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Congregation leaving the Reformed Church in Nuenen by Vincent van Gogh: a combined multi-instrumental approach to analyse the painting's stratigraphy in support of varnish removal

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Abstract

Vincent van Gogh's painting *Congregation Leaving the Reformed Church in Nuenen* from the collection of the Van Gogh Museum in Amsterdam was executed in 1884 and partially repainted by the artist in 1885. The painting was restored in 1961, however, the details of this treatment were not documented. After being stolen from the museum in 2002 and finally recovered in 2016, the *Church* was subjected to an extensive technical examination campaign which started in 2017. The aims were to: characterise the stratigraphy of both initial and later paint layers (including identification of the painting materials used by Van Gogh), evaluate the condition of the painting and assess the feasibility of the desired restoration treatment. Portable X-ray fluorescence spectrometry (XRF) was performed to non-invasively identify elements related to pigments in the paint layers of the two painting campaigns. To further identify constituent materials and comprehend the painting's complex stratigraphy, a single paint sample was collected and embedded in resin for analysis by means of Optical Microscopy, Scanning Electron Microscopy with Energy Dispersive X-ray spectrometry (SEM-EDS) and Fourier Transform Infrared spectrometry - Attenuated Total Reflectance (FTIR-ATR). Additional non-invasive measurements were performed in a MOLAB campaign in 2018 by two complementary and portable analytical techniques: Optical Coherence Tomography (OCT) and reflection FTIR spectroscopy were used to gain further insight into the painting's stratigraphy and identify surface layers across various regions of the painting. The presence of an original varnish under the paint from 1885 (and therefore likely applied by Van Gogh himself) was revealed by OCT. It was characterised as being protein based by FTIR-ATR and reflection FTIR spectroscopy. Based on the knowledge on the artist's varnishing practice, it could be concluded that this most likely concerns an egg white varnish for the first time found in an early work by Van Gogh. The upper varnish layer, however, was identified as an alkyd resin applied during the aforementioned 1961 treatment. The combined use of FTIR and OCT enabled non-invasive in situ assessment of solvent cleaning procedures aimed at the selective removal of the 1961 restoration varnish with the preservation of Van Gogh's original varnish. Specifically, OCT and FTIR analyses were carried out before, during and after each cleaning test to carefully assess the condition of the painted surface and that of the original

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varnish. The results of the cleaning tests aided in fine-tuning the procedure of varnish removal during the restoration process.

Keywords: Varnish removal, Cleaning painting, Non-invasive examination, FTIR, OCT, XRF, Van Gogh, MOLAB, Egg-white varnish, Alkyd resin varnish

Introduction

Congregation Leaving the Reformed Church in Nuenen from the collection at the Van Gogh Museum in Amsterdam (Fig. 1, left) was painted by Vincent van Gogh (1853–1890) in early 1884 while he lived with his parents in Nuenen.[1] He gave the painting—which depicts the church where his father worked as a minister—as a gift to his mother who was at that moment confined to bed with a broken leg. In a letter [1, letter 428] to his brother Theo on or about 3 February 1884, he wrote "Fortunately Ma's mood is very equable and content, considering her difficult situation. And she amuses herself with trifles. I recently painted the little church with the hedge and the trees for her." A year and a half later, in the autumn of 1885, Van Gogh returned to the painting and partially repainted it. The identification and dating of both painting sessions were published in [2, entry cat. 6, pp. 58–65]. As illustrated by a sketch the artist included in the aforementioned letter 428 and the drawing *The Reformed Church at Nuenen* (Kröller-Müller Museum, Otterlo) [2, p. 61], the original composition featured only one figure in the foreground: a peasant carrying a spade, which is still visible in the X-radiograph of the painting [2, p. 61]. In the second painting session, Van Gogh painted over the peasant, and applied touches of autumn colours to the trees, hedges and foreground. He also added churchgoers—among them women in mourning shawls—which may be a reference to the death of Van Gogh's father only a couple of months earlier, in the spring of the same year.

Together with another early work by Van Gogh, *View of the Sea at Scheveningen*, the painting was stolen from the Van Gogh Museum during a robbery in 2002 [3]. Both were recovered in 2016 near Naples, Italy. After being returned to the museum in Amsterdam, they were subjected to a detailed examination in order to determine their condition. Although *View of the Sea at Scheveningen* was considerably damaged, *Congregation Leaving the Reformed Church in Nuenen* was barely harmed during its almost fourteen years of absence. Prior to the rehanging of the *Church* painting in the galleries, the museum considered whether to remove the aged yellowed varnish that added an undesired overall gloss and warm tint to the composition (Fig. 1).

In order to create a tailored treatment method for this painting, further knowledge had to be gained regarding the number of varnish layers present, the reasons why they were applied and by whom as well as their composition and solubility properties. It was also important to locate the varnish layers in relation to the overall layer build-up of the painting given by the two separate painting sessions.

Only little is known about Van Gogh's varnishing practice. From a few letters he sent to his brother Theo in 1883 and 1885 it appears that he either varnished his paintings himself or asked Theo to do so, once the paint layers had dried [2]. The function of this varnish layer was not so much to produce a glossy surface, but rather to saturate the colours, and therefore to bring out the nuances of the fairly dark palette typical for Van Gogh's works in his early career [1, letter 389]. He distinguishes between 'egg white' and 'varnish', without specifying the material of the latter.

Hardly anything has been recorded concerning the restoration history of the painting. As a transport document in the archives of the Van Gogh Museum indicates it was sent to the restorer J.C. Traas in 1961, together with two other works by Van Gogh. Although the exact nature of the treatments carried out was not recorded,[2] recent research based on Fourier transform infrared spectrometry (FTIR) and gas chromatography-mass spectrometry (GC-MS) analyses has revealed a synthetic, alkyd-based varnish layer on one of these paintings (Vincent van Gogh, *Sunflowers*, 1889), which can be linked to this specific restoration [4]. Therefore, the possibility of the presence of an alkyd varnish also on the surface of *Congregation Leaving the Reformed Church in Nuenen* needed to be taken into consideration.

The initial microscopic examination of the entire painting could not answer the above posed questions regarding the present varnish layers, but it did reveal instead a further complicating factor, namely a locally disrupted paint and varnish structure, of which the cause and origin was then unknown. In some areas, the surface had a cobblestone-like character (Fig. 2a), and under magnification it appeared as if particles from the paint layers might have migrated into the varnish layer (Fig. 2a and b). Furthermore, tiny semi-transparent 'lumps' or 'islands'

[1] Van Gogh stayed in Nuenen from Dec. 1883-Nov. 1885.

[2] The painting is wax-resin lined, but it is unclear whether this was done during the 1961 intervention.

Fig. 1 Vincent van Gogh, *Congregation Leaving the Reformed Church in Nuenen*, January/February 1884 and autumn 1885, oil on canvas, 41.5 cm × 32.2 cm, Van Gogh Museum, Amsterdam (Vincent van Gogh Foundation), before restoration treatment. Left: visible light photography; right: photography of UV-induced fluorescence (filters: 85B and UV 2E pale yellow, Tiffen), image showing the yellow green fluorescence of the varnish layer. Spots of examination mentioned in the text marked: IRn: reflection FTIR spectroscopy; OCTn: Optical Coherence Tomography; PMn: photomicrographs; S1: sample collection spot for Optical Microscopy, SEM-EDS, and FTIR-ATR; TESTn: spots where cleaning tests were performed and monitored by OCT and reflection FTIR spectroscopy

were observed scattered across large parts of the painting, sometimes accompanied by fine cracks in the upper paint layers (Fig. 2c and d). All together, these phenomena raised the question as to whether the varnish could be safely removed without harming or indeed interfering with the original structure of the painting.

In order to clarify these aspects a micro-sample including the ground, paint and varnish layers was taken from the painting. It was embedded as a cross-section and examined using optical microscopy (OM), scanning electron microscopy with energy dispersive X-ray spectrometry (SEM-EDS) and Fourier transform infrared spectrometry - attenuated total reflectance, (FTIR-ATR) to gain information about the stratigraphy and the chemical composition of both original and later constituent materials. In addition, pigments in the painting were characterized using hand-held non-invasive X-ray fluorescence (XRF) spectrometry. Following this first round of examinations, selected areas of the entire surface of the painting were examined non-invasively using optical coherence tomography (OCT) and reflection FTIR spectroscopy. These portable instruments were accessed through the Mobile Laboratory for cultural heritage, MOLAB [5], of the European Research Infrastructure for Heritage Science E-RIHS (http://www.e-rihs.eu/). The OCT/FTIR integrated approach is well established in examination of paintings for the simultaneous measurement of chemical, optical and morphological changes [6, 7], and allows for a series of varnish removal tests to be monitored. Therefore, these complementary techniques were well suited to both supplement the initial investigations of the irregular surface phenomena (Fig. 2) and to provide feedback on the cleaning methodology of the painting during this restoration.

Methods/experimental

Locations of all examination spots mentioned in this report are given in Fig. 1, left and (for XRF) in Additional file 1: Fig. S1.

X-ray fluorescence spectrometry (XRF)

To gain an overview of the pigments that Van Gogh employed in this painting, the chemical elements were identified in a total of 17 locations (Additional file 1: Fig. S1) using a portable X-ray fluorescence spectrometer, the Bruker Tracer 5i. This instrument consists of a low power Rhodium X-ray tube and a Silicon-Drift energy dispersive X-ray detector. A collimator with a diameter of 3 mm was used for the analysis. The measurements were performed under atmospheric conditions, using a tube voltage of

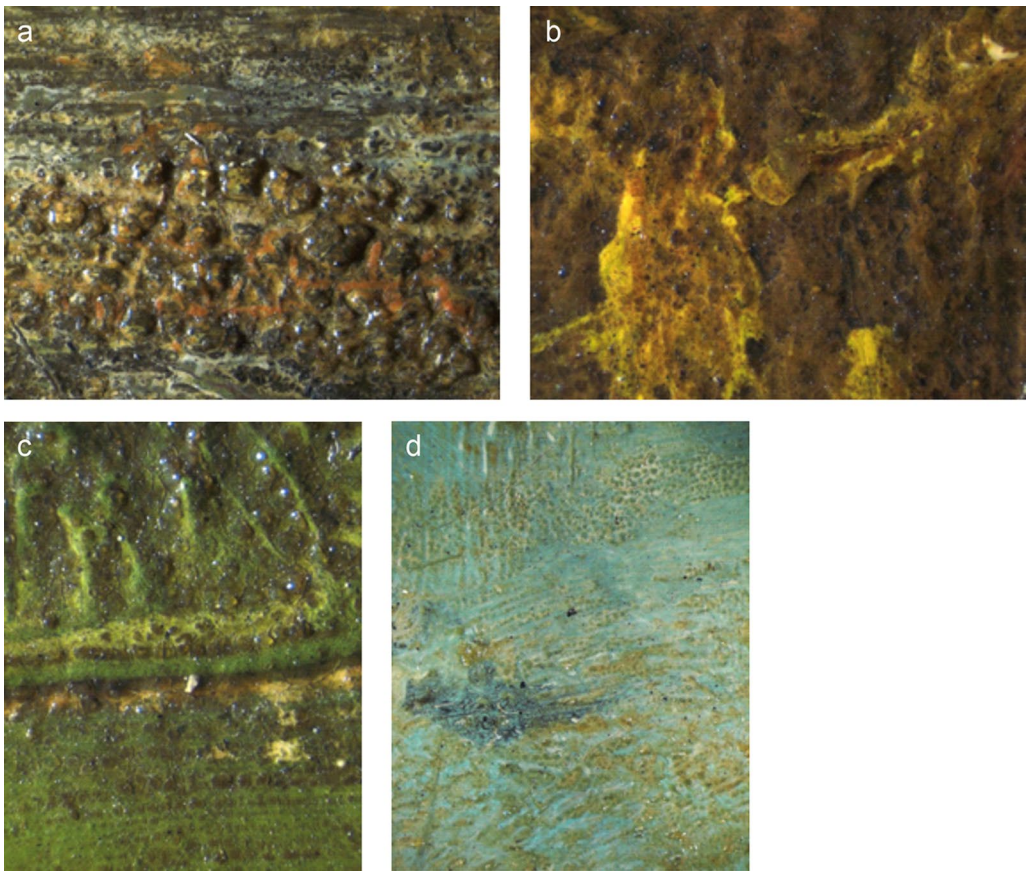

Fig. 2 Range of different phenomena observed on the painting's surface. Photomicrographs taken in: **a** raking visible light at ×25 magnification; **b** raking visible light at ×40 magnification; **c** raking visible light at ×40 magnification; **d** scattered visible light at ×25 magnification. For the location of the images, see Fig. 1 (left)

40 kV and 6 μA current. The acquisition time was 10 s in measurements 1–4 and 60 s in measurements 5–17.

Optical microscopy (OM) and scanning electron microscopy with energy dispersive X-ray spectrometry (SEM-EDS)

A single micro-sample including the full stratigraphy was taken from the painting (for location see Fig. 1 left), embedded in a polyester resin (Polypol PS230) and polished perpendicularly to the surface to obtain a cross-section. This was examined under a Zeiss Axioplan 2 optical microscope, both with bright field illumination and UV-induced fluorescence. The filter set 'UV H365' used for examination in UV-light consists of the following filters: excitation BP 365/12, beam splitter FT 395 and emission LP 397.

For further identification of the pigments, a Jeol JSM 5910 LV scanning electron microscope was used with a Thermo Fisher silicon drift detector system for energy dispersive X-ray spectrometry (SEM-EDS). The primary electron beam energy was 20 kV. The cross-section was examined in the low-vacuum mode (30 Pa).

Fourier transform infrared spectrometry by attenuated total reflectance (FTIR-ATR)

FTIR-ATR imaging was performed on the cross-section of the sample by means of a Perkin Elmer Spectrum 100 FTIR spectrometer combined with a Spectrum Spotlight 400 FTIR microscope equipped with a 16 × 1 pixel linear mercury cadmium telluride (MCT) array detector. A Perkin Elmer ATR imaging accessory employing a germanium crystal was used for ATR imaging.

Optical coherence tomography

Optical coherence tomography is an optical, non-invasive technique for examination of subsurface, semi-transparent structures with micrometric resolution, utilised mostly as a medical diagnostic tool [8] but also applied successfully for examination of various heritage objects [9, 10]. In this study a high resolution (2.2 μm

axial resolution in paint media of refractive index $n_R = 1.5$ and 12 µm lateral resolution) spectral domain OCT instrument was used. The painting was analysed with broadband infrared radiation (750–960 nm) of power at the surface not exceeding 0.8 mW and fluence about 30 mJ/cm^2—see [10] for details of the hardware and data processing.

The OCT cross-sectional images (tomograms or B-scans) are presented in false colour scale with colours representing local scattering properties of the examined structures: the areas not-scattering IR radiation or not reached by it are shown as black. The layers of increasing, but still moderate scattering properties, are shown in cold colours (from blue to green respectively), whereas the centres of high scattering are represented in warm colours—from yellow to red. The presented tomograms are vertically stretched for better legibility: cross-sections are 10 mm (Figs. 4 and 6) or 12 mm (Fig. 7) wide but only 0.556 mm high. Scale bars in all the tomograms are equivalent to 200 µm in both directions. It is worthwhile to mention that OCT primarily detects in-depth distances as optical and not geometrical ones, but all tomograms presented herein are corrected for this effect with use of a common value of 1.5 for refractive indices of varnishes and binders. The possible error caused by the divergence from the real values is below the resolution of the instrument in case on thin layers present here.

In this contribution, OCT data are presented not only in the form of tomograms, but also as *en-face* topography maps (h, i in Figs. 6 and 7) of examination areas showing topography of the paint surface before and after the varnish removal tests were carried out. In order to generate such maps, a surface profile was extracted from 3D OCT data cube composed of 150 B-scans and collected after every cleaning step. Then the surface profiles obtained after cleaning are subtracted from the ones acquired before, which enable assessing the bulk of material removed in every step in a form of a differential maps [6], also coded in false colour scale (j in Figs. 6 and 7). Here, for conciseness only maps obtained after the final cleaning step are shown.

Reflection FTIR spectroscopy

Non-invasive FTIR analyses (a total of 26 measurements) were performed using the portable FTIR spectrometer ALPHA produced by Bruker Optics (Germany/USA-MA). The instrument is equipped with a SiC globar source, a "rock solid"-design interferometer (with gold mirrors) and a DLaTGS detector. An external reflectance module with an optical layout of 22°/22° allows contactless measurements from areas of ca. 3 mm (Ø) visible on the PC monitor by a USB camera interfaced with the spectrometer. The IR spectra were collected in a spectral region ranging from 7000 to 350 cm^{-1} by setting 146 scans and a resolution of 4 cm^{-1} and visualized in pseudo-absorption mode (Log (1/R); R = reflectance). Spectra collected from a gold flat mirror were used as background.

µFTIR spectroscopy

The cotton swabs used for the varnish removal tests (see next sub-section) were put in closed glass vials with pure ethanol for 2 h in order to extract the material eventually removed during the tests. Subsequently, the extracts were deposited on an aluminium plate and after drying analysed in the laboratory by µFTIR spectroscopy in transflection mode [11]. The instrument used is a Jasco Fourier Transform Infrared (FTIR) 4100 spectrometer equipped with a ceramic light source, a Michelson interferometer and a nitrogen cooled mercury cadmium telluride (MCT) detector. The spectrometer is coupled to a Jasco IMV 4000 optical microscope with three objectives (Cassegrain 16X, Cassegrain 32X and ATR). For the analysis of the swab extracts, the areas were selected by the Cassegrain 16X. The IR spectra were collected in the range from 7000 to 600 cm^{-1} with a spectral resolution of 4 cm^{-1} and using 4000 scans.

Methodology of the instrumental monitoring of varnish removal tests

Prior to restoration, a number of tests were performed to determine the solubility properties of the upper varnish layer that was to be removed. During the preliminary phase, different organic solvents were tested in various locations of the painting. Wettened cotton swabs were carefully rolled over the surface and the reaction was observed under the stereomicroscope. It appeared that the best effect was obtained with pure ethanol, which dissolved the varnish material efficiently without visually affecting the paint layers. This solvent was thus selected to be employed in the subsequent varnish removal tests that were monitored in-situ and step by step by means of FTIR and OCT. Additionally, the extracts from swabs were also analysed by µFTIR spectroscopy. Two of these tests will be discussed in more detail in the following section, both of which were conducted in areas that are representative of the painting's complexity. They were expected to assist in determining whether the non-original varnish could be removed without harming the original structure underneath.

- test no. 7: pure ethanol applied in 3 steps by rolling consecutive times with cotton swabs. Step 1: 24 rolls; step 2: 12 rolls; step 3: final cleaning under the microscope (Fig. 6),

- test no. 9: pure ethanol applied in 2 steps by rolling with cotton swabs. Step 1: 18 rolls; step 2: 12 rolls (Fig. 7).

The areas selected for the cleaning tests (10×10 mm^2 for test no. 7 and 12×12 mm^2 test no. 9) were marked by 'windows' cut in transparent Mylar foil. As the first part of every test, before commencing actual cleaning, a FTIR spectrum was collected in the middle of the marked square area. The painting was then placed horizontally under the OCT head with a scanning beam projecting down and OCT data was collected. Afterwards, the solvent cleaning procedure was applied by rolling cotton swabs on the surface of varnish without moving of the OCT head (with 43 mm distance from the lens to the painting). Then the OCT data were collected again for the repeatability of scans and thus for a precise comparison between the scans obtained before and after this treatment step. Subsequently, the painting was transferred for FTIR analysis on the same marked area, and, for the next cleaning step, repositioned again under the OCT instrument. This procedure was repeated for as many steps as necessary. Due to the relatively large size of the FTIR data collection spot (3 mm) it was possible to reposition the painting for consecutive measurements within the designated foil window. For the OCT measurements, however, a micrometric scale precision is necessary for the comparison of tomograms and generation of differential maps from subsequent cleaning steps. Mechanical limiters were used for coarse positioning of the painting after relocation with precision of about 50 µm in X,Y directions but with no control in Z direction. Software matching of the B-scans taken from the OCT data collected before and after repositioning enabled achieving the desired micrometric alignment (better than the distance between B-scans in Y direction: 80 µm, better than twice the lateral resolution in X direction and better than twice the axial resolution in air in Z direction).

Results and discussion

Stratigraphy and chemical characterization of painting materials

X-ray fluorescence spectroscopy

To investigate the palette Van Gogh used for each of his two painting campaigns, 17 spots were analysed by XRF, covering different colours of the composition (Additional file 1: Table S1). Six of these analyses were taken from areas that—based on the microscopic examination of the painting's surface—include the initial paint layers from the beginning of 1884 (measurements 2, 4 and 12–15) and ten from areas that comprise both the paint of this first campaign and the 1885 additions (measurements 1, 3, 5–9, 11, 16 and 17). For one location it was not certain whether it is composed of the paint layers of one or both painting campaigns (measurement 10).

Lead was identified in all measurements and is associated with the use of lead white. This pigment is not only the main component of the ground layer, but it was also used in the paint layers. In addition to lead white, zinc was detected, probably present in the form of zinc white. It was chiefly found in some of the analyses that include the 1885 additions, such as in the wall of the church (measurement 3), in the figures' clothing (measurements 5–7), and in the paints of the foreground (measurements 16 and 17). On the contrary, in the paint layers of the 1884 campaign no (measurements 13 and 15) or only trace amounts of zinc (measurements 2, 4, 12 and 14) were detected, in accordance with earlier findings [12].[3] One of these spots containing a trace amount of zinc is a turquoise brushstroke in the sky (measurement 14), whereas in the areas that appear more white and blue only lead was identified (measurements 13 and 15). It is remarkable that the turquoise paint also contained a significant amount of titanium, possibly in the form of titanium white. Because this white pigment came onto the market in the 1920s, it indicates that it does not concern an original brushstroke, but a later retouching.[4]

Apart from the apparent differentiation of the use of white pigments in the two painting phases, particularly in the later added yellow and orange paint strokes of the hedge (measurements 8 and 9) a relatively large response for iron was obtained, indicating the use of different shades of ochre in these 1885 additions.

Next to the whites and ochres, several other pigments were identified in the paint layers from 1884 to 1885. In the sky, Prussian blue was indicated by the enhanced iron peak in the spectrum of the blue sky (measurement 13) when compared to the white passage (measurement 15). Van Gogh presumably used the same pigment in 1885 to paint the blue coat of one of the figures (measurement 5), as indicated by the detection of iron, though the presence of cobalt blue could not be fully excluded here.[5] In addition, except for two analyses of the sky (measurements 14 and 15), mercury was identified in each spot, which is characteristic of the red pigment vermilion. Moreover, Naples yellow, indicated by the co-presence of lead and antimony, was detected in several areas. The detection of

[3] In the three paintings examined from the first half of 1884 no zinc white was detected, while zinc white was identified or probably present in 15 out of the 16 investigated paintings dated 1885.

[4] The retouching appears somewhat darker in the UV fluorescence image, see Fig. 1, right. As there is no loss visible in this area in the X-ray, the retouching might have been applied in order to cover the disrupted paint surface.

[5] In the blue coat and white hat of the figures in front of the hedge (measurements 5 and 6) as well as in the green paint in the foreground (measurement 16) a peak around 6.9 keV was detected, which matches the $K_{\alpha 1}$ emission line energy of cobalt. However since the response for zinc was very high in these areas, this peak may rather correspond to the escape peak of the $K_{\alpha 1}$ emission line of this element, which similarly appears at 6.9 keV.

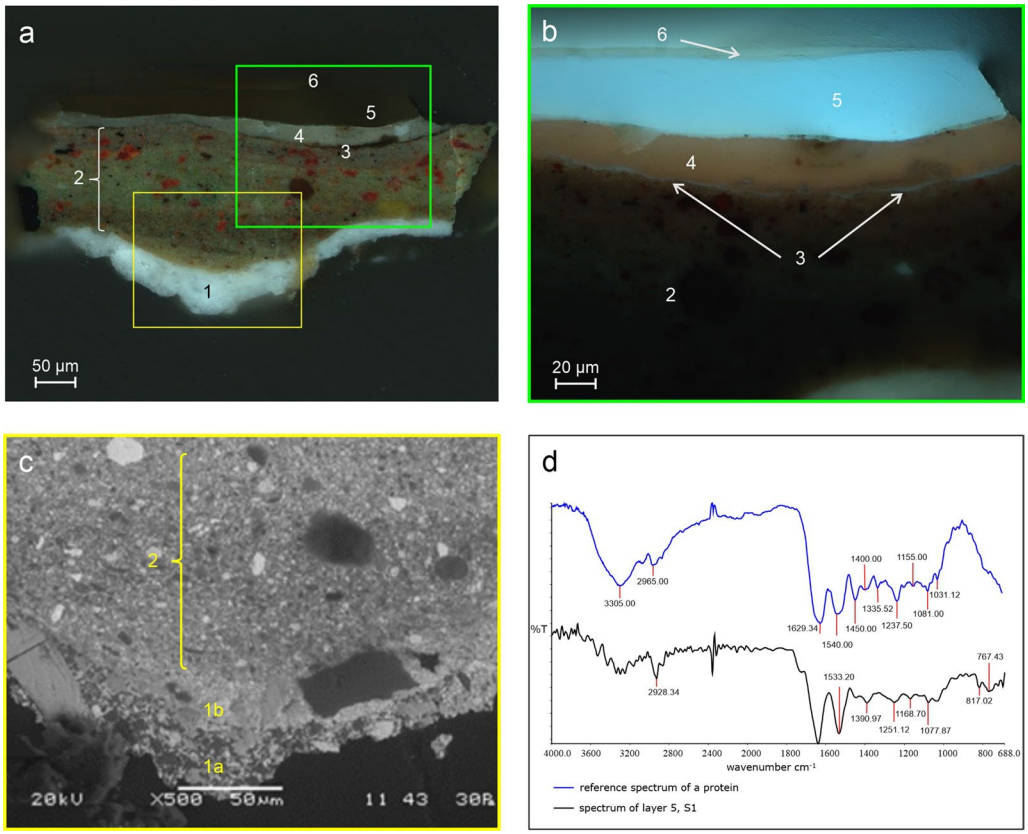

Fig. 3 Results of examination of the cross-section of the sample taken from the lower edge of *Congregation Leaving the Reformed Church in Nuenen* (spot S1 in Fig. 1, left); **a** image taken in bright field illumination; **b** UV-induced fluorescence; **c** Backscattered Electron Image; **d** FTIR-ATR spectrum of layer 5

a small amount of chromium in a few spots suggests that chrome yellow or orange may also be present, or alternatively chromium oxide green.

Paint sample analysis by OM, SEM-EDS and FTIR-ATR

The cross-section of the paint sample (Fig. 3) taken from the lower edge of the painting comprised ground, paint and varnish layers. SEM-EDS was used to identify elements present in ground and paint layers. The backscattered electron image (BEI) revealed that the canvas was prepared with two ground layers (Fig. 3c, layers 1a and 1b). Both layers within the ground consist of lead, calcium, barium and sulphur—with individual particles containing either mainly lead, calcium or barium and sulphur—indicating the presence of lead white, calcium carbonate and barium sulphate respectively. The components of the ground differ in the amount of calcium present: the top ground layer comprises considerably less of this element than the bottom layer. The barium- and sulphur-containing particles appear as large, angular crystals in the BEI, characteristic for the natural variety of barium sulphate called 'barytes'. In addition, a single, large particle containing silicon was detected. On top of the ground, several paint layers containing mixtures of pigments (2) were applied wet-on-wet. The main component in the paint is probably lead white, while the relatively large orange-red pigment particles correspond to vermilion, as confirmed by the identification of mercury and sulphur. Next to these, yellow and red ochres were indicated by the detection of aluminium, silicon and iron, and ultramarine was suggested by the presence of sodium, aluminium, silicon, sulphur and potassium in one of the fine blue pigment particles. In addition, a little black pigment could be observed in the paint layers and a small amount of barium sulphate is probably also present, as indicated by the presence of barium in the layer. These paint layers are covered with a very thin transparent layer that fluoresces when excited by UV (3). This may concern a saturating or retouching varnish that enables the artist to resume painting after having interrupted his working process for a short period.[6] The paint layer that Van Gogh continued working with (4 in Fig. 3a, b) contains mostly lead white pigment, as the

[6] Van Gogh also used such retouching varnishes in the *Potato Eaters* (F82) [13].

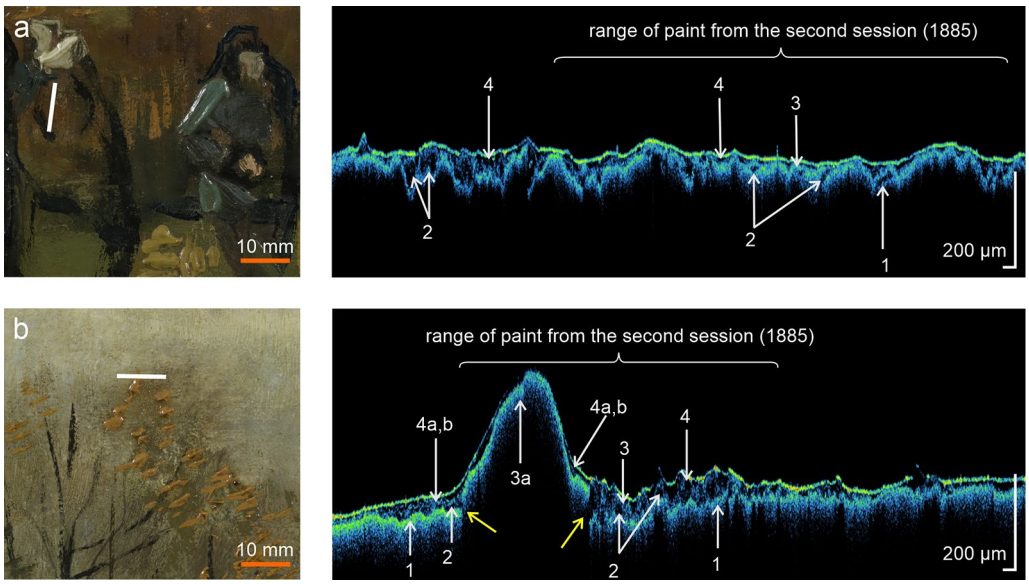

Fig. 4 OCT results collected at spots OCT15 (**a**) and OCT21 (**b**). White lines in the macrophotographs mark the exact location and direction of the OCT tomograms. Layers visible: (1) initial paint layers from 1884, (2) Van Gogh's initial varnish from 1884, (3, 3a) Van Gogh's painted addition from 1885, (4) later, non-original varnish layer(s) from the 1961 restoration treatment. Yellow arrows mark the discontinuity of the paint layer

detection of lead reveals, with a little carbon black added and a trace of red ochre, as demonstrated by the identification of aluminium, silicon and iron in a red particle in the layer. Strikingly, this layer has a pale pinkish fluorescence in UV.

On top of these initial paint layers, two—possibly even three—transparent layers were observed in the cross-section, of which the lowest (5) is rather thick and fluoresces light blue under UV. With FTIR-ATR imaging it was identified as a protein-containing material (Fig. 3d), and in combination with the microscopic examination of the painting's surface, the observed semi-transparent islands and lumps could be related to this layer. Considering that Van Gogh mentioned the proteinaceous egg white as a varnish in two letters to his brother Theo [1, letter 389 and 497], this was a strong indication for this particular material to be present here. By contrast, the upper (possibly) two layers (6)—which show a rather yellowish fluorescence—and the organic intermediate layer (3) could not be adequately analysed by FTIR-ATR given their thickness of only a few micrometres.

On the basis of these initial investigations, the composition of the varnish layers and their location in relation to the paint layers of the first and second campaign could not be completely resolved. Therefore, the integrated OCT/reflection FTIR approach was applied as discussed in the next section.

OCT/reflection FTIR on the painting's surface

Further characterisation of the complex stratigraphy of paint and varnish layers as well as their topography was achieved by employing the non-invasive optical coherence tomography technique. The painting was examined in 35 spots within which OCT detected two layers of varnish in 30 spots as already observed in the paint sample cross section. The upper layer is thin and continuous with the average thickness of 10–12 µm, and is in some cases noticeable as two layers. In combination with the results obtained by reflection FTIR (see below), this varnish can most likely be related to the restoration treatment carried out in 1961. The bottom varnish layer on the contrary is less continuous and can frequently be seen in the form of round-shaped lumps of transparent material of various sizes (30–100 µm in diameter), probably concerning the original egg white varnish by Van Gogh.

Figure 4 shows exemplary stratigraphy of the painting in two spots: OCT15 (a), OCT21 (b), see Fig. 1 for locations. In both cases, OCT imaging proved the existence of the two sessions of painting. In Fig. 4a the right-hand side of the tomograms corresponds to the bottom part of the examined area (a woman's brown cloak); this part was added by Van Gogh in 1885. From top, a single thin layer of the upper varnish may be seen (4), applied on top of the paint layer constituting the artist's modification from 1885 (3). Below this layer, lumps of transparent material

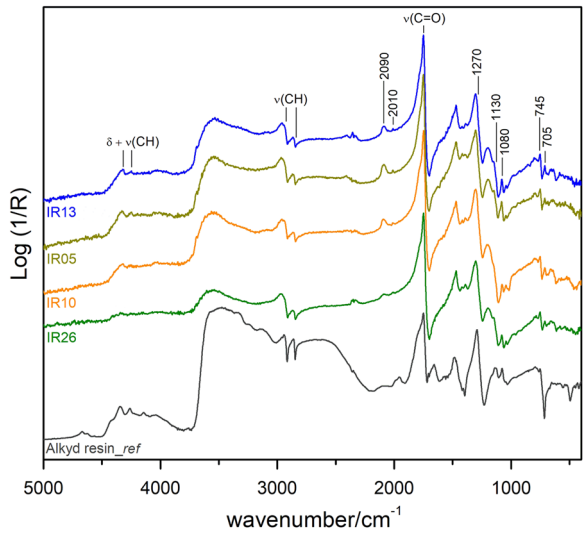

Fig. 5 Selected reflection FTIR spectra collected in the areas indicated in Fig. 1, left, in comparison with a reference IR spectrum of an alkyd resin (grey spectral profile)

(the bottom varnish layer) can be observed (2), separating the overpaint from the initial paint layers from 1884 (1).

From Fig. 4b it can be assessed that the overpaint layer of Van Gogh's 1885 intervention (3) in this case is limited to the central part of the tomogram, which corresponds to the area of the autumn leaf impasto (3a) applied on top of the varnished painting (2). This is clearly shown by an interruption in the continuity of the paint layer stratigraphy marked by the yellow arrows in Fig. 4b. Furthermore, on the left-hand side of the same tomogram three transparent layers are visible (2, 4a, 4b) on top of the initial paint (1). Also on top of the later impasto from 1885, two thin layers of (non-original) varnish may be observed, originating from the 20th century restoration treatment (4a, b).

All the non-invasive FTIR spectra collected in different areas of the painting allowed the characterization of this uppermost varnish. These IR profiles (Fig. 5) showed intense derivative-shaped bands of the carbonyl asymmetric stretching mode (1740–1730 cm^{-1}) and the C–O–C stretching at ca. 1270 cm^{-1} that, in combination with sharp CH signals (stretching and combination bands in the spectral ranges 3000–2800 cm^{-1} and 4400–4200 cm^{-1} respectively), indicated the presence of an alkyd resin [14]. This was also confirmed by the detection of the doublet at ca. 1130 and 1080 cm^{-1}, ascribable to the C–O stretching and C–C deformation mode respectively [14], as well as by the aromatic out of plane CH bindings at ca. 745 and 705 cm^{-1} [15]. Moreover, in most of the collected IR spectra the marker bands for Prussian blue (C≡N *stretching* at ca. 2090 cm^{-1}) and bone black (diagnostic feature at ca. 2010 cm^{-1}) from the underlying paint layer/s were recognized [16].

From the OCT and FTIR measurements described above, it was established that the upper, visually degraded, varnish layer(s) were an alkyd resin. These could now quite firmly be linked to the restoration carried out by Traas in 1961—based on the knowledge that he varnished another painting with the same material that year. The lower transparent layer is located either under the paint from Van Gogh's second painting campaign or, in areas that had not been altered by the artist, directly under the alkyd varnish. This layer had been identified in the sample cross-section (Figs. 1 and 3) as a protein-based material by FTIR-ATR and can probably be linked to Van Gogh's varnishing practice of using egg white in that period.

As mentioned above, rather than forming a continuous layer, the egg white varnish is present in the shape of (semi-)transparent lumps or islands, which is also visible in the micrographs of the painting's surface (Fig. 2). This is most likely the result of the characteristic shrinking that takes place during the drying process of egg white. Consequently, this feature has also led to cracks in the underlying semi-dry paint layers, with which the egg white has formed a tight bond. The disrupted paint surface can thus be linked to the application of this particular material.

These poor properties of egg white were absolutely well-known at the time, as both historic and contemporary sources advised artists against using this material as

(See figure on next page.)

Fig. 6 **a** Localisation of cleaning test no. 7 (the yellow circle indicates the area analysed by portable FTIR); **b**, **c** photomicrographs (VIS raking light and UV-induced fluorescence) of this area after treatment (see Fig. 2a for the photomicrograph before varnish removal). White squares mark areas mapped with OCT, white rectangles exact location of OCT tomograms shown in panels **f** and **g**; **d** reflection FTIR spectra collected before (uncleaned) and after each step of the test in comparison with a reference spectrum of an alkyd resin (characteristic alkyd signals marked with arrows): Si: silicate component; LW: lead white; P: protein; **e**: FTIR spectra recorded in transflection mode from the swab extracts in comparison with a reference spectrum of an alkyd resin and that corresponding to the blank (extract of pure cotton in ethanol). Asterisks mark signals probably affected by those of the blank; **f**, **g** OCT tomograms collected before and after varnish removal, description of layers: (1) initial paint layer from 1884, (2) Van Gogh's original varnish, (3) later varnish layer from the 1961 treatment; **h**, **i** OCT topography maps collected before and after varnish removal; **j** OCT differential map showing amount of removed material

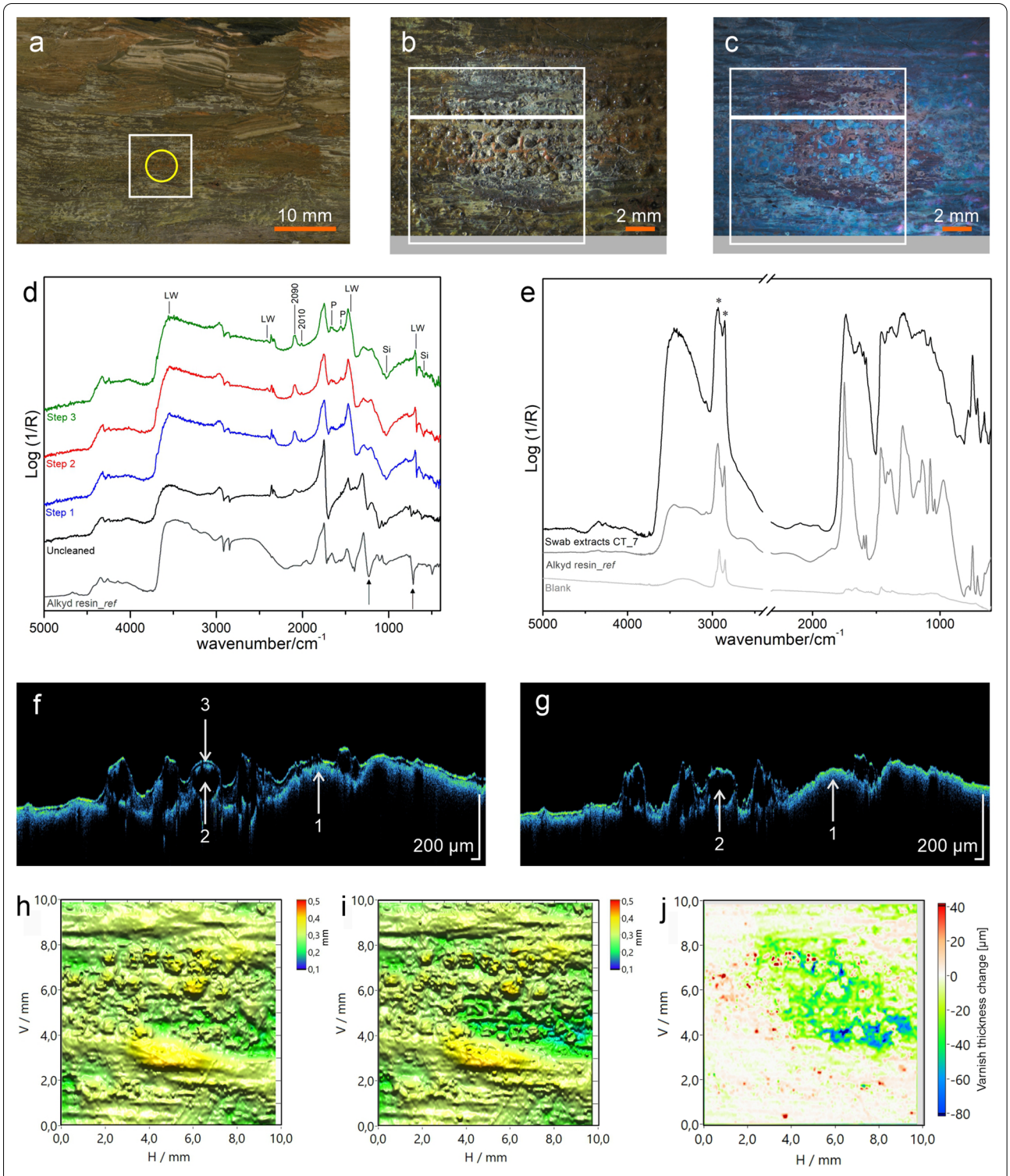

Fig. 6 (See legend on previous page.)

a varnish [17]. It appears however, that Van Gogh was not bothered by these warnings, as he used it nevertheless.[7] He had written a letter to his brother a couple of months earlier, in Sept. 1883, asking Theo to "go over it [a painted study] with the white of an egg in about a week, or some varnish in a month's time, to lift them" because it "has sunk in a lot" [1, letter 389]. In April 1885, again he mentions this material "Yesterday I took it [the painting The Potato Eaters] to an acquaintance of mine in Eindhoven, who is painting. In 3 days or so, I'll go over there and lift it with a little white of egg and finish off a few details" [1, letter 497]. These declarations seem to confirm that Van Gogh did apply egg white to his paintings in order to (re-) saturate the colours and/or as a preparation to retouching his own works. So far *Congregation Leaving the Reformed Church in Nuenen* is the only early work by Van Gogh in which such an original egg white varnish has been identified. However, in four other paintings from the collection of the Van Gogh Museum dating from later in the artist's career in 1888, egg white varnishes have been previously detected by a combination of staining tests for protein and FTIR analysis [18–20].

Since the egg white varnish in *Congregation Leaving the Reformed Church in Nuenen* was most probably applied by Van Gogh himself, the cleaning tests were focused on the selective removal of the uppermost alkyd resin layer(s) while preserving the original varnish.

Assessment of the cleaning tests

The stratigraphy of the superficial layers of the painting in the area of cleaning test no. 7, localized in the lower left-hand part of the painting (TEST 7 in Fig. 1), was established by means of OCT. The upper synthetic varnish (3 in Fig. 6f) was clearly visible, levelling out the relief of the bottom varnish (2) which is manifested in the form of round-shape lumps. After the second cleaning step, the upper varnish was entirely removed (Fig. 6g). It is worthwhile to note that the remaining lower varnish shows a similar UV-induced fluorescence (Fig. 6c) to that of the uncleaned surface (with both alkyd and lower varnish). In the OCT topography maps it can be assessed (Fig. 6h, i) that the surface after the cleaning test regained its original roughness (relief) as a result of removing of the upper synthetic varnish, which is also clearly visible in the raking light photomicrograph (Fig. 6b). Analysis of the OCT differential map (Fig. 6j) showing the amount of the removed material confirms that the removal of the upper layer was not homogeneous in terms of the varnish thickness. Not surprisingly, in the areas corresponding to the most protruding points of the bottom varnish (round white islands localized within the cleaned area in Fig. 6j) the layer of the synthetic varnish was the thinnest. Consequently, the cleaning resulted in only a little change of the varnish thickness. Some areas (marked red in the differential map) show the accumulation of material, probably as a result of transferring some varnish during cleaning with the swab.

Comparing the non-invasive FTIR spectra collected before and after cleaning test no. 7 (Fig. 6d), an intense decrease of the alkyd marker bands at ca. 1270 and 745 cm^{-1} (signals indicated with arrows in Fig. 6d) was promptly recognized already after the first cleaning step. However, in all the spectra acquired after the test a very weak broad band around 1270 cm^{-1} was still visible suggesting the possible presence of alkyd resin residues. The sharp CH signals and the intense carbonyl band, instead, were related to the oil binder of the underlying paint layer/s. In addition, cleaning led to an increase in the intensity of the marker bands for Prussian blue and bone/ivory black, as well as to the appearance of spectral features associated with further pigments like an earth/ochre (silicate component signals labelled with Si in the figure) and lead white (hydrocerussite, see bands marked with LW). Two small bands at ca. 1650 and 1550 cm^{-1} could be correlated to amide I and amide II groups (P signals), respectively, of the lower protein-based varnish. However, the strong inverted band between 1500 and 1400 cm^{-1}, associated with the antisymmetric stretching (ν_3) CO_3^{2-} of lead white, hampered certain identification of the protein component. This, in fact, emerged more clearly after the cleaning performed on a dark yellow-orange area without lead white (test no. 9 in Figs. 1 and 7a–c). In the corresponding OCT tomograms obtained before cleaning test no. 9 (Fig. 7f) two varnish layers can be distinguished, albeit not as well as in cleaning test no. 7. The removal of the upper layer (3) can be seen by comparison of Fig. 7f, g. The effect of cleaning can be, however, better assessed in the OCT topography maps (Fig. 7h, i), where the original roughness of the surface is revealed. In the differential map (Fig. 7j) the accumulation of the varnish around the edges of the test can be seen.

In order to confirm the efficacy of the cleaning test for the selective removal of the alkyd resin, the material extracted from the cotton swabs was analysed in the laboratory using μFTIR spectroscopy. Despite some spectral overlapping (CH signals) with residues of the solvent used for the extraction, the spectra obtained (Fig. 6e) showed all the characteristic bands of an alkyd resin. In addition, the analysis of the extracts obtained from the cotton swabs used for each cleaning step (test no. 9, see Fig. 7e) allowed an approximate assessment of the removed material amount.

[7] Van Gogh preferred a matte surface in the paintings he created during his later career in France and therefore generally did not varnish them at all.

Restoration treatment

Once the feasibility of removing the alkyd resin varnish safely and without harming any original paint and varnish material was confirmed by the monitored tests, the treatment was carried out by applying a tailored method. Instead of rolling a cotton swab over the sensitive surface, it was decided to swell the alkyd resin with small pieces of Evolon® [21] tissue wetted with ethanol. In addition, the tissue was covered with Mylar in order to speed up the swelling process by reducing the evaporation of the solvent. Due to the still rather strong cohesive power of the softened alkyd resin film it was decided to gently brush it off the surface with a stiff-bristled brush, and to remove residues with a set of microtools. The entire treatment was carried out under the stereomicroscope.

After the varnish removal, a very thin spray varnish of Regalrez® 1094[8] was applied to the surface in order to resaturate the dark colours—especially the figures in the foreground—without adding any extra gloss to the surface, in accordance with Van Gogh's ideas. The natural balance of matt and more glossy areas was thus retained. Also, with the yellowed alkyd resin layer removed, the two painting campaigns, from the winter of 1884 and the autumn of 1885, can now be discerned and appreciated again as such: the cold tint of the light blue winter sky contrasting with the warm colours of the autumn leaves—Fig. 8.

Conclusion

In this study, the combination of the applied techniques made it possible to visualize the stratigraphy of *Congregation Leaving the Reformed Church in Nuenen*, to gain information on the painting materials used by Van Gogh, and to understand the origin of the complex surface phenomena observed. This knowledge was crucial to test and define the most opportune cleaning procedure corresponding to the specific complexity and needs of the surface in different areas of the painting.

The painting was created in early 1884 and partially reworked by Van Gogh in late 1885. The initial XRF-measurements revealed the artist's palette used in both painting campaigns. Cross-section analysis provided information about the layer build-up showing that there are at least two varnish layers present. The lower varnish was identified with FTIR-ATR as protein-based, which was also confirmed by reflection FTIR spectroscopy in situ. Unlike the paint cross-section, in the OCT tomograms both painting sessions were recognized and, therefore, it could be determined that the proteinaceous layer is located between the paint layers of the first and second campaign. Consequently, this varnish was applied between spring 1884 and fall 1885, presumably by Van Gogh himself. From the knowledge on the artist's varnishing practice, it can be concluded that this most likely concerns an original egg white varnish. This layer appears to be responsible for the disrupted paint surface—due to its shrinking properties during the process of drying.

By employing reflection FTIR spectroscopy, identification of the uppermost varnish as an alkyd resin made it possible to link it to the 1961 restoration treatment. In contrast to the underlying original egg white varnish, it formed a rather glossy continuous layer that had pooled in the interstices of the paint surface, and its yellowish colour was for a great part responsible for the undesired patchy appearance of the painting.

The combination of results gained from optical coherence tomography and reflection FTIR facilitated the characterisation of both varnish layers, their composition, location as well as origin/dating and made it possible to determine that only the upper, non-original alkyd resin one, should be removed and that the lower, original egg white varnish, was to be left untouched. The monitoring of the cleaning procedure was performed by the simultaneous measurement of chemical, optical and morphological changes of the object step by step. Additionally, the

(See figure on next page.)

Fig. 7 **a** Localisation of cleaning test no. 9 (the yellow circle indicates the area analysed by portable FTIR); **b**, **c** photomicrographs (VIS raking light and UV-excited fluorescence) of this area after treatment (see Fig. 2b for the photomicrograph before varnish removal). White squares mark areas mapped with OCT, white rectangles exact location of OCT tomograms shown in panels **f** and **g**; **d** reflection FTIR spectra collected before (uncleaned) and after each step of the test in comparison with a reference spectrum of an alkyd resin (characteristic alkyd signals marked with arrows). Si: silicate component; P: protein; **e** FTIR spectra recorded in transflection mode from the swab extracts in comparison with a reference spectrum of an alkyd resin and that corresponding to the blank (extract of pure cotton in ethanol). Asterisks mark signals probably affected by those of the blank; **f**, **g** OCT tomograms collected before and after varnish removal, description of layers: (1) initial paint layer from 1884, (2) Van Gogh's original varnish, (3) later varnish layer from the 1961 restoration treatment; **h**, **i** OCT topography maps collected before and after varnish removal; **j** OCT differential map showing amount of removed material

[8] Regalrez® 1094 is a highly stable, low molecular weight, non-polar (hydrocarbon) resin, which for this purpose was dissolved in Shellsol D 40®, a mixture of paraffinic and naphthenic hydrocarbons C9-C11 (1 weight part : 9 volume parts).

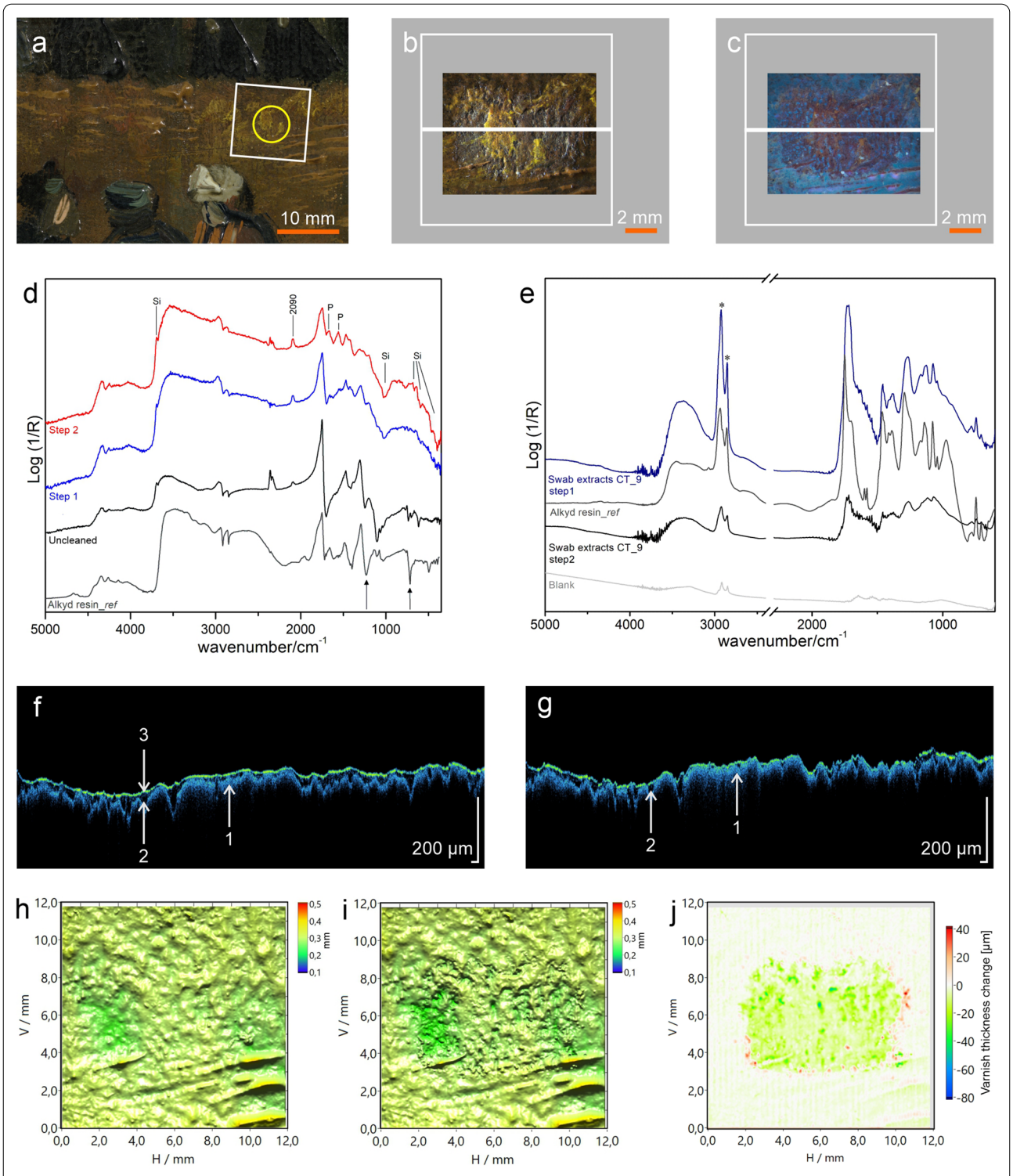

Fig. 7 (See legend on previous page.)

Fig. 8 *Congregation Leaving the Reformed Church in Nuenen*, Vincent van Gogh, 1884–85, oil on canvas, 41.5 cm × 32.2 cm, Van Gogh Museum, Amsterdam (Vincent van Gogh Foundation). Left: visible light photography, after restoration treatment; right: UV-induced fluorescence imaging, after restoration treatment

selective action of the solvent restricted to the non-original varnish was confirmed. This allowed fine-tuning of apt cleaning strategies for the restoration treatment that would re-establish a balanced appearance of the painting regarding its surface gloss and saturation of colours.

Abbreviations

OCT: Optical coherence tomography.; FTIR spectroscopy: Fourier transform infrared spectroscopy.; FTIR-ATR: Fourier transform infrared spectrometry by attenuated total reflectance.; XRF: X-ray fluorescence; OM: Optical microscopy; SEM-EDS: Scanning electron microscopy with energy dispersive X-ray spectroscopy; BEI: Backscattered electron imaging.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1186/s40494-022-00789-0.

Fig. S1. Congregation Leaving the Reformed Church in Nuenen, spots of examination by portable X-ray fluorescence spectrometer. **Table S1.** Identification of elements by means of X-ray fluorescence spectrometry.

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Author contributions

MI contributed to the concept and writing of the paper; the acquisition, analysis and interpretation of OCT data, and has drafted the paper and approved the submitted version. PM contributed to the acquisition, analysis and interpretation of the FTIR data collected in situ and in laboratory on the cotton swabs, and has drafted the paper and approved the submitted version. KP examined the painting technique and condition of *Congregation Leaving the Reformed Church in Nuenen*, initiated and oversaw the scientific investigation and analysis in her role as project leader, and carried out the (monitored) cleaning tests as well as the actual restoration of the painting. She contributed to the concept, writing and revision of this paper and approved the submitted version. BD contributed to the acquisition of the FTIR data collected in situ, revised the paper and approved the submitted version. LC and CM contributed to the development of the methodology for the non-invasive cleaning monitoring by portable FTIR and for the laboratory analyses of the cotton swabs, they have revised the paper and approved the submitted version. MG performed and interpreted the XRF-analysis on the painting, and the OM and SEM-EDS on the paint sample. She contributed to the writing of the paper and approved the submitted version. SG performed FTIR-ATR analysis on the paint sample. She contributed to the writing of the paper and approved the submitted version. PT contributed to the acquisition, analysis and creation of new software used for OCT data as well as to the concept, writing and revision of this paper and approved the submitted version. All authors have agreed both to be personally accountable for the authors' own contributions and to ensure that questions related to the accuracy or integrity of any part of the work, even ones in which the author was not personally involved, are appropriately investigated, resolved, and the resolution documented in the literature. All authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

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