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Micronised Egyptian blue pigment: A novel near-infrared luminescent fingerprint dusting powder



PIGMENTS

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1. Introduction

The detection of latent fingerprints is extremely useful in forensic investigations to establish evidence of contact between the criminal, the victim, and/or the crime scene [1-3]. To that end, a wide range of chemical and physical methods have been developed to aid in their detection and recovery, the choice of which is dependent on the nature of the surface involved [1,2]. The most common approach to detecting latent fingerprints on non-porous items has been the use of dusting powders, which work through physical adhesion to the latent fingerprint deposit [1-5]. It has been estimated that in the United Kingdom, approximately 50% of the 60,000 fingerprint identifications per annum arise from powdered fingerprints [1]. Historically, a wide range of materials have been utilised as fingerprint powders; including carbon black, iron oxide-based magnetic powder and titanium dioxide; in attempts to achieve improved visualisation across a wide variety of surfaces and conditions [1–5]. In recent times, this has included attempts to use natural products such as turmeric as safe and inexpensive powders [6]. Several commercially available powders exhibit luminescence in the visible region of the electromagnetic

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ABSTRACT

In this paper we demonstrate that micronised Egyptian blue pigment can be used as a safe and simple material to visualise latent fingerprints on non-porous surfaces as near-infrared luminescent impressions. This allows the detection of latent fingerprints on highly patterned and reflective surfaces that have proven challenging with existing techniques.

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spectrum, which to some extent can negate the interferences exhibited by highly patterned or coloured surfaces [1,2,4]. However, there remain many fluorescent, highly patterned and/or reflective surfaces that continue to prove troublesome [7–9].

An alternative approach is to use dusting powders that exhibit near-infrared (NIR) luminescence [10–12]. As very few substrates luminesce in the NIR region of the spectrum, such powders can highlight ridge detail while avoiding interference caused by inherent background luminescence [9–13]. To date there are limited examples of such powders in the open literature. Chadwick and co-workers reported an NIR luminescent fingerprint powder based on adsorption of an organic dye (STaR 11[®]) on finely powdered aluminium oxide, mixed with commercial magnetic fingerprint dusting powder. However, the developed impressions were still affected by underlying patterns on the surface, and the luminescence intensity of the developed fingerprints was observed to decrease over time. In addition, the powder itself had a limited shelf life [10]. More recently, King et al. have demonstrated that a biorganic powder based upon an algae, spirulina platensis, can be used to develop fingerprints with imaging in the NIR region [11].

Egyptian blue (also known as cuprorivaite) is the earliest known synthetic pigment; first prepared in ancient Egypt around 3600 BCE and used extensively until the 4th century CE [14–18]. Qualitative studies by Sir Humphrey Davy and others revealed that the pigment was likely to be copper-based, and contained large



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proportions of silica and calcium oxide [16,18–20]. Subsequent evidence from X-ray diffraction (XRD) established its primary composition to be calcium copper silicate (CaCuSi₄O₁₀) [21,22]. Researchers have demonstrated that Egyptian blue exhibits strong photoluminescence in the NIR region when excited at around 630 nm, with a quantum yield of 10.5% at the maximum emission of 910 nm [23–26]. Egyptian blue is also very stable, with painted artefacts dating several thousand years old still showing strong NIR luminescence [18,26]. The high stability to light, oxygen, pH and temperature of Egyptian blue, as well as its long luminescence lifetime, have been noted in a recent application to the development of novel optical sensors [27]. These characteristics led us to investigate Egyptian blue as a NIR luminescent latent fingerprint dusting powder for highly patterned surfaces.

2. Materials and methods

2.1. Materials

A single source of Egyptian blue pigment (Rublev Colours, CA) was used for this research. The following commercially available fingerprint dusting powders were used: Velvet Black, titanium dioxide, Blitz Red[®] fingerprint powder (Criminal Research Products, LLC), and bichromatic powder donated by the Western Australia Police Fingerprint Bureau. The following substrates were used during the course of the study; white glazed porcelain bathroom tiles, aluminium soft drink cans and various glass surfaces.

2.2. Collection of fingerprint specimens

Latent fingerprints were collected from male and female donors (aged between 19 and 25 years) on a variety of substrates. Two varieties of fingerprints were deposited; charged and uncharged. Charging is achieved by rubbing the tips of the fingers across the nose and forehead to transfer sebaceous lipids to the contact ridges of the fingers before touching the substrate [28,29]. Uncharged fingerprints were obtained by simply touching the substrate.

2.3. Micronising process

A McCrone micronising mill was used to micronise the Egyptian blue. This uses 48 corundum (crystalline alumina) cylindrical grinding pellets held in a plastic screw-topped jar. A slurry was made of the pigment (around 1 g) in ethanol (5 mL). This slurry was then added to the screw-topped jar containing corundum pellets. The jar was loaded horizontally into the mill and clamped into place, before micronising for the desired time period. Once micronising was completed, the jar was removed from the mill and its contents poured into an evaporating basin over low heat to evaporate off the solvent.

An estimate of the average particle size of the powders produced was obtained using optical microscopy. Particles in the recorded images were measured against the scale bar (included by the camera in each image) and recorded. If the particle measured was not approximately spherical, the average of the height and width of that particle was used. The average of all of the particles contained in the images was then calculated.

2.4. Application of fingerprint dusting powders and micronised Egyptian blue

Fingerprint powders and micronised Egyptian blue were applied using fingerprint brushes donated by the Western Australia Police Forensic Division. The two types of brushes used were based on natural fibre (camel hair) and synthetic fibre (fibreglass).

2.5. Luminescence spectroscopy

Uncorrected steady state emission and excitation spectra were recorded on an Edinburgh FLSP980-S2S2-stm spectrometer equipped with a 450 W xenon arc lamp and a Hamamatsu R5509-42 photomultiplier. Emission and excitation spectra were corrected for source intensity (lamp and grating) and emission spectral response (detector and grating) by a calibration curve supplied with the instrument.

2.6. X-ray diffraction measurements

XRD was performed using a Bruker D8 X-ray diffractometer using a position sensitive detector (PSD). The range examined was $7.5^{\circ}-90^{\circ}$ with an interval of 0.0015° every 0.7 s.

2.7. Photography

A modified Canon 40D camera was mounted on a benchtop macro stand in a darkened room. Modification was made by removal of the internal infrared blocking filter that reduces the natural sensitivity of CCD and CMOS sensors to infrared radiation. The camera was then connected to a live feed television screen. This aided both focusing and reviewing of the images captured. The camera was fitted with a Canon EFS 18–55 mm lens and a lensmounted RM90 filter, which transmits radiation from approximately 900 nm. The lens-mounted filter was removed for photographs in the visible region.

Developed fingerprints on various substrates were illuminated either with a white LED square array (Camera Electronics, North Perth) or a forensic light source (Rofin Polilight PL500, white light setting). The light source was kept at a 45° incident angle to the substrate and a distance of 22 cm.

3. Results and discussion

Initial experiments were carried out with a commercial sample of Egyptian blue pigment, which was used without further treatment. Excitation and emission spectra of the pigment were obtained, which aligned well with literature values for both the excitation and emission characteristics of Egyptian blue [23,24]. (Fig. 1).

The blue colour of the material and its luminescent nature strongly suggested that this was indeed cuprorivaite, but chemical analysis by XRD was carried out to confirm that the commercial



Fig. 1. Excitation and emission spectra of commercial sample of Egyptian blue with associated electronic transitions.

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