
Pigment-Medium Interactions in Oil Paint Films Containing Lead-based Pigments

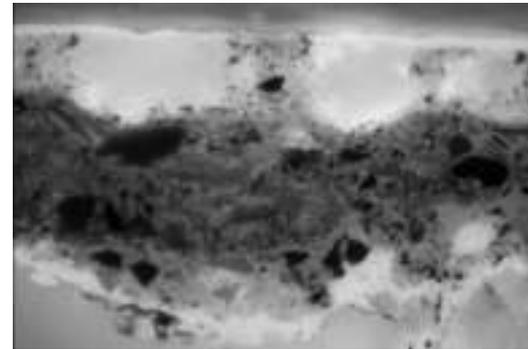
During the routine examination of paintings, translucent white lumps or inclusions are often observed in oil paints containing lead-based pigments, including red lead and lead-tin yellow 'type I.' These lumps vary in size, but are usually most easily visible under the microscope, either in cross-sections of paint samples or on the paint surface (Figure 1).

Figure 1 Lorenzo Costa, *A Concert* (NG 2486), c.1485–95. Poplar, 95.3 x 75.6 cm. Detail of the woman's sleeve and green bodice. The lumpy texture of the green paint is caused by inclusions in the lead-tin yellow underpaint.



In an example from Moretto da Brescia's *Virgin and Child with Saints Hippolytus and Catherine of Alexandria* from the National Gallery (NG 1165) of about 1538–40 (Figure 2a), large white inclusions with an opalescent appearance can be seen in cross-section in the red lead and vermilion mix that

Figure 2a and b Moretto da Brescia, *The Madonna and Child with Saints Hippolytus and Catherine of Alexandria*. Paint cross-section from the red hose of Saint Hippolytus. Two large white inclusions can be seen in the uppermost red layer, which contains red lead and vermilion. 2a. Normal light, 2b. ultraviolet light.



makes up the red of St Hippolytus's hose. These inclusions are seen to fluoresce when illuminated with ultraviolet light (Figure 2b). The inclusions may sometimes be large enough to be seen with the naked eye and appear as pustules that protrude through the surface of the paint. In some cases they may be visible in the X-radiograph of a painting.

Inclusions are quite commonly seen in the red ground layers of seventeenth-century Dutch paintings, which often contain some red lead (as a drier) mixed with red earth pigment. An example of this was observed on a painting by Bartholomeus van Bassen (*An Imaginary Church*, The Royal Pavilion, Libraries and Museums, Brighton and Hove, UK).¹ In a cross-section (Figure 3), one particularly large inclusion is visible in the red ground layer, which has erupted through the upper layers of paint, giving the whole painting surface a pronounced gritty texture. Unreacted red lead particles surround the white translucent pustule.

Figure 3 Bartholomeus van Bassen, *An Imaginary Church*, 1627. Brighton Museum and Art Gallery. Paint cross-section from the brown foreground. The lower red ground layer contains red earth and red lead, over which is a second brownish-grey ground layer (lead white, black, brown, and red lead). A very large inclusion originating in the lower red ground layer is visible.



The same phenomenon has also been observed in samples from wall paintings executed in an oil medium.² It is related to, but mechanistically different from the dramatic lightening of red lead-containing paint films due to conversion to lead carbonate.³ The conversion to lead carbonate is frequently seen in wall paintings due to the more extreme (often very

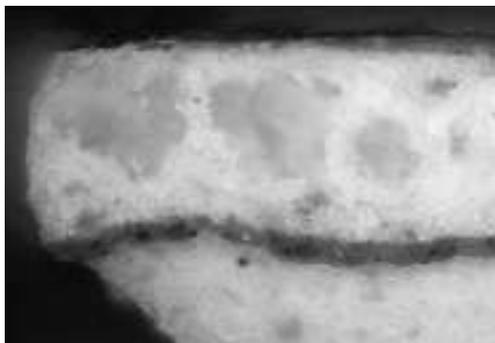
damp) environmental conditions to which they are exposed. This conversion occurs not only in oil, but also in a variety of binding media.

Inclusions have been noted in descriptions of paint samples published as early as the 1970s.⁴ They have variously been interpreted as interstices or ‘bubbles’ within the film resulting from the use of an aqueous binding medium such as egg tempera, or as indicative of the use of a mixed medium or emulsion (with the inclusions being protein or other non-glyceride material).^{4,5} It has also been suggested that the lumps are a coarse grade of lead white deliberately added to the paint to give it texture.⁶ It is however, only relatively recently, as a result of the availability of Fourier transform infrared (FTIR) microscopy and other analytical techniques, that it has been possible to analyse them reliably, and a number of studies have been undertaken.⁷

This article summarises the findings of our recent studies of the phenomenon undertaken at the National Gallery in London. (Full details of this first large-scale study to provide direct evidence for the nature of inclusions have been published in the *Technical Bulletin*.^{8,9}) Previous studies have tended to focus on Northern European seventeenth-century works, but our study demonstrates that the phenomenon is not confined to this period, but is ubiquitous in oil paintings from all over Europe during the period in which lead-based pigments including red lead, Pb_3O_4 , and lead-tin yellow ‘type I,’ Pb_2SnO_4 , were used as pigments.

Detailed analyses were carried out on samples from some 35 paintings, ranging in date from the thirteenth to the eighteenth centuries. The study examined a larger group of paintings than had previously been examined, with the aim of providing a broader view of the occurrence of lead soap inclusions and hence a deeper understanding of the mechanism and consequences of their formation. The samples were analysed using optical microscopy, energy dispersive X-ray analysis (EDX) in the scanning electron microscope (SEM), X-ray diffraction (XRD), FTIR microscopy, and gas chromatography–mass spectrometry (GC–MS). In ad-

Figure 4 The Virgin and Child with Saint John, *German School, 16th century, reverse of the Master of the Saint Bartholomew Altarpiece, Saints Peter and Dorothy (NG 707)*. Cross-section from the highlight of the Virgin’s crown. Large translucent white inclusions are visible within the lead-tin yellow paint layer.



dition, the records of examination of cross-sections held in the Scientific Department of the National Gallery (which date back to the 1950s) demonstrate that these inclusions are particularly common in paint films containing a significant proportion of red lead or lead-tin yellow ‘type I.’

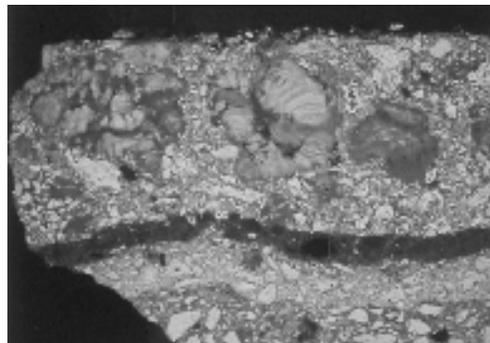
Microscopic appearance and analysis of the inclusions

Figure 4 shows a cross-section of a sample from a painting of *The Virgin and Child with Saint John* by an unknown sixteenth-century German painter, on the reverse of the *Saints Peter and Dorothy* (NG 707) panel of about 1505–10 by the Master of the Saint Bartholomew Altarpiece. Rounded white opalescent inclusions between 30 and 50 microns in size are visible in a yellow paint layer containing only lead-tin yellow of the ‘type I’ form. Again, as with the sample from the painting by Moretto, the inclusions fluoresce under ultraviolet light and can be seen to be inhomogeneous, with variations in the strength of the fluorescence. This inhomogeneity is even clearer in the back-scattered image (BSI) in the SEM (Figure 5) and distinguishes the inclusions from ordinary coarse particles of lead white that might have been deliberately added to the paint.

EDX analysis in the SEM detected only lead in the inclusions, a result that was true for all of the inclusions examined, even when they were present in lead-tin yellow-containing paints such as this. As in many of the other examples, more highly scattering lead-rich regions with a lamellar structure are visible (which appear lighter in the BSI), usually in the centre of the inclusion, surrounded by less scattering areas that correspond to the regions which fluoresce more strongly under ultraviolet light.

That these lead-containing inclusions only occur when lead-based pigments such as red lead or lead-tin yellow are bound in oil films provides the first clue to the nature of these particles, suggesting that a reaction between the lead-containing pigment and the oil medium might play a part in their formation. FTIR analysis of the inclusions gave remarkably consistent results and showed that the composition of the inclusions was very similar in all the examples studied. In every case they were found to comprise lead carboxylates (lead fatty acid soaps) and lead carbonate (in the basic and/or neutral form), as has also been reported

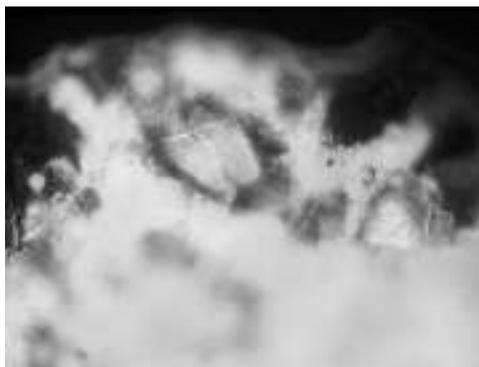
Figure 5 Reverse of NG 707. Back-scattered electron image of the cross-section from the Virgin’s crown (shown in Figure 3).



by other researchers.¹⁰ Using a FTIR microscope and a diamond micro-compression cell, it was possible to obtain good-quality transmission spectra of the inclusions. The lead soaps were identified by comparison with the literature and standards of various lead soaps prepared in the laboratory.^{8,11} Where the inclusions were very homogenous and quite transparent, and interference from other components such as lead carbonate and the oil binding medium was minimal, the lead soaps could be identified as those of palmitic and stearic acids (palmitic and stearic acids are the major monocarboxylic fatty acid components of aged drying oils).¹²

While the results are very consistent, there was some indication from the FTIR spectra evidence that the amount of lead carbonate in the inclusions is variable, as might be expected given the variation in translucency observed in cross-sections under the microscope. The distribution of the components in the inclusions was investigated by FTIR microscopy on a sample of lead-tin yellow paint from Lorenzo Costa's *A Concert* (NG 2486), c.1485-95, where the inclusions are relatively large (Figure 1). The inclusion analysed has a fairly opaque centre with a halo which is more translucent and which also fluoresces more strongly in ultraviolet light (Figure 6). FTIR microscopy demonstrated that the haloes are rich in lead fatty acid soaps while the more opaque centres of the inclusions are rich in lead carbonate.

Figure 6 Lorenzo Costa, *A Concert*. Unmounted paint fragment from a lead-tin yellow highlight on the brocade of the woman's sleeve. Translucent 'haloes' can be seen around a more opaque core in the inclusions.



The lead carbonate in Costa's *Concert* is present in the basic or hydrocerussite form, $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$, as was found to be the case in the majority of the samples examined.

Thermally-assisted transmethylation GC-MS was used to investigate the presence of fatty acids and dicarboxylic acids (produced by oxidative degradation of a drying oil medium) in the inclusions and surrounding paint.^{12,13} Drying oils are rich in the stable, fully saturated monocarboxylic fatty acids palmitic and stearic acids. In addition, fresh drying oils contain unsaturated fatty acids that, because of their double bonds, are reactive and can cross-link up to form polymeric material that allows the oil films to set or 'dry.' These unsaturated fatty acids can also breakdown, on reaction with oxygen, to form smaller molecules, including the dicarboxylic

acid azelaic acid, that can be detected in abundance in aged oil films by GC-MS. Analysis of inclusions separated from the bulk of the paint layer indicated that they contain palmitic and stearic acids (or their derivatives) but generally very little azelaic acid was found. This finding is consistent with the FTIR spectra where there was no indication of the presence of lead azelate.¹⁴ In some cases where inclusions form a large part of the paint layer, GC-MS results for the paint sample as a whole also show a reduced azelaic acid content.

The discovery that the presence of lead soap inclusions can have this effect on the fatty acid ratios measured by GC is particularly significant since a low azelaic to palmitic acid ratio is characteristic of non-drying fats such as those in egg.¹² On the basis of a low azelate level, some previous analyses of the binding media of red lead-containing paint films (before the era of FTIR microscopy) concluded that medium contained egg yolk.¹⁵ If the binding medium does indeed contain egg yolk it must, of course, contain protein, which can now be detected by FTIR microscopy.¹⁶ In all of the cases where GC-MS gave a low azelaic to palmitic acid ratio, FTIR microscopy was used to check for the presence of protein, but none was detected. The 'low azelate effect' seems to be associated with the presence of the inclusions and must be partly responsible for belief that inclusions are globules of protein (emulsion).

Discussion

Red lead (Pb_3O_4) in linseed oil has been extensively studied because of its use as a corrosion-inhibiting paint for iron.¹⁷ It has therefore long been known that lead ions in red lead react with the fatty acids in linseed oil to form lead soaps. Our most recent systematic study of the reaction rates of lead salts with fatty acids has shown that other lead-based pigments and salts can also react to form metal soaps, but that reaction rates vary.⁹ For example, lead white (basic lead carbonate) can also react with fatty acids, but the reaction is much slower than for red lead, and indeed no inclusions were found in lead white-containing paint layers in the works examined in the London study, and only low levels of lead soaps were detected by FTIR, spread throughout the films.⁸

The component of the paint that is responsible for lead soap formation is not always immediately obvious. It may not be the major constituent of the mixture, or the only lead-containing species present. In Francisco Zurbarán's painting of *A Cup of Water and a Rose on a Silver Plate* (NG 6566) c.1630, the warm grey shadows of the white cup contain lead soap inclusions. The major component of this paint is lead white, but it also contains some lead-tin yellow, yellow earth, and black. There are more inclusions in the areas depicting the shadows of the cup than in the whiter highlights, suggesting that it is the lead-tin yellow, rather than the lead white, that is responsible for the formation of the inclusions. In the red ground layers of many seventeenth-century Dutch paintings (see Figure 3), inclusions are seen which seem to derive from the small amount of red lead added to the red earth pigment as a drier and of which little or nothing remains.

Recent work suggests that impurities in certain pigments, linked to their method of manufacture, may in fact be responsible for soap formation.¹⁸ Lead-tin yellow 'type I' (Pb_2SnO_4) was traditionally made by heating lead and tin oxides or lead and tin metals and adding red lead with further heating. There is evidence that in the traditionally manufactured pigment there is often unreacted lead oxides or tin oxides in the resulting pigment.¹⁹ It seems likely that it is the lead oxide component in the pigment that forms soaps, rather than Pb_2SnO_4 itself.¹⁸ If lead-tin yellow itself does not react, or reacts very slowly, this would suggest that lead oxide impurities must have been common as the presence of inclusions in lead-tin yellow-containing paints is ubiquitous and almost characteristic. In a similar way, lead white traditionally produced by the 'Dutch' or 'stack' process may contain unconverted lead acetate, and it is possible that it is the presence of lead acetate or other lead salts that are responsible for the reported examples of inclusions in lead white films.²⁰ Lead white itself does not appear to react significantly, while lead acetate and basic lead acetate react rapidly with fatty acids.⁹

By the nineteenth century, when the use of red lead was much reduced and lead-tin yellow had become obsolete, a large number of other lead-containing materials were being added to paint, primarily to improve its handling or drying properties.²¹ It seems likely that most of the occurrences of lead soap inclusions observed in nineteenth- and twentieth-century paintings derive from the interaction of these, often very soluble, lead compounds with the oil medium. For example, lead acetate (sugar of lead) was added to paint layers that now show paint defects, including ground staining, blooming, and inclusions.²² Zinc-containing pigments, which had been introduced by the nineteenth century, also readily react with fatty acids to form zinc soaps.²³

While pure lead white does not appear to form inclusions, from the analyses undertaken in London, it is clear that most inclusions contain lead carbonate in addition to lead carboxylates. Our studies conducted at the National Gallery using test films have indicated that, in the presence of carbon dioxide and under conditions of high relative humidity (70% RH or above), red lead in any binding medium can be converted to basic lead carbonate.³ It is possible that a reaction between red lead or components of red lead (and by analogy other lead salts or their components) and carbon dioxide is occurring in parallel with the formation of lead soaps, yielding the lead carbonate found in the inclusions. It is also possible, however, that only lead soaps are formed initially and that these go on to react with carbon dioxide to form lead carbonate. On balance, it seems likely that the lead carbonate associated with the inclusions forms via the lead soaps, because of the lamellar structure seen in some of the larger inclusions, which suggests that it is 'precipitating' from the lead carboxylate.

Exactly how and why lead soap inclusions form is still not fully understood. In a well-prepared oil paint film, pigment particles will be uniformly dispersed throughout the film. It might therefore be expected that lead soaps will also remain evenly spread throughout the film. However, the lead soaps

in many paintings have formed characteristic pustules or agglomerations. For the inclusions to form there must be slow migration of material through the paint film, leading to the formation of coagulated masses of lead soaps. This migration, and the subsequent growth of the inclusion, has in many cases led to distortion of the surrounding paint layers. It is also not clear how quickly the inclusions form.

While the formation of blooms on the surface of paintings indicates that materials can move through paint films, it is not clear what drives the migration and aggregation.^{23,24} Changes in polarity of film during ageing may lead to incompatibility between the oil matrix and more mobile components such as saturated fatty acids and their soaps, causing a phase separation.^{22,24} There is also evidence that there may be a 'concentration factor' involved, with migration only occurring once fatty acids or metal carboxylates have reached a critical level. The high degree of intermolecular order that is likely to exist within the inclusions perhaps also contributes to the driving force that separates the metal soaps from the more amorphous oil film matrix.^{14,25}

The main components of the inclusions have been shown to be the metal soaps of palmitic and stearic acid (both saturated monocarboxylic acids). The absence of other lead soaps, including lead azelate, from inclusions may also be linked to the intermolecular order in the inclusions which makes their incorporation into the inclusions unfavourable since they will not be readily compatible with the ordered lamellar structure that is likely to exist in regions containing long chain monocarboxylate soaps. Alternatively, the absence may simply be linked to hydrophobicity or the mobility of the fatty acid components – lead soaps of palmitic and stearic acids are expected to be much more mobile than lead soaps of azelaic acid, which might explain the lowered azelate levels noted earlier. Thus areas in a film containing pustules will become enriched in palmitate and stearate because of the migration of these species into the inclusion from elsewhere in the film.

Conclusions

A number of important conclusions can be drawn from this study. The translucent inclusions present in oil films containing red lead, lead-tin yellow, and some other lead-based materials, are comprised of lead fatty acid soaps and lead carbonate, formed as a result of reaction of the pigment with the oil binding medium. Such inclusions have been found in paintings from a broad range of geographical locations and dates, demonstrating the widespread nature of this phenomenon.

The lead soap inclusions are not likely to be a deliberate addition to the original paint, as they serve no obvious purpose; they would have siccative properties but the lead pigments with which they are found are themselves good driers. The coarse and lumpy texture of the paint where inclusions are large is unlikely to be a deliberate effect intended by the artist, as has sometimes been thought and they are often encountered in layers that would not be visible. Instead, the inclusions will have formed over a period of time, after the painting was completed, by migration and agglomeration of the lead fatty acid soaps.

An understanding of this reaction, and its effect on fatty acid ratios, has consequences for the interpretation of the results of analysis of the binding medium, particularly if GC-MS is the only analytical technique employed. The presence of inclusions within a paint layer has been shown to affect the fatty acid ratios. Low levels of azelaic acid have regularly been found, which could lead (and has led in the past) to the erroneous conclusion that the binding medium of the paint is egg tempera, or that a mixed medium or emulsion has been used. FTIR microscopy has, however, confirmed that none of the samples examined in this study contain protein.

Inclusions may also pose a problem during cleaning of paintings, as the rather soft waxy lead soaps are vulnerable to mechanical damage. This is evident in Moretto's *Virgin and Child with Saints* (NG 1165) where the tops of the pustules have been flattened. However, lead soaps do not seem to be particularly soluble in commonly used cleaning agents. The rough, gritty surface created by the inclusions can also cause problems during varnishing. Dirt sometimes becomes trapped in the soft lead soaps when they are exposed at the paint surface, which can be visually disturbing in light areas of paint; the white spots created by exposed pustules in dark paint are similarly very noticeable. Thus, a better understanding of the origin and effects of lead soap inclusions is important for the interpretation and treatment of the wide range of paintings that demonstrate this phenomenon.

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