Cyclododecane as a Contrast Improving Substance for the Terahertz Imaging of Artworks

John W. Bowen, Tom Owen, J. Bianca Jackson, Gillian C. Walker, John F. Roberts, Dominique Martos-Levif, Pauline Lascourrèges, David Giovannacci, and Vincent Detalle

Abstract— This paper presents measurements of the terahertz properties of the art conservation substance cyclododecane, demonstrating that it can act as a contrast improving agent in the terahertz imaging of concealed wall paintings. Results are presented which show that the terahertz optical properties of cyclododecane are dependent on the rate at which it has cooled from the melt. Based on the results, a theoretical explanation of the contrast enhancement mechanism is postulated. The findings presented here may lead to the development of novel coating materials that could improve the quality of terahertz images in a variety of fields, not just in art conservation.

Index Terms—Cultural heritage, terahertz imaging, terahertz spectroscopy.

I. INTRODUCTION AND MOTIVATION

CYCLODODECANE (CDD) is a wax-like cyclic hexane ($C_{12}H_{24}$) commonly used by art conservators as a volatile binding medium to consolidate and protect works of art with delicate, porous or friable surfaces during their transportation or cleaning [1],[2]. It is particularly useful for this purpose because in addition to being easily applicable, durable and water resistant, it is completely reversible and sublimates away at room temperature over the course of some weeks or months, leaving no residues or need for mechanical and chemical removal.

Recent work [3] has provided evidence that, in addition,

CDD may have the ability to increase contrast in terahertz images of some artworks, although until now the potential mechanism has remained unclear. This paper presents the results of an investigation of the terahertz properties of CDD, how they depend on the application method and associated rate of cooling, and, based on these, presents an explanation of the contrast improvement mechanism.

The application of terahertz imaging and spectroscopy to cultural heritage is a relatively new field [4], but has already demonstrated the potential to provide non-destructive information complementary to other imaging and analysis techniques on the structure and state of preservation of objects such as paintings [5], ceramics [6], manuscripts [7], wooden artifacts [8], and mummified remains [9]. Recent work has extended this list to include stone [10] and oxidized metal objects [9]. One area in which terahertz imaging is particularly advantageous is the imaging and analysis of historic wall paintings [11],[12]. Many wall paintings have, at some point in their history, been plastered or painted over, sometimes repeatedly, or have been repaired and repainted. As terahertz radiation can penetrate through overlying paint layers and plaster several millimeters in thickness, can show contrast between different pigments, and, if time domain reflection imaging is used, can differentiate and isolate individual layers within a multilayer structure, it provides a means of imaging concealed wall paintings which are beyond the reach of other imaging modalities.

The work presented in this paper was carried out in response to terahertz imaging experiments on a fragment of wall painting from the Louvre collection, which was undergoing conservation. The painting, "Les Trois Hommes Armés de Lances" (Fig. 1), is from the collection amassed by Giampietro Campana (1808-1880). As an initial part of the conservation process, the face of the painting was temporarily consolidated with CDD.

While being conserved, the painting was subjected to a number of analytical techniques. Although the painting has the appearance of an authentic Roman fresco depicting three men carrying spears, x-ray radiography confirmed that it is pieced together from smaller fragments of mortar bound in plaster. It is hypothesized that the mortar dates from the 1st C C.E., and the plaster from the 19th C C.E. While some of the visible

Fig. 1. Photograph of "Les Trois Hommes Armés de Lances" taken after conservation. The yellow box indicates the region corresponding to the terahertz images in Figs. 3(a) and 3(b). © LRMH

Fig. 2. Terahertz imaging set-up, taken before conservation was completed. © LRMH

painting, such as the men's heads, may date from the 1st C C.E., Fourier transform infrared spectroscopy (FTIR) and Energy-dispersive X-ray spectroscopy (EDS) indicated the presence of Prussian blue pigment at the surface, which only came into use after 1708. In addition, stratigraphy, EDS and FTIR, indicated that the first paint layer was painted using a fresco technique and then covered by other paint layers applied using a secco technique (pigments with a glue binder). Furthermore, it is known that the fragments were restored with retouching when they arrived in the Campana collection in the 19th C. Therefore, there is good reason to believe that 19th C plastering and over-painting may be covering earlier pigments in some regions of the painting.

Because of the suspicion that subsurface pigments may be present, terahertz reflection imaging was carried out on the painting. The terahertz images were recorded using a Picometrix T-Ray 4000 terahertz time-domain imaging system. A co-linear imaging head with a 25.4 mm focal length

was used to allow normal incidence reflection images to be obtained. The spot size at the focus was approximately 2 mm in diameter, heuristically optimized at the surface of the painting. The head was raster scanned across the painting surface using x-y translation stages, with a scanning step size of 1 mm. The time domain signals were recorded with a sampling interval of 0.078125 ps, with 10 waveform averages per pixel at a 5 pixel per second acquisition rate. A photograph of the set-up is shown in Fig. 2. Note that the painting in Fig. 2 has a noticeable, uneven, dull white coating, indicative of the presence of CDD.

Terahertz images of the painting were recorded twice: initially in early June, after the painting had been coated with CDD, and then again 3.5 months later. The initial imaging experiments revealed what appeared to be a face from a piece of original Roman fresco underneath the currently visible surface of the painting (Figs. 3(a) and 3(b)). The image is reconstructed from the peak maximum of the time-domain signal. The face is located underneath the toga of one of the spear-carrying figures and is of a comparable size and style to the faces of those figures. The image as presented in Fig. 3(b), also includes features from the surface painting; notably the bright linear bar-like features to the right-hand side of the image, which correspond to the folds of the toga.

When repeated three months later, the terahertz image of the sub-surface face displayed significantly worse contrast (Fig. 3(c)). Although the features of the face are still visible, the returned signal level is considerably reduced compared to the initial image in Fig. 3(b), the lower signal-to-noise ratio resulting in a granular appearance in the region of the face. However, in contrast, it should be noted that the signal strength from the features to the right-hand side of the image in Fig. 3(c) that originate from the surface painting of the folds of the toga, is comparable to that for the same features in the initial image in Fig. 3(b). Thus, over the course of the 3.5 months between recording the two images, the subsurface face showed a significantly reduced signal level and degraded image contrast, whereas the features due to the surface painting showed no such deterioration, ruling out the possibility that the difference between the two images is due to differences in the operation, set-up or alignment of the imaging system between the two dates. As the painting was coated with CDD prior to recording the first image, it is postulated that the CDD acted as a terahertz contrast enhancer which improved the terahertz transmission through the top layer of plaster and enhanced the underlying pigment contrast. By the time the second terahertz image was recorded, the CDD had sublimated, resulting in a lower contrast image of the subsurface regions.

The remaining sections of this paper discuss a series of measurements on the terahertz optical properties of CDD, carried out in an attempt to understand the mechanism behind the observed contrast improvement.

II. Sample Preparation and Measurements

CDD is available in two forms: as a bulk crystalline solid and as an aerosol spray. The solid form can be applied to

(a)

(b)

(c)

(a)   (b)   (c)

Fig. 4. Photomicrographs of cross-sections through the CDD samples showing the crystalline structure: (a) slow cooled, (b) medium cooled, (c) fast cooled.
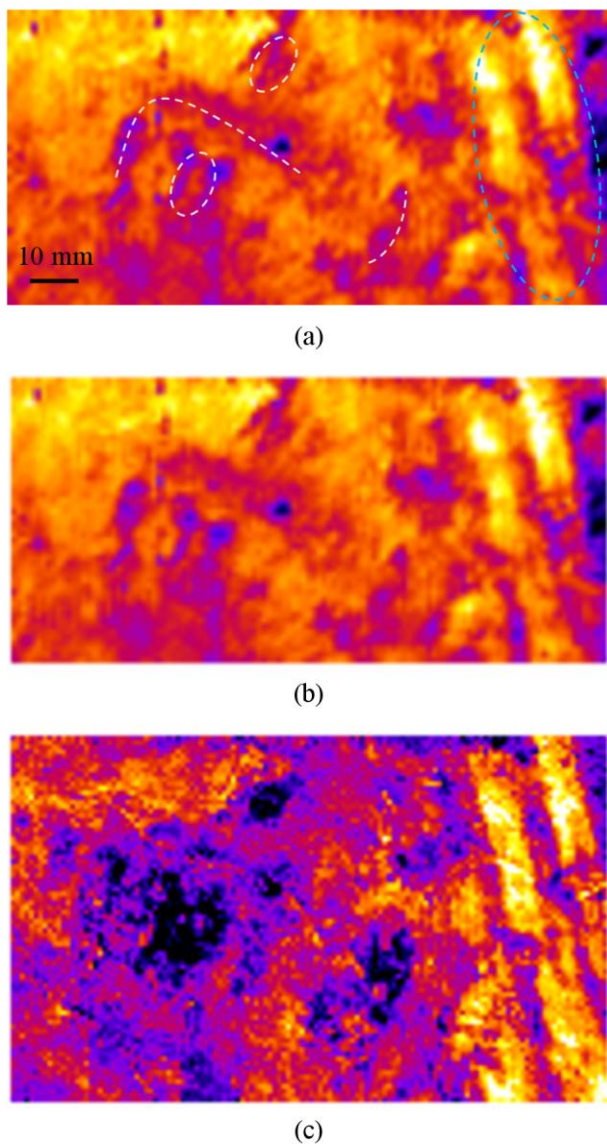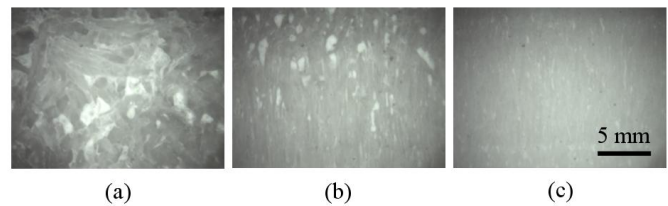
Fig. 3. Terahertz images of the region outlined by the yellow box in Fig. 1, reconstructed from the maximum peak in the time domain: (a) image recorded in early June, with white dotted overlay highlighting the main features of the face and blue dots highlighting the folds of the toga, (b) the same image as (a) but without the overlay, (c) image including the same region but recorded 3.5 months later. In the color scale used, bright yellow corresponds to regions with the highest amplitude, scaling through purple to black for regions of lowest amplitude.

objects either as a melt, using a heated spatula, heated spray gun or brush, or as a solution, dissolved in a non-polar solvent such as octane, naptha or white spirit. The method of application affects not only the density and thus the hydrophobicity of the resulting film, and the depth to which it penetrates into a porous surface, but is also a factor, along with environmental temperature and humidity, in determining how rapidly the film sublimates. Application as a melt is thought to produce the densest film [2]. The CDD applied to "Les Trois Hommes Armés de Lances" was done so as a melt.

As part of the conservation process, a protective paper was applied on top of the CDD using Paraloid B72 in ethanol/acetone. Prior to the terahertz imaging, the paper and Paraloid had been removed using a solvent, and the CDD had been partially thinned (mechanically and with air ventilation), leaving a layer less than 1 mm thick on the plaster.

In order to obtain reliable measurements of the terahertz optical constants of CDD, relatively large, free-standing, parallel-faced, slab-like samples of pure CDD were required. This ruled out their production by the aerosol and solvent-based approaches, as these are only suitable for the production of thin films of CDD on a supporting substrate. Therefore, the samples were produced by pouring molten CDD into aluminium moulds and allowing them to set. The melting point of CDD is around $58 - 61$ °C. The sample dimensions were 110 mm $\times$ 110 mm by between 20 mm and 25 mm thick.

Since CDD forms different sized crystals depending on how rapidly it is cooled, three different samples were created (Fig. 4). The first was allowed to cool naturally at room temperature. This is the "medium-cooled" sample. The second sample was cooled as rapidly as possible using liquid nitrogen ("fast cooled"), and the third was cooled as slowly as possible by placing it in a warm oven and then turning the oven off with the door closed ("slow cooled").

The optical constants of the samples were determined using dispersive Fourier transform spectroscopy (DFTS). This was accomplished using a hybrid Mach-Zehnder/Martin-Puplett interferometric spectrometer, the optical layout of which is illustrated in Fig. 5. The DFTS technique allows direct measurement of the frequency dependent amplitude and phase insertion loss of the sample in transmission, from which the frequency dependent complex refractive index and absorption coefficient can be determined directly [13]. The DFTS instrument used in this study has a frequency independent beam-waist size of 26 mm at the sample. While this large beam-waist requires relatively large samples (lateral dimensions of at least three times the beam-waist size, in order to avoid beam truncation effects), it minimizes the errors in optical constant determination that can arise in time-domain spectroscopy systems with small beam-waists at the sample location [14]. The instrument incorporates a high-pressure mercury arc lamp as the terahertz source, and an InSb hot electron bolometer as the detector, covering the frequency range from 100 GHz to 1 THz. The measurements presented in this paper were recorded at a frequency resolution of 3 GHz.
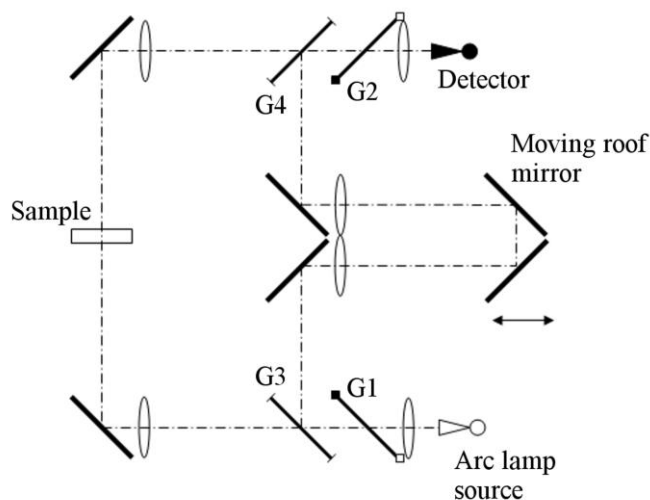
Fig. 5. Optical layout of the dispersive Fourier transform spectrometer. G1 and G2 are wire grid polarizing beam-splitters with wires at 45° to the plane of the instrument. G3 and G4 are wire grid polarizing beam-splitters with wires parallel to the plane of the instrument. The bold lines represent plane mirrors and elliptical shapes represent lenses used for beam control. The dot-dashed line indicates the optic axis.

III. RESULTS

Fig. 6 shows the real part of the refractive index and absorption coefficient of the samples over the frequency range for which the signal-to-noise ratio allows their reliable extraction. For both parameters, there is clearly a significant dependence on the rate at which the samples cooled from the melt. In the case of the absorption coefficient (Fig. 6(b)), the slow cooled sample shows the highest absorption, while the fast cooled sample is the least absorbing. This can be attributed to a combination of two processes: internal scattering within the polycrystalline samples, and crystal polymorphism.

Internal scattering within the samples can occur at the crystal boundaries. As there are many scattering events that can occur as the radiation propagates through each sample, the path length taken by the multiply scattered radiation will be greater than in a sample with negligible internal scattering. Consequently, because of this extended path length, the radiation is subject to greater attenuation due to the basic material absorption. This results in an effective increase in the absorption coefficient of the sample. Crystal size-dependent effects of this sort have been observed in other granular samples in the terahertz range [15].

The degree of scattering is dependent on the size of the crystals in each CDD sample relative to the wavelength. The largest crystals in the slow-cooled sample are easily visible with the naked eye, at around a millimeter along their shortest axis, while there are no crystals visible by eye in the fast-cooled sample, suggesting that any crystals are less than 40 micrometers in size. This range between millimeters and tens of micrometers is comparable in scale to the range of wavelengths in the terahertz range, and so the precise sizing of the crystals has a large effect on the optical properties of
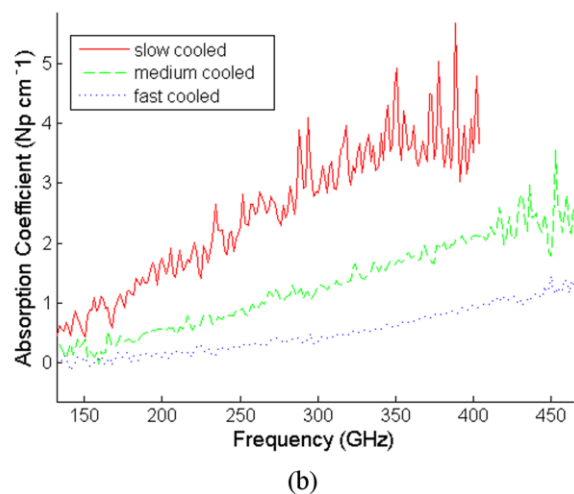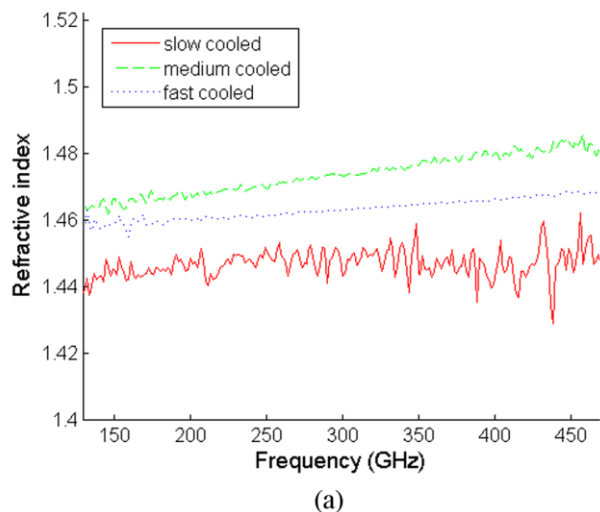

(a)

(b)

Fig. 6. (a) Refractive index and, (b) absorption coefficient of CDD samples.

cyclododecane in the terahertz range. In the fast cooled sample, the crystal size is small compared to the wavelength, so that there is little scattering of the radiation at the crystal boundaries. In contrast, in the slow cooled sample, the crystal size is comparable to or greater than the wavelength, resulting in significant internal scattering and thus increased absorption. The medium cooled sample has a crystal size intermediate between the other two, resulting in an intermediate level of internal scattering and absorption. Only the fast cooled sample has a small enough crystal size to place it in the Rayleigh scattering regime, but it is clear from Fig. 6(b) that its absorption coefficient does not follow the fourth power dependence on frequency expected of Rayleigh scattering, and so the frequency dependence of the material absorption coefficient must also play a significant role.

The second process that may contribute to the observed difference in optical constants for the different samples is crystal polymorphism. It is know that many materials exists in more than one crystalline form (polymorphs), leading to different chemical and physical properties. In the terahertz range, different polymorphs can exhibit different vibrational
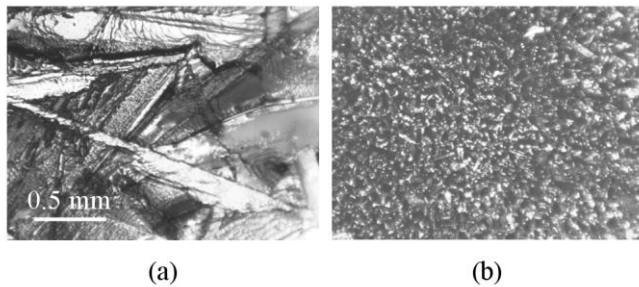
Fig. 7. Photomicrographs of thin layers of CDD on microscope slides showing crystal polymorphism: (a) slow cooling rate, (b) fast cooling rate.

Fig. 8. Photomicrograph of a cross-section through a layer of CDD (built up from three layers) deposited on plaster (bottom).

spectra and thus have differing frequency-dependent absorption coefficients and refractive indices [16]. Although no localized absorption features are visible within the frequency range covered in Fig. 6, visual inspection of the CDD samples indicates the existence of different polymorphs corresponding to the different cooling rates. Because of an orientational dependence of the crystalisation within the moulds, the differences are most easily observed on the face of the samples rather than in the cross-sections shown in Fig. 4. To illustrate the different polymorphs, Fig. 7 shows photomicrographs of thin layers of CDD deposited from melt onto glass microscope slides. Fig. 7(a) was deposited onto a heated microscope slide, resulting in a slower cooling rate than the unheated slide used for Fig. 7(b). The crystal forms are very different in the two cases, with long needle-like crystals in the slowly cooled case and much smaller, more nearly round crystals in the rapidly cooled case. Therefore, it is expected that the optical constants of the CDD samples will be dependent on crystal polymorphism as well as scattering.

The refractive index (Fig 6(a)) shows a more complicated dependence on cooling rate, with the slow cooled sample having the lowest refractive index and the medium cooled sample the highest. This behavior can be seen to be consistent with that shown by the absorption coefficient if one considers the relative proportion of radiation transmitted directly through the sample to that which has undergone multiple internal scattering into the forward-going direction. In the slow cooled sample, much of the multiply scattered radiation is absorbed within the sample, so that a high proportion of the detected radiation is composed of that which has been transmitted directly through the CDD crystals. In the medium cooled sample, less of the multiply scattered radiation is absorbed internally, so that there is a more significant proportion that is scattered into the forward-going direction. This scattered component will take a longer path length through the sample than that transmitted directly, resulting in an increased refractive index. Because of the small size of the crystals, the least scattering occurs within the fast cooled sample, resulting in a smaller proportion of forward scattered radiation and therefore a lower refractive index than that of the medium cooled sample. Nevertheless, the refractive index of the fast cooled sample is still higher than that of the slow cooled sample, suggesting the presence of some internal scattering. The presence of polymorphism can be expected to further modify the r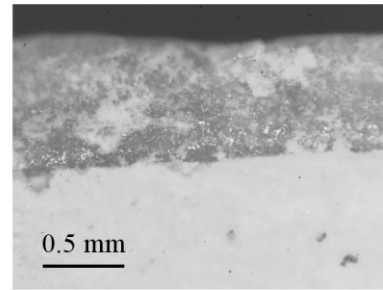efractive index, with the result that the measured refractive index is a combination of effects due to both scattering and polymorphism.

IV. DISCUSSION

The optical constants of CDD are the key to understanding why it can be expected to improve contrast in the terahertz imaging of concealed wall paintings. When applied in the form of a melt in art conservation, it is done so as a relatively thin layer which is in direct contact with the object, penetrating into porous materials. Consequently, it cools rapidly, and so the optical constants of the CDD applied in this manner can be expected to be close to those for the fast cooled sample presented above. This is borne out by the cross-section of CDD deposited on plaster shown in Fig. 8. Similarly, when applied as a solution or via aerosol spray, large crystals do not form, and again the optical constants of the fast cooled sample can be considered representative. For the fast cooled case, the absorption is low and the refractive index lies between 1.46 and 1.47. In comparison, the refractive index of typical wall plaster is around 2.0 throughout the terahertz range.

Plaster is a porous, granular material, consisting of a matrix of plaster particles separated by air gaps. As the refractive index of the plaster particles differs from that of air, an amount of internal scattering will occur if terahertz radiation is transmitted through a piece of plaster, due to scattering at the interfaces between the plaster particles and air gaps. This scattering increases the absorption of the radiation in a manner similar to that seen for the CDD samples presented above.

However, as CDD has a refractive index which is intermediate between that of plaster and air, if it is applied to a plaster sample so that it soaks into it, it will act as a refractive index matching medium, replacing air in the porous plaster matrix, reducing the loss of terahertz light due to scattering in the plaster. This mechanism is illustrated diagrammatically in Fig. 9.

In the case of terahertz reflection imaging of a wall painting covered with a layer of plaster, scattering will occur both at the front surface of the plaster and within the plaster layer, reducing both the signal power falling on the subsurface paint layer and the returned signal reflected from this paint layer. However, application of CDD will reduce both the front surface and internal scattering, resulting in a larger return signal from the subsurface paint layer, and thus an improved signal-to-noise ratio and enhanced image contrast in the
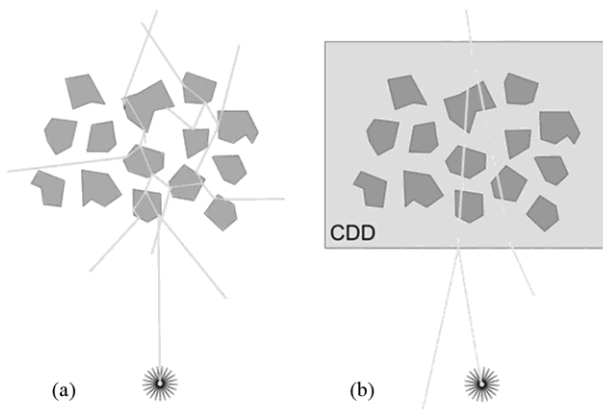
Fig. 9. Diagrammatic representation of effect of cyclododecane as a refractive index matching medium: (a) simplified illustration of ray path through aggregate material, (b) ray path with matched refractive index medium.

resulting terahertz image.

An analogous technique, known as tissue optical clearing, is used to reduce scattering and enhance the transparency of biological tissue in the visible and near-infrared regions [17],[18].

V. CONCLUSION

Motivated by imaging experiments on the wall painting "Les Trois Hommes Armés de Lances", which showed evidence of a sub-surface Roman painting, this paper has demonstrated that the art conservation substance CDD can improve the signal-to-noise ratio and image contrast in terahertz images of concealed wall paintings. Measurements of the complex optical constants of CDD in the terahertz range have indicated how these are dependent on the crystal size formed under differing cooling rates from the melt. It has been shown that this behaviour can be explained in terms of internal scattering within the CDD samples. Based on these measurements, an explanation behind the image enhancing properties of CDD has been presented. The measurements suggest that CDD can act as a refractive index matching medium, replacing air in the porous plaster matrix, reducing scattering and absorption in the plaster, and improving the signal-to-noise ratio and sub-surface image contrast.

It is recognised that the application of a foreign substance, such as CDD, to an artwork may be considered controversial and, in a sense, negates the non-invasive nature of terahertz imaging. There is always the danger that such a substance may cause some damage to the artwork, even if it does sublimate away completely with time. Therefore, the authors do not advocate the use of CDD purely for the purpose of terahertz imaging. However, if CDD has been applied to a wall painting as part of a carefully controlled conservation process, it provides an excellent window of opportunity in which to gain improved images. Indeed, one conclusion that can be drawn from this paper is that the maximum benefit is gained if the painting is imaged as soon as possible after the CDD has been applied.

The image improvement seen will be dependent on the porosity of the base material and the CDD application technique, as these will affect the depth to which the CDD penetrates. It is noted that the refractive index of CDD is not an exact match to that of plaster, and so a material with a closer match would give an even greater improvement. Therefore, there is the potential to develop other novel refractive image matching materials tailored for application to a wide variety of porous materials. Consequently, this work may prove to be a breakthrough advance in terahertz sub-surface imaging through porous materials in general.

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