and inorganic compounds. Due to their complexity, they can be classified into different categories. Among them, the usual classification is performed according to the type of interaction or the nature of the chemical bonds between the organic and inorganic components. As mentioned earlier, curcumin contains a fraction that acts as a strong chelating agent, and thus, hybrid materials synthesized from curcumin belong to class II, which consists of materials formed through covalent or ionic bonds [19]. For the synthesis of a hybrid material containing curcumin and a metal as organic and inorganic phases, respectively, a third phase with metal-reducing properties is needed. In this scenario, the use of *Kappa*-carrageenan (κ -car) as a reducer agent has attracted interest [20]. κ -car is a polysaccharide extracted from red macroalgae, featuring non-toxic, low-cost, edible, biodegradable, and biocompatible properties [21]. The negatively charged surface of κ -car enables easy interaction with positively charged metal ions through electrostatic attraction. Moreover, the presence of hydroxyl (OH) groups facilitates the reduction of these metals [22].

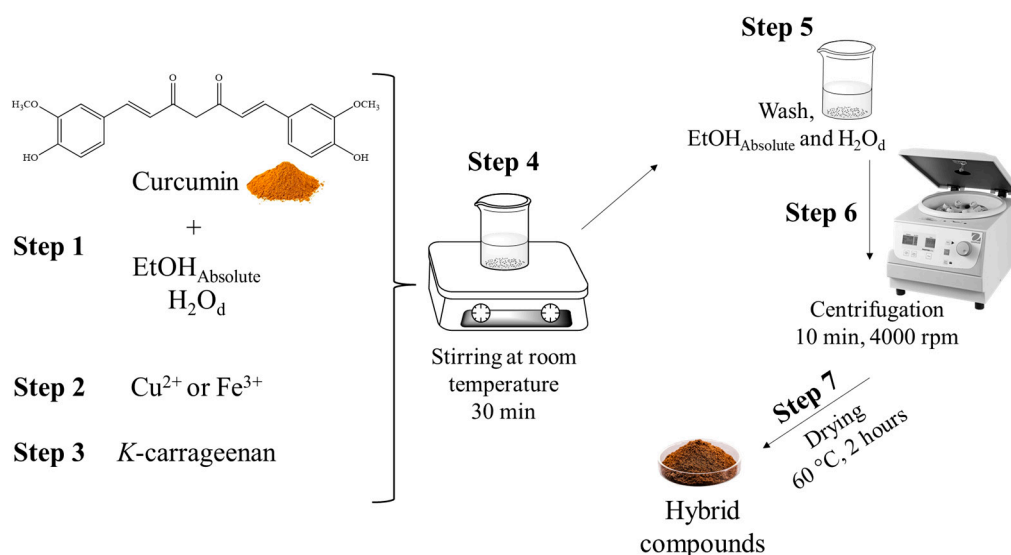
According to what has previously been reported, in this work, we continue our efforts to develop derivatives of organic compounds with applications in papilloscopy [7,23–32]. Therefore, this study reports the development of a new hybrid material of curcumin and *Kappa*-carrageenan combined with two low-cost copper and iron metals. The hybrid materials were evaluated for thermal stability and solution behavior. Finally, these hybrid composites were tested as potential LFP developers, offering a novel, low-toxicity alternative for fingerprint analysis.

2. Materials and Methods

The curcumin (diferuloylmethane, 65% purity) and *Kappa*-carrageenan were purchased from Sigma-Aldrich (St. Louis, MO, USA). Acetonitrile was purchased from Biograde (Durham, USA, USA-HPLC $\geq 99.99\%$). Tetrahydrofuran (THF) was purchased from Honeywell (Charlotte, USA, USA-HPLC $\geq 99.99\%$). Fingerprint powder (developer, Gold[®]) was purchased from Sirchie[®] (Youngsville, LA, USA). The other chemicals were of analytical grade and from commercial sources.

2.1. Synthesis of the Hybrid Materials

The hybrid materials were synthesized following a one-pot methodology adapted from Li et al. (2021) [33]. Briefly, in a beaker, we added copper acetate monohydrate ($\text{Cu}(\text{CO}_2\text{CH}_3)_2 \cdot \text{H}_2\text{O}$, Vetec-P.A) (0.1359 g, 0.0012 mmol) or iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, Vetec-P.A) (0.1834 g, 0.0008 mmol) into curcumin (0.5000 g, 0.0014 mmol) previously dissolved in a solution of ethanol (8 mL) and deionized water (4 mL). Then, *Kappa*-carrageenan (1.0000 g, 0.0023 mmol) was added to the mixture in the beaker, which reacted for 30 min at room temperature. The mixture was rinsed with deionized water (200 mL) and absolute ethanol (200 mL). Finally, the material was centrifuged (10 min, 4000 rpm) to separate the solid–liquid phases. The solid fraction was dried in an oven at 60 °C for 2 h. Scheme 1 presents the synthesis of new hybrid materials.



Scheme 1. Scheme for the synthesis of new hybrid materials. Step 1. Dissolution of curcumin in absolute ethanol and distilled water. Step 2. Addition of Cu^{2+} acetate or Fe^{3+} chloride. Step 3. Addition of *K*-carrageenan. Step 4. Stirring for 30 min. Step 5. Addition of distilled water and absolute ethanol for washing. Step 6. Centrifugation to separate the solid–liquid phases. Step 7. Drying of the hybrid materials in an oven at 60 °C for 2 h.

2.2. Characterization

The ultraviolet–visible spectroscopy (UV-Vis) of pure curcumin and both Fe and Cu hybrid materials was performed using a Bel LGS53 instrument, with the samples diluted in methanol solvent. The samples were also investigated by Fourier transform infrared spectroscopy (FT-IR), model SPIRIT (Shimadzu, Tokyo, Japan), using potassium bromide (KBr—[0.5%]). X-ray diffraction (XRD) patterns were recorded in the 2θ range from 10° to 60° using a Shimadzu XRD-6000 instrument. Thermogravimetric analysis (TGA) was performed using a Shimadzu DTG-60 instrument. The samples were evaluated in N_2 atmosphere, from 30 to $800^\circ C$ and with a heating rate of $10^\circ C/min$. To determine the color of the samples, *Kappa*-carrageenan, curcumin, copper acetate, iron chloride, copper hybrid, and iron hybrid samples were analyzed in a CR-400 colorimeter (Konica Minolta), with an observation angle of 10° , calibrated with porcelain, configured with D65 light. All analyses were performed in triplicate. The CIELab system (L^* , a^* , and b^*) was used to determine the colorimetric parameters. In this system, L^* represents lightness (from 0 (black) to 100 (white)), a^* corresponds to the green–red axis (negative values indicate shades of green, while positive values indicate shades of red), and b^* corresponds to the blue–yellow axis (negative values indicate shades of blue, while positive values indicate shades of yellow). To identify the variation between colors, ΔE was used, according to the following equation:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

where ΔE is the variation in all colors; ΔL is the variation in lightness; Δa is the variation in the red–green coordinates; and Δb is the variation in the blue–yellow coordinates [33].

2.3. High-Performance Liquid Chromatography with UV-Vis Detector (HPLC-UV-Vis)

The chromatographic analyses were performed using a Thermo Scientific Ultimate 3000, Waltham model, USA, with a standard auto-injector and UV-Vis detector. The separation of analytes was carried out on a C18 column (Ascentis, Bellevue, WA, USA) with a particle size of $5\ \mu m$, a pore size of $90\ \text{\AA}$, and a column size of $150\ mm \times 4.6\ mm$. For the determination of curcuminoids in the commercial curcumin sample, we employed an adapted method based on [34], where the isocratic mode was used with a mobile phase of 0.001% phosphoric acid (in water) and acetonitrile (20:80, v/v). The analysis was performed at a temperature of $45^\circ C$, with an injection volume of $20\ \mu L$ and a flow rate of $0.8\ mL/min$, for 5 min. The detection wavelength was 425 nm, and the samples were diluted in acetonitrile.

For the determination of the solution stability of curcumin and the hybrid materials, the method employed was adapted from Kumar et al. (2021) [16]. The isocratic mode was used, and a mobile phase mixture was employed: acetonitrile, tetrahydrofuran, and 2% acetic acid (aqueous) (26.3:63.2:10.5 v/v). The analysis was carried out at room temperature for 7 min. The injection volume was $20\ \mu L$, the flow rate was $1\ mL/min$, detection was achieved at 254 nm, and the samples were dissolved in acetonitrile. The analysis was conducted by program time: time zero (immediately after sample dilution), after 2 h, and after 24 h.

2.4. Preparation of Hybrid Composites

An adapted methodology previously described by Modwi was used to prepare the Fe and Cu hybrid composites [35]. Briefly, copper or iron precursor, silver mica powder (TiO_2/Ag_2O), carboxymethyl cellulose, and cellulose acetate were combined in the proportions of (20:15:15:50), respectively. The mixture was added to 200 mL of absolute ethyl alcohol. The solution was sonicated in an ultrasonic bath for 30 min. The resulting solution was transferred to a container of a dispersing mill (PE-075), where 150 g of zirconia beads

and 50 mL of ethyl alcohol were added. The composites were processed for 3 h at a rotation speed of 18,000 RPM. The final solution was centrifuged for 10 min at 4000 RPM. The supernatant was discarded, and the powder was dried in an oven at 100 °C for 4 h.

2.5. Determination of the Concentration of Cu and Fe in Hybrids

For the determination of Cu and Fe elements, the hybrids were first digested through an acid digestion process in a digestion block (Tecnal, Piracicaba, Brazil), following the methodology described by Fioravante [36]. Thus, 0.25 g of the samples was weighed into a glass tube, and 5 mL of nitric acid was added. The mixture was heated in a digestion block for 2 h at 100 °C. Subsequently, 2 mL of hydrogen peroxide was added, and the solution was returned to the digestion block for another 2 h at 100 °C. After cooling, the content was transferred to polypropylene tubes and brought to a volume of 12 mL with deionized water.

The elements were quantified using a flame atomic absorption spectrometer (FAAS), model AAnalyst 200 (Perkin Elmer, Singapore, Singapore), equipped with a hollow cathode lamp for each analyte to be determined (Lumina, Perkin Elmer) and a deuterium arc lamp as a background corrector. Additionally, acetylene (Linde, Barueri, Brazil) was used as the fuel gas, and compressed air was used as the oxidant gas. The calibration curve was prepared from a 1000 mg L⁻¹ standard containing the elements to be determined.

2.6. Papilloscopy

For fingerprint development, the powder technique was used with the aid of specific brushes (132LBW and CFB100, acquired from Sirchie®). The developed fingerprints were then photographed with a Canon EOS Rebel T6 18MP digital camera, with the close-up + 458 mm lens, a distance of 9 cm, a focus of 5.6, and the automatic ISO speed photo mode. For better visualization, three white LED lamps were used to improve the brightness with a black background under the fingermarks. The fingerprint images were processed in Adobe Photoshop. All the surfaces used for the deposition and development of fingerprints were washed with water and neutral soap after being photographed. All the photos taken were deleted after the publication of this work. The method of Pacheco et al. (2021) [28] was used for the deposition of natural and sebaceous fingerprints. Natural fingerprints are those that donors deposit after 30 min of washing their hands. For sebaceous prints, donors were asked to rub their thumbs on sebaceous parts of the face such as the forehead or nose. Natural and sebaceous fingerprints, a depletion series, and a comparison with standard Gold® powder acquired from Sirchie® were evaluated.

The evaluation of latent, natural, and sebaceous fingerprints from four donors, developed with copper and iron hybrid composites, was carried out according to the scale proposed by Sears et al. (2012) [37] (Table S1—Supplementary Information). This assessment was conducted by five independent analysts, all of whom hold Bachelor's degrees in Forensic Chemistry from the Federal University of Pelotas. To determine the final score, located to the left of the respective image in each corresponding figure, the most frequently assigned evaluation among the five analysts was chosen.

For SEM analysis, a Shimadzu SSX-550 Superscan was used. A glass slide was used as the substrate for the deposition of natural and sebaceous fingerprints, which were further developed with the Fe and Cu hybrid materials. The substrate was fixed with carbon tape, gold-coated, and analyzed at 15 kV. Secondary electron imaging was used, along with the following analytical parameters: AccV of 15.0 kV and a 4.0 probe.

3. Results

The influence of metals on the color of hybrid materials was observed, and the images are shown in Figure 1. It can be observed that curcumin (Figure 1B) and *Kappa*-carrageenan (Figure 1C) exhibit saffron yellow and whitish colors, respectively. Additionally, after the addition of *Kappa*-carrageenan in curcumin (Figure 1E), only the hue was altered, while the overall color remained unchanged, which agrees with the L^* , a^* , and b^* values, as indicated in Table S2 (Supplementary Information). In the presence of metals (Figure 1F,G), significant changes in the color of the materials were clearly noticed. As shown in Table S1 (Supplementary Materials), the a^* and b^* values for the copper hybrid material (a^* 9.10 and b^* 58.44) and the iron hybrid material (a^* 2.41 and b^* 11.98) decreased compared to the values obtained for the curcumin/*Kappa*-carrageenan mixture (a^* 16.46 and b^* 82.17), suggesting that the red and yellow components were reduced. Among the hybrid materials, the copper hybrid exhibited the highest red and yellow values, indicating that Cu^{2+} has a more significant role in enhancing the color of curcumin/*Kappa*-carrageenan. Additionally, the L^* values were reduced for the copper (L^* 43.92) and iron (L^* 14.33) hybrid materials, indicating that they exhibited a darker color compared to the curcumin/*Kappa*-carrageenan (L^* 63.40), which is consistent with reports from the literature [33]. Additionally, when determining the concentration of Cu and Fe in the hybrid materials, values of 13.31 and 13.51 mg g^{-1} , respectively, were obtained, thus confirming the presence of the metals in their respective hybrid materials.



Figure 1. (A) copper acetate; (B) curcumin; (C) *Kappa*-carrageenan; (D) iron chloride; (E) curcumin/*kappa*-carrageenan; (F) copper hybrid; (G) iron hybrid.

Figure 2A shows the UV–Vis spectrum of curcumin and the Cu and Fe hybrid materials. The curcumin exhibited maximum absorption at the wavelength of 425 nm and weak absorption at 262 nm [38]. The samples containing Cu and Fe exhibited more intense absorption bands, with a shift of approximately 6 nm [39]. Furthermore, bands at 419 nm and 255 nm were clearly observed for the Cu hybrid material, while peaks at 428 nm and 257 nm are observed for the Fe hybrid material. The crystal structure of samples was further investigated through XRD, and the results are presented in Figure 2B. The XRD of curcumin exhibits its typical crystal structure with intense peaks, especially at angles below 30 degrees, similar to what has been reported in other similar studies [40]. For the *Kappa*-carrageenan samples, an amorphous structure was observed, similar to that reported by other authors [41]. The amorphous behavior remained predominant for the hybrid

samples, where no peaks corresponding to the crystal planes could be identified. The FT-IR was used to identify the functional groups on the surface of the samples, and the results are displayed in Figure 2C,D. The infrared spectra of curcumin show a typical band at 3461 cm^{-1} corresponding to phenolic stretching (O-H). Thus, aromatic stretching (C=C) and stretching vibrations (C=C) were observed at 1629 cm^{-1} and 1509 cm^{-1} , respectively. Stretching vibrations (C=O) were observed at 1599 cm^{-1} , and bending vibrations (C-H) and aromatic stretching (C-O) vibrations were noticed at 1427 cm^{-1} and 1280 cm^{-1} , respectively. Finally, stretching vibrations (C-O-C) were observed at 1025 cm^{-1} [42]. The FT-IR of the Fe and Cu hybrid materials exhibit similar bands to those observed in curcumin. Furthermore, the vibration modes underwent shifts in their wavenumber, indicating changes in the vibrational state of curcumin upon binding to the metal ions [43,44].

TGA was performed on the hybrid materials, curcumin, and *Kappa*-carrageenan, and the results are shown in Figure 3. *Kappa*-carrageenan (Figure 3A) exhibited three stages of weight loss, which is consistent with previous reports [45]. The first thermal event occurred around $218\text{ }^{\circ}\text{C}$. The second and third thermal event occurred at approximately $531\text{ }^{\circ}\text{C}$ and $747\text{ }^{\circ}\text{C}$, respectively. The decomposition of curcumin began around $333\text{ }^{\circ}\text{C}$, with total degradation occurring around $575\text{ }^{\circ}\text{C}$, reaching 45.43%, which is in accordance with the literature reports. The TGA curve for Curcumin/*Kappa*-carrageenan (Figure 3B) shows three stages of mass losses of 12.43%, 14.29%, and 10.46% at temperatures of $207\text{ }^{\circ}\text{C}$, $342\text{ }^{\circ}\text{C}$, and $722\text{ }^{\circ}\text{C}$, respectively. Finally, two and three stages of mass loss were observed for the iron and copper hybrid materials, respectively. Both samples exhibited the greatest mass loss in the first thermal event, 17.25% and 18.12%, at temperatures of $198\text{ }^{\circ}\text{C}$ and $229\text{ }^{\circ}\text{C}$, respectively [33].

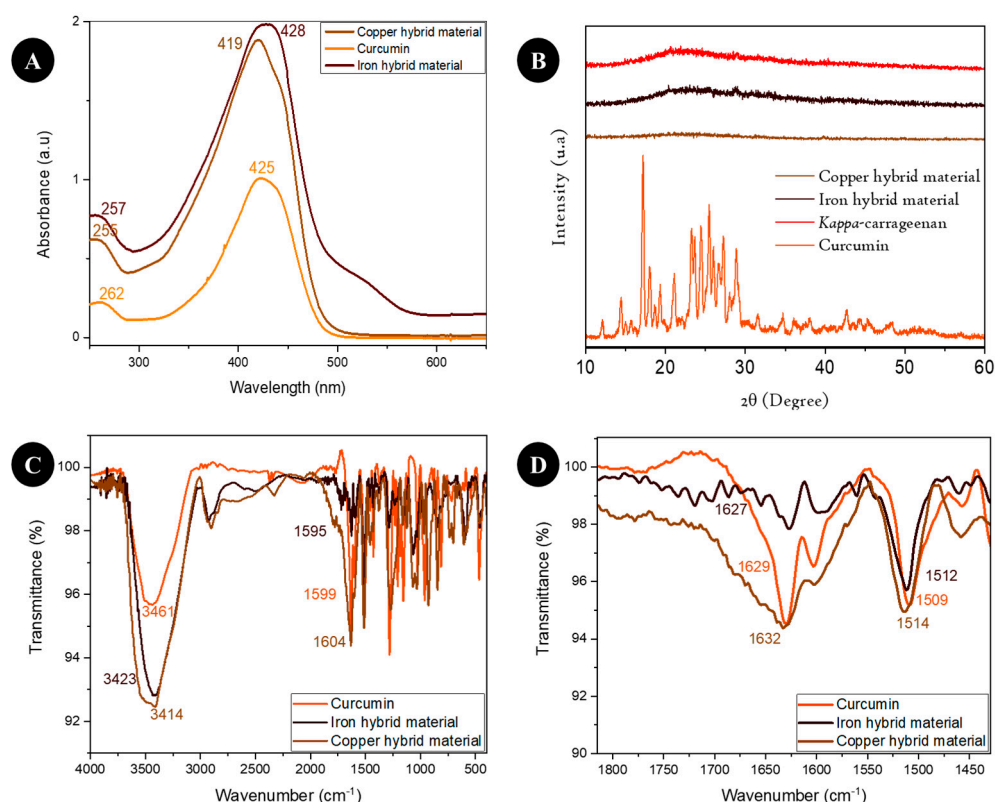


Figure 2. (A) UV-Vis spectra; (B) X-ray diffractogram; (C) FT-IR spectra; (D) zoomed-in FT-IR spectra.

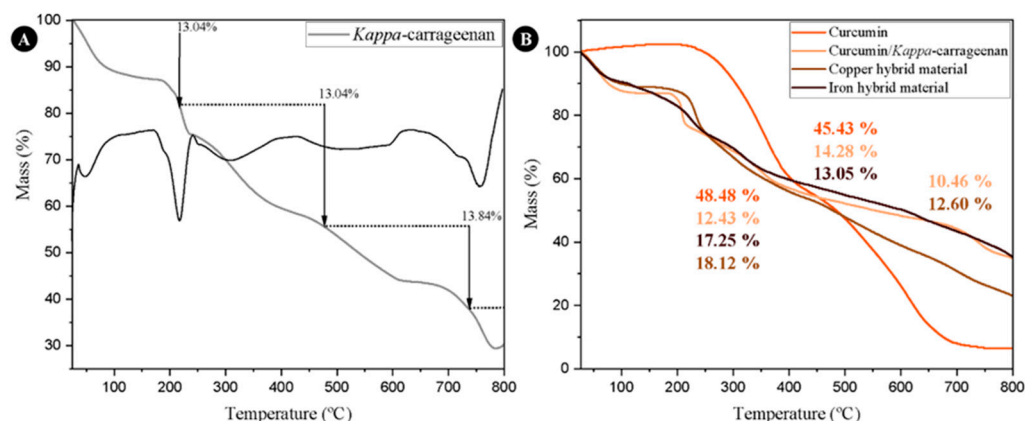


Figure 3. TGA curves of (A) *Kappa-carrageenan*, (B) curcumin, curcumin/*Kappa-carrageenan*, and hybrid materials.

In the analysis of curcuminoids in a commercial sample of natural curcumin, three distinct peaks were observed, as shown in Figure S1 (Supplementary Information). Peak 1 has a retention time of 2.957 min, Peak 2 has a retention time of 3.103 min, and Peak 3 has a retention time of 3.263 min. To study curcumin and hybrid material degradation, the samples were injected into the HPLC at different time intervals starting from the moment of dissolution. During this period, the samples were incubated in clear glass vials and left in an unprotected lit area throughout the analysis period. Figure 4A shows the chromatograms of the curcumin solution at different time points, while Figure 4B presents the chromatogram of the curcumin solution after 24 h, where the presence of more than one peak can be noted. In Figure 4C,D, the chromatograms of copper and iron hybrid materials, respectively, are shown. It can be observed that new peaks were not detected after 24 h.

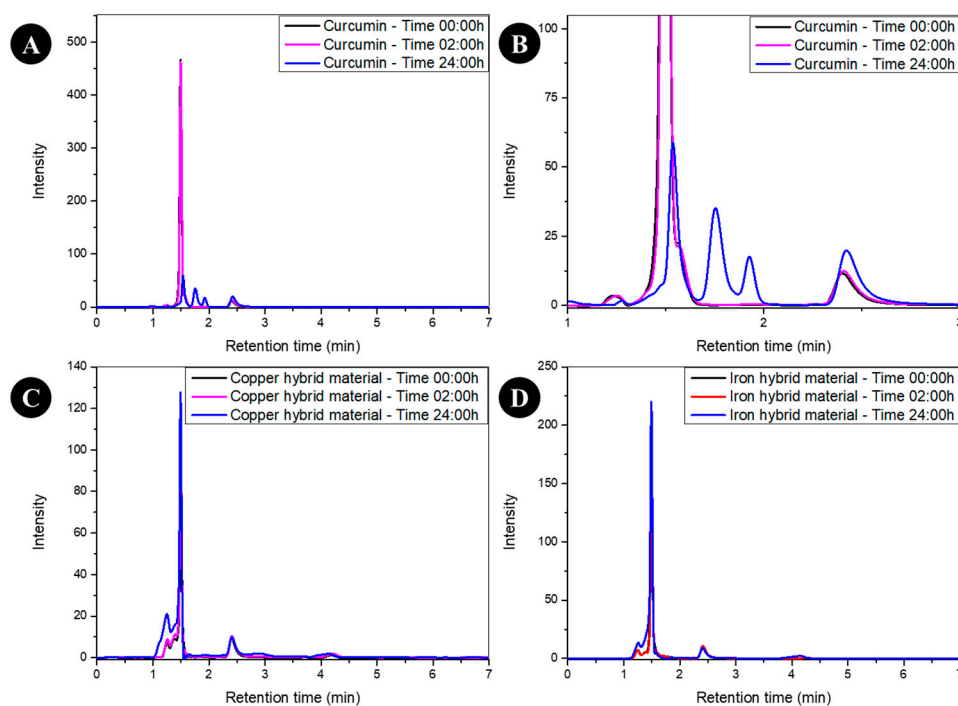


Figure 4. (A) Chromatogram of the analyzed curcumin solution at different time intervals; (B) enlargement of the curcumin chromatogram at the 24:00 h time point; (C) chromatogram of the analyzed copper hybrid material solution at different time intervals; (D) chromatogram of the analyzed iron hybrid material solution at different time intervals.

In the development of fingerprints using composites generated from hybrid materials, an initial study was conducted with a variety of donors. To convert the visual appearance of the fingerprints developed with powders into numerical values, recognizing that the researchers in this study were not fingerprint identification experts, a scoring system was employed instead of relying solely on the detection and counting of minutiae. This scoring system has been in use for over 35 years. The fingerprint evaluation scheme is presented in Table S1 (Supplementary Information). Cu and Fe composites were then applied to develop fingerprints using the physical powdering method on a glass surface. Figures 5 and 6 show the natural and sebaceous fingerprints from four donors, along with their respective scores.

Figures 7 and 8 represent a depletion series from donor 3, developed with copper and iron composites. In Supplementary Figures S2–S7, the depletion series from the other donors developed with both composites can be observed.

Figure 9 shows the same fingerprint (natural and sebaceous) developed on the left with composites and on the right with commercial Gold[®] powder. Supplementary Figures S8 and S9 display the development achieved for the same natural and sebaceous fingerprints from the other donors, using both composites and the commercial powder.

The SEM images (Figure 10) provided a detailed analysis of the development of latent fingerprints using both composites. Magnifications of 40 $\times$, 600 $\times$, and 2000 $\times$ were applied to investigate the minutiae of the fingerprints, the interaction of the particles with the surface, and the shape and size of the particles. For this analysis, latent fingerprints, both natural and sebaceous, were deposited on glass surfaces and developed with Cu and Fe composites. These magnification levels offered a more precise view of the particle adhesion to the ridges of the fingerprints and the effectiveness of the composites in developing the fingerprints.

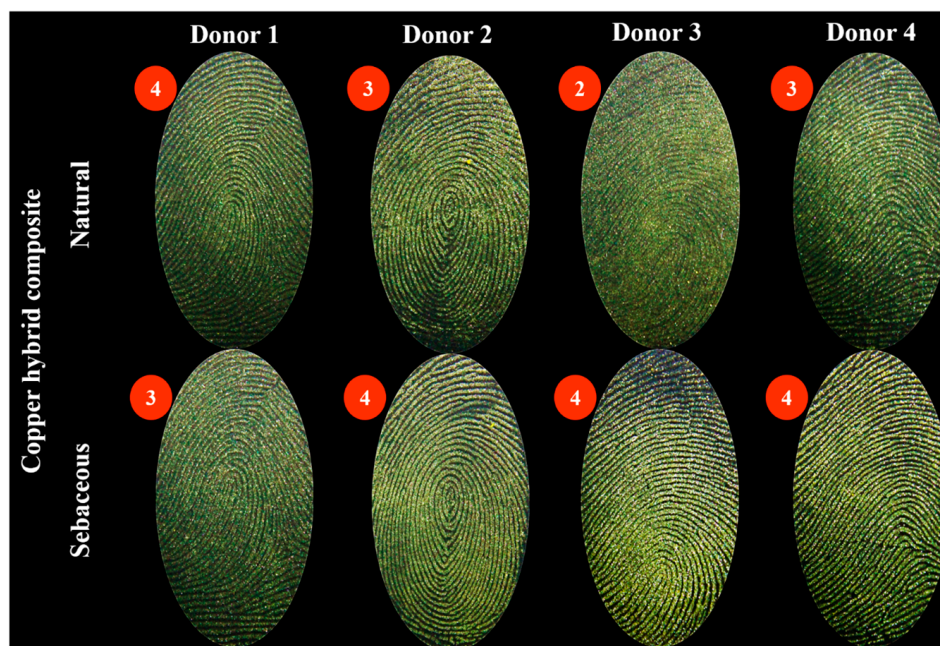


Figure 5. Natural and sebaceous fingerprints developed with the Cu hybrid composite and their respective scores.

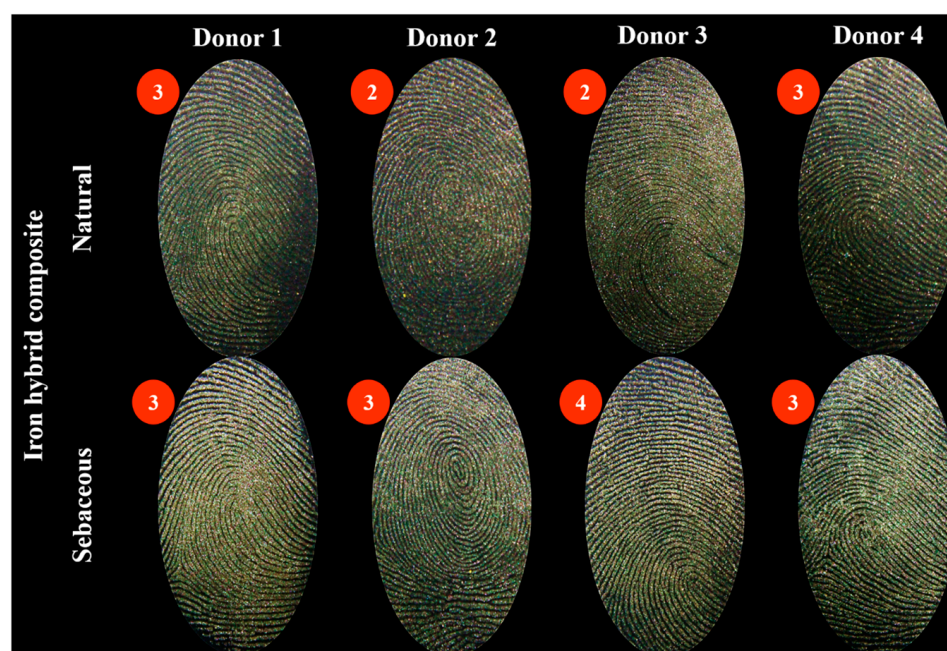


Figure 6. Natural and sebaceous fingerprints developed with the Fe hybrid composite and their respective scores.

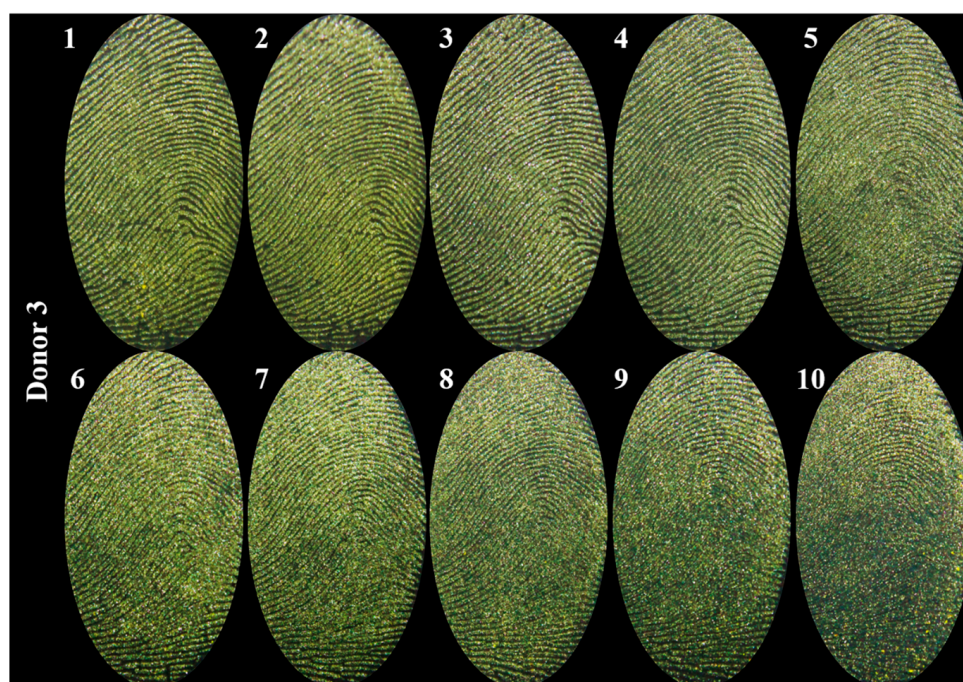


Figure 7. Depletion series developed with the Cu composite from donor 3.

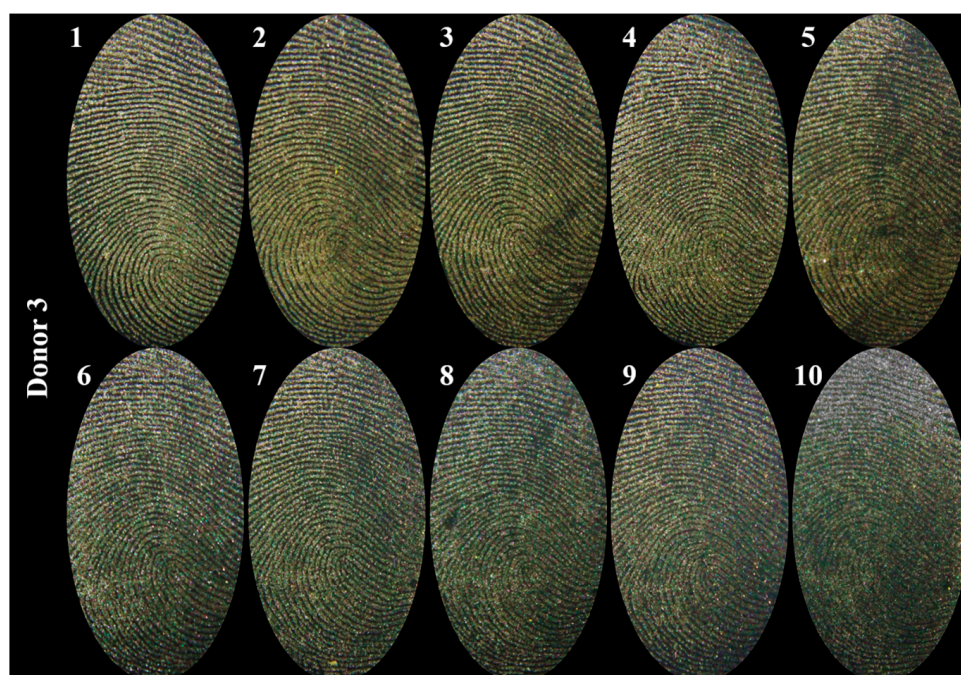


Figure 8. Depletion series developed with the Fe composite from donor 3.



Figure 9. Half-and-half fingerprint (natural (N) and sebaceous (S)) from donor 4, with the left half developed using composites and the right half developed with Gold[®] commercial powder purchased from Sirchie[®].

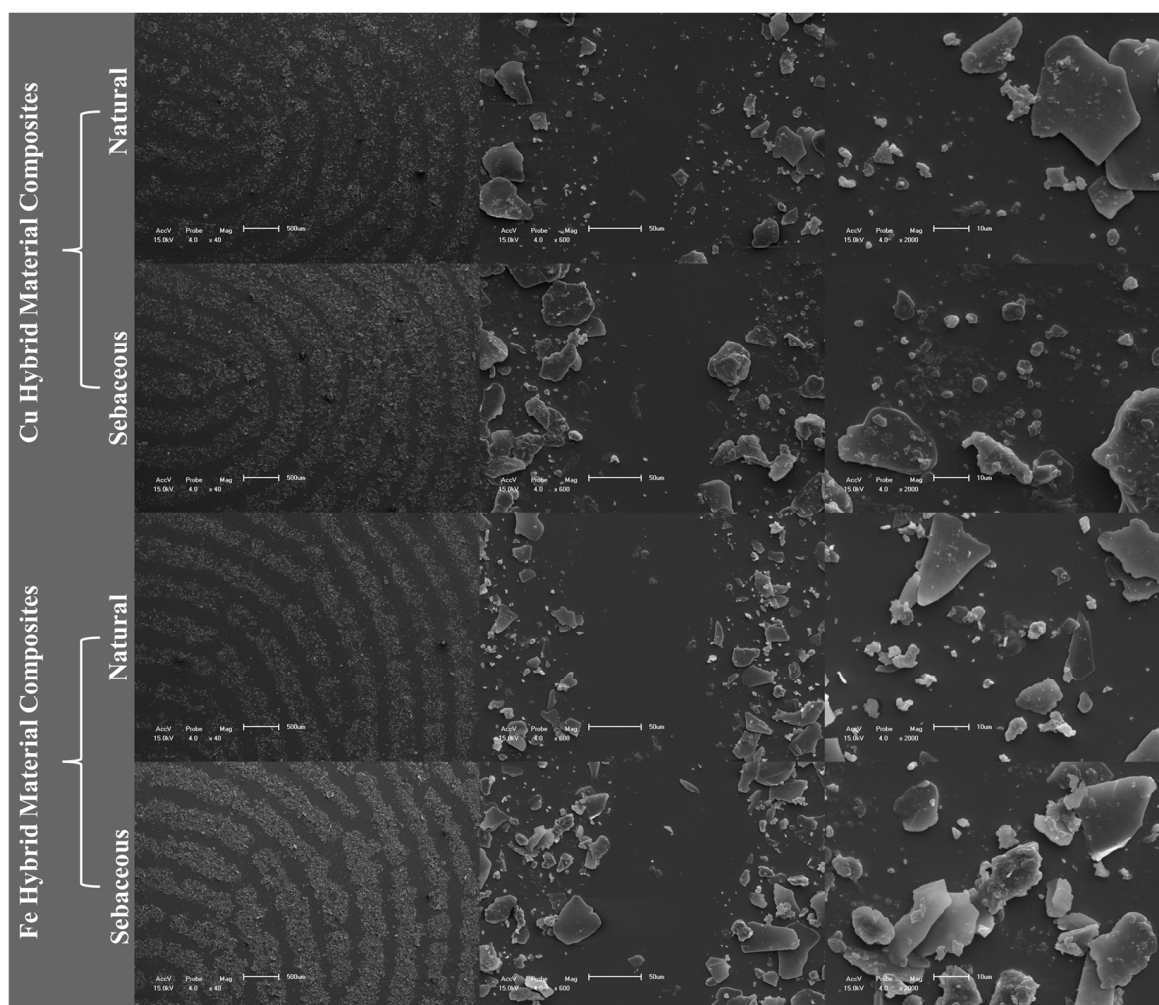


Figure 10. SEM analysis of natural and sebaceous latent fingerprints developed with Cu and Fe composites on a glass surface.

4. Discussion

According to the literature, it is known that the conjugated β -diketone portion of curcumin is a strong chelating agent, capable of interacting with several metal ions through a coordination bond, which can modify the color of the organic phase of curcumin [46,47]. Thus, the colorimetry results indicated the effective formation of hybrid materials, evidenced by the color change after the addition of these metals and confirmed by their concentration found in the materials.

Thus, the UV-Vis, X-ray, and FT-IR results suggest that the one-pot synthesis was efficient in forming hybrid materials using *Kappa*-carrageenan as a reducing agent. A strong absorption in the 400–550 nm range of the UV-Vis spectrum (Figure 2A) was observed for all samples, corresponding to the blue and green light region, which agrees with the colorimetric results. The shift in wavelength from curcumin to the hybrid materials suggests an interaction between the carbonyl group of curcumin in metal coordination [39]. The XRD analysis (Figure 2B) of curcumin confirmed its typical crystalline structure [40], while both *Kappa*-carrageenan and the hybrid materials exhibited amorphous structures. FT-IR was used to identify the functional groups on the surface of the samples, and the results are displayed in Figure 2C,D. The infrared spectra of curcumin show a characteristic band at 3461 cm^{-1} corresponding to phenolic stretching (O-H). The aromatic stretching of (C=C) and stretching vibrations of (C=O) were observed at 1629 cm^{-1} and 1509 cm^{-1} , respectively. The stretching vibrations of (C=O) were observed at 1599 cm^{-1} , while the

bending corresponding to (C-H) and the aromatic stretching of (C-O) were noticed at 1427 cm^{-1} and 1280 cm^{-1} , respectively. Finally, stretching vibrations related to (C-O-C) were observed at 1025 cm^{-1} [42]. The FT-IR of the Fe and Cu hybrid materials, shown in Figure 2C,D, exhibited similar bands to those observed in curcumin, with shifts in wavenumber indicating changes in the vibrational state of curcumin upon binding to the metal ions [43,44].

The thermal stability of hybrid materials is crucial in their practical applications. *Kappa*-carrageenan exhibited three thermal events (Figure 3A): the first corresponded to moisture loss, while the second and third events were related to the decomposition of the carbohydrate skeleton and complete degradation. Curcumin began with the dehydroxylation process, followed by its total degradation. The curcumin/*Kappa*-carrageenan composite showed similar behavior to pure *Kappa*-carrageenan (Figure 3B). On the other hand, the hybrid materials displayed differences due to the varying amounts of curcumin present in the samples. Thus, the TGA curves indicate that the hybrid materials possess superior thermal stability to pure curcumin [34].

The presence of three peaks in the chromatogram (Figure S1—Supplementary Information) can be explained by the composition of commercial curcumin (65%), which contains two other curcuminoids: desmethoxycurcumin and bisdemethoxycurcumin [48], representing the remaining 35% of the sample. Thus, the method adapted proved to be efficient, as the three peaks did not overlap. In addition, it provided a fast analysis time of 5 min [33]. The curcuminoid diferuloylmethane, commonly known as curcumin, is the largest fraction present in the sample, as curcumin was acquired at 65% purity. Therefore, the peak referring to the retention time of 3.263 min, which has the greatest area and intensity, represents curcumin. Desmethoxycurcumin is shown by the second largest fraction of the sample and was identified with a retention time of 3.103 min. Finally, bisdemethoxycurcumin, which denotes the smallest fraction of the sample, was identified with a retention time of 2.957 min. Several reports describe the same order of curcuminoids, with bisdemethoxycurcumin being the first peak and curcumin the last peak [49,50], and no interference in the formation of hybrid materials was found, since all curcuminoids contain the β -diketone fraction responsible for binding the metals. The absence or presence of methoxy groups (the only difference between the curcuminoids) does not influence the process, as they remain intact after the synthesis of the hybrid materials [18].

The instability of curcumin has been investigated in previous studies, where curcumin was dissolved in ethanol [42] or in phosphate-buffered saline (PBS) [16]. Under these conditions, degradation products such as vanillin, vanillic acid, and ferulic acid were identified [18,51]. In particular, curcumin degradation was detected after 24 h of incubation, observed by a reduction in the corresponding peak height, as previously reported [16]. Figure 4A,B clearly illustrate the decrease in curcumin peak intensity after 24 h, as well as the appearance of new peaks corresponding to degradation products with retention times longer than that of curcumin, consistent with the findings of Kumar et al. (2021) [16], as shown in Figure 4B. In contrast, no degradation products were observed in the hybrid materials. Figure 4C,D show the same intensity and peak behavior at all analyzed times, corroborating the results of Kumar et al. (2021) for curcumin-based metal complexes [16].

The use of powders to develop fingerprints is crucial in addressing the limitations of curcumin, especially in terms of stability. The previous results demonstrated that the combination of curcumin with Cu and Fe metals led to a significant increase in the stability of samples, which is one of the key requirements for practical use [52,53]. Based on these results, the potential of these compounds as developers for latent fingerprints was investigated. Composites were applied to fingerprints from various donors, aiming to capture variations related to age, sex, and other characteristics. It is known that factors such

as occupation, health (both physical and mental), diet, and medications directly affect the fingerprint composition. Both natural and sebaceous fingerprints were evaluated to assess interactions with the water-soluble and lipid-soluble compounds [24,54]. Through the method of Sears et al. (2012) [37], the visual appearance of the fingerprints was converted to numerical values for classification. The results demonstrated that both composites were effective in developing natural and sebaceous fingerprints from all donors, resulting in clear images on glass surfaces. The composites adhered to the fingerprints without having adhesion to the glass, providing good contrast in the images (Figures 5 and 6). In numerical values, the lowest scores, such as zero (no evidence of mark) and one (evidence of contact without ridge details), were not assigned to any fingerprint developed with the composites.

Only one natural fingerprint from the Cu hybrid composite (donor 3) (Figure 5) and two from the Fe hybrid composite (donors 2 and 3) (Figure 6) showed limited development, with about 1/3 of the ridge details visible (score 2). The Cu hybrid composite showed a high level of development for natural fingerprints, with complete ridge details (score 4) for donor 1 and strong development, showing between 1/3 and 2/3 of ridge details (score 3), for donors 2 and 4 (Figure 5). In the case of the Fe hybrid composite, natural fingerprints exhibited strong detail (score 3) for donors 1 and 4 (Figure 6). The Cu composite (Figure 5) more efficiently developed sebaceous fingerprints than natural ones, with a score of 4 for donors 2, 3, and 4 and a score of 3 for donor 1. Thus, all sebaceous fingerprints developed with this powder showed very strong or strong detail. Similarly, the Fe composite (Figure 6) also showed superior performance in sebaceous fingerprints compared to natural ones, with a score of 3 for donors 1, 2, and 4 and a score of 4 for donor 3, indicating well-defined fingerprints with strong or very strong detail levels. These findings indicate that, although the same deposition and development method was applied consistently, the influence of the donor on the development process was evident. Nevertheless, the results were consistent and satisfactory, with the Cu composite showing higher contrast and better scores compared to the Fe composite.

To evaluate the sensitivity of the composites in developing fingerprints, depletion series were performed. These series consist of successive contacts of the same finger with the surface, resulting in a progressive reduction in the amount of residue left with each new contact. Thus, the effective development of fingerprints left in the final contact indicates higher sensitivity in the developing powder, as there is less material available for adhesion [37]. Figures 7 and 8 present the depletion series for donor 3, developed with Cu and Fe composites, respectively. Although the Cu composite showed better performance with the previously discussed fingerprints, when comparing the depletion series for donor 3, greater clarity was observed in the revelations made with the Fe composite. This composite demonstrated good development, with excellent sharpness and contrast in most deposits, except for the tenth, where there was a significant decrease in quality. On the other hand, the Cu composite showed better clarity in the first four deposits, but from the fifth onward, there was a progressive decline in the quality of development. This decline may be attributed to the higher contrast provided by the Cu composite, which could have hindered the clarity of the fingerprints in deposits with less sebaceous residue. Similar results were observed for the other donors in the depletion series, as shown in Supplementary Figures S2–S7. Although the Fe composite offers lower contrast compared to Cu, it demonstrated superior sharpness in the depletion series for all donors, often effectively revealing fingerprints up to the tenth deposit. In contrast, the Cu composite showed satisfactory development, with good results up to the fourth deposit, followed by a progressive decline in quality from the fifth onward.

It is important to consider that fingerprints exhibit inherent variability in their composition, including factors such as chemical composition, the amount of material deposited,

and the pressure applied during deposition. To minimize the impact of these variables on fingerprint development, the use of half-and-half fingerprint tests is an effective approach. In this method, fingerprints are deposited and physically split along the centerline, allowing each half to be developed using different techniques or powders (powdering method) [37]. This “half-and-half” strategy, applied to the comparative analysis between synthesized powders and commercially available powders, is particularly relevant as it allows the evaluation of whether the developed material performs similarly to those currently used by the Federal Police [32]. Figure 9 illustrates the natural and sebaceous fingerprints of donor 4, developed using the half-and-half method. The left halves were developed with Cu or Fe composites, while the right halves were developed with the commercial Gold[®] powder. Good development of natural fingerprints was observed with both composites, exhibiting a quality comparable to that of the commercial Gold[®] powder. In the case of sebaceous fingerprints, the Cu composite showed a performance similar to that of the commercial powder, while the Fe composite demonstrated superior sharpness compared to the commercial powder. Figures S8 and S9 (Supplementary Information) show the half-and-half fingerprints of the other donors. A similar behavior was observed in the development of the composites, with a performance equivalent to, or better than, that of the commercial powder. When comparing both composites, the Fe one stood out by providing greater sharpness, resulting in clearer definition of the fingerprint minutiae; this may be related to the weak magnetic cohesion that can favor the uniform distribution of particles in certain contexts, such as when applied to irregular surfaces. In the case of iron (Fe) composites, this property allows the material to be easily dispersed and adhere to other surfaces, which can result in greater-definition details when applied as a fingerprint developer. In contrast, materials with stronger magnetic cohesive forces tend to form agglomerates and may have a lower capacity for adhesion and homogeneous distribution [55,56].

So far, hybrid material composites have been evaluated as developers for latent fingerprints using various methods, all applied to glass surfaces. Considering that the surface on which a fingerprint is deposited can significantly influence the effectiveness of its development, the final analysis of these powders included tests on surfaces representative of those encountered in operational work. For this, the same composites were applied to different surfaces commonly found in forensic investigations [37]. Examples of these surfaces can be seen in Figure 11.

Figure 11 illustrates the development of latent fingerprints with both composites on porous and non-porous surfaces. Objects commonly found in real-world scenarios were selected, including glass bottles, metal knife surfaces, plastic knife handles, wooden door surfaces, metal doorknobs, and the glass screens of smartphones. Both composites were effective in enhancing latent fingerprints on both light and dark surfaces, as well as porous and non-porous ones, demonstrating the versatility of the developed powders. The revealed fingerprints exhibited sufficient detail to be used in human identification processes.

The SEM images (Figure 10) provide a detailed analysis of the minutiae in both natural and sebaceous fingerprints developed with both Fe and Cu composites. At 40× magnification, it can be observed that the Fe composite offers greater clarity compared to the Cu composite, which is directly related to the strong adhesion of the Fe composite to the fingerprint compounds, while interacting minimally with the glass surface, as evidenced in the 600× magnified images. Regarding morphology, both composites exhibit irregular shapes and heterogeneous particle sizes [28], ranging from 50 µm to less than 10 µm. Despite these characteristics, the composites were effective in developing latent fingerprints, providing sufficient detail for human identification.

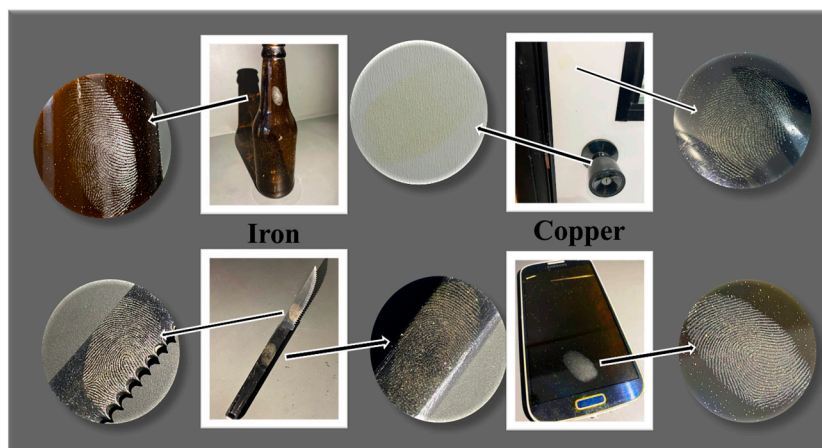


Figure 11. Development of latent fingerprints using Cu and Fe composites on different objects representing porous and non-porous surfaces: glass bottle, doorknob and door, knife and knife handle, and a black smartphone.

5. Conclusions

The hybrid copper and iron materials were synthesized via a one-pot methodology using natural curcumin and *Kappa*-carrageenan as a reducing agent. These materials were characterized by different techniques such as FT-IR, UV-Vis, XRD, and FAAS. The observed color changes and enhanced stability of these materials can be attributed to the coordination of Cu^{2+} or Fe^{3+} metal ions with curcumin, as well as potential interactions with *Kappa*-carrageenan. To verify whether curcumin hybridization effectively improved its stability, thermogravimetric analysis was performed, showing that the hybrid materials had greater thermal stability than curcumin. Solution stability studies using liquid chromatography revealed that curcumin showed degradation product peaks after 24 h of solvent exposure, whereas the hybrid materials remained stable after 24 h. The hybrid composites were further applied as latent fingerprint developers. The results showed that both materials effectively developed natural and sebaceous fingerprints from various donors, with good clarity and contrast on glass surfaces. In successive contact depletion series, the Fe composite demonstrated higher sensitivity, revealing sharp fingerprints up to the tenth contact, while the Cu composite showed lower quality after the fifth contact. In comparative tests with commercial Gold[®] powder, both hybrid materials performed similarly, with the Fe composite standing out for its fingerprint sharpness. Additionally, when applied to surfaces representative of forensic scenarios (such as glass bottles, metal doorknobs, knife surfaces, and smartphones), the composites were versatile, revealing sufficient fingerprint details for human identification on both porous and non-porous surfaces. Overall, these findings highlight the potential of hybrid materials to enhance forensic applications and contribute to the sustainable use of natural resources.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/colorants4010003/s1>, Table S1: Classification scheme used for the evaluation of developed fingerprints; Table S2: Color parameters of reagents and hybrid materials; Figure S1: Chromatogram of commercial natural curcumin; Figure S2: Depletion series developed with the Cu composite from donor 1; Figure S3: Depletion series developed with the Fe composite from donor 1; Figure S4: Depletion series developed with the Cu composite from donor 2; Figure S5: Depletion series developed with the Fe composite from donor 2; Figure S6: Depletion series developed with the Cu composite from donor 4; Figure S7: Depletion series developed with the Fe composite from donor 4; Figure S8: Half-and-half fingerprint (natural (N) and sebaceous (S)) from donors 1, 2, and 3, with the left half developed using Cu composite and the right half developed with

Gold® commercial powder purchased from Sirchie; Figure S9: Half-and-half fingerprint (natural (N) and sebaceous (S)) from donors 1, 2, and 3, with the left half developed using Fe composite and the right half developed with Gold® commercial powder purchased from Sirchie®.

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