Chapter 23
Efflorescence on the Paintings of Edwin Austin Abbey: Examination, Analysis, and Cleaning of Surface Bloom on *The Spirit of Light*

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23.1 Introduction

The American artist Edwin Austin Abbey (1852–1911) began his career in New York City as an illustrator for *Harper's Weekly*, and first began oil painting in 1889 after relocating permanently to England. Passionate about the intersection of architecture, painting, and sculpture, Abbey completed several mural commissions throughout his 20-year painting career, most notably for the Delivery Room of the Boston Public Library and the rotunda of the Pennsylvania State Capitol Building in Harrisburg. Abbey painted these mural commissions in his purpose-built studio in Gloucestershire, England, which he shared for several years with fellow American painter John Singer Sargent [13].

A crucial component of Abbey's artistic process was the production of numerous sketches both before and during the painting of the final composition. More than 600 of Abbey's paintings, mainly oil sketches, were given to the Yale University Art Gallery (YUAG) in 1937 along with the contents of his studio. Many of these paintings are unvarnished or selectively varnished preparatory studies for mural commissions and, rarely exhibited or treated, over half of these paintings exhibit efflorescence.

This study examines the relationship between Abbey's materials and efflorescence formation on the oil-on-canvas study *The Spirit of Light* (Fig. 23.1a). This work was painted in England between 1902–1908 as a preparatory study for a lunette in the rotunda of the Pennsylvania State Capitol Building. The lunette depicts

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Fig. 23.1 *The Spirit of Light* before treatment in (**a**) visible light and (**b**) ultraviolet light (370 nm). Photo Credit: Yale University Art Gallery

several classically garbed women floating upwards into a sky filled with silhouetted oil derricks. As one of four lunettes celebrating Pennsylvania history and industry, *The Spirit of Light* conveys the power and wonder of electricity to create light [18]. In this preparatory figure study, Abbey focuses on capturing the graceful positioning and soaring upward movement of the two figures at the far right of the composition.

Severe efflorescence was present on dark passages of the painting, obscuring the composition and drastically altering the tonality and sheen of the work. The formation of efflorescent material corresponds to the location of selective, artist-applied coatings that visibly fluoresce under ultraviolet excitation (Fig. 23.1b). In preparation for treatment, research was conducted to identify the composition of the efflorescence and to examine its relationship to the locally applied varnishes and the materials within the stratigraphy of the painting. The findings yielded discoveries about Abbey's painting technique and significantly informed the subsequent cleaning of the painting.

23.2 Painting Materials and Technique

The Spirit of Light was painted on a linen canvas that was commercially primed with two off-white ground layers. The first ground layer is chalk-based and the second contains lithopone, or coprecipitated barium sulfate and zinc sulfide.[1] Like many of Abbey's preparatory studies, the oil paint layers were applied broadly and freely with diverse handling that ranges from thicker impasto to sweeping, dripping washes of thinned paint.[2] Abbey worked both wet-into-wet and wet-into-dry, scraping or scumbling in areas of compositional change. Paint application indicates that this study was worked and modified over a period of at least several months.

In addition to using both additive and subtractive painting techniques to achieve his aesthetic goals, Abbey appears to have mixed resins and oil-based varnishes into his paint, as well as applying them in discrete layers. This seemingly spontaneous and highly variable mixing is visible in the different intensities of fluorescence seen within and on top of the paint layers when viewed under ultraviolet light. The cross-sectional sample reveals Abbey's prevalent use of earth pigments in the background and shows the application of an oil-based coating on top of still wet paint layers (Fig. 23.2a, b). Lead sulfate was identified in this coating layer (Fig. 23.2c).[3]

Abbey's use of retouching varnishes, both soft and hard resins, and driers is well-documented in the Roberson Archives at the Hamilton Kerr Institute. Abbey's account from 1884–1904 records his frequent purchase of Vibert's Vernis à Peindre and Vernis à Retoucher in addition to the driers Siccatif de Courtrai and Siccatif de Haarlem [1].[4] Based upon observations of his painting technique, Abbey may have

[1] Characterization of the ground layers was conducted with Fourier transform infrared spectroscopy (FTIR) and field emission-scanning electron microscopy/energy dispersive spectroscopy (FE-SEM/EDS). Dispersed samples of the ground layers were analyzed with FTIR on a single diamond window using a Thermo Continuum infrared microscope coupled to a Nicolet 6700 bench-top spectrometer in transmission mode with the MCT-B detector. SEM-EDS was performed on a cross-sectional sample embedded in Bio Plastic™ resin and polished with Micro-Mesh® abrasive sheets. The sample was carbon coated and analyzed with a Hitachi SU8230 UHR Cold Field Emission SEM with a Bruker XFlash 5060FQ Annular EDS detector in back-scattered electron mode.

[2] The binding medium and coating material were analyzed using the RAdICAL + ESCAPE approach by pyrolysis/gas chromatography/mass spectrometry (Py/GC/MS) with thermally assisted hydrolysis methylation using tetramethylammonium hydroxide after [17].

[3] Lead was detected in the coating layer in the cross-sectional sample with FE-SEM/EDS. Lead sulfate was identified in a dispersed sample of the coating analyzed with FTIR. The coating was determined to be composed of a drying oil using Py/GC/MS.

[4] Siccatif de Courtrai and Siccatif de Haarlem are reported to be composed of copal dissolved in a solvent such as oil of spike or turpentine. Siccatif de Courtrai was noted to contain lead compounds to accelerate drying [4].

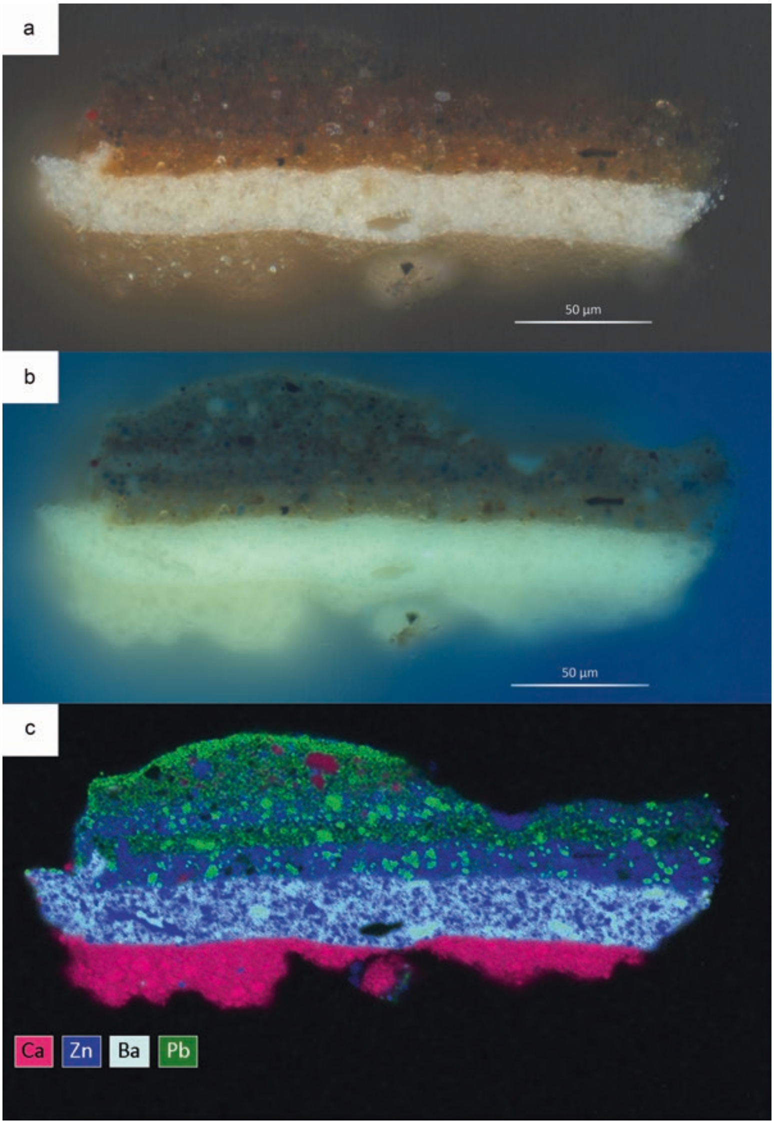

Fig. 23.2 Cross-section from brown background (**a**) epi visible darkfield mode (**b**) UV-induced visible autofluorescence mode (**c**) False color elemental map, 20 kV, 20 mA: calcium = pink, zinc = blue, barium = white, lead = green

layered and added these resins and driers freely to his paint and coating layers as he worked.

23.3 Visual Characteristics, Morphology, and Identification of Efflorescent Material

The efflorescence on *The Spirit of Light* closely corresponds to the distribution of a selectively applied oil-based coating, forming in drips and splatters. Under magnification, the efflorescent material appears white or colorless and crystalline, and has the greatest thickness in the valleys of the canvas weave (Fig. 23.3a). Although it appears powdery, it is tenaciously held to the surface and cannot be brushed away.

In order to observe the morphology of the efflorescent material, the top surface of a paint flake from the varnished brown background was imaged with SEM.[5] Imaging revealed rounded plates emerging from the varnished surface. The plates are homogenous in size, approximately 1–3 μm wide and 75–100 nm thick (Fig. 23.3b). There is a lower layer of plates parallel to the surface and an upper layer of lifted plates (Fig. 23.3c). Diffuse reflection of light by the irregular surface of the upper layer of plates causes the white appearance of the efflorescence. EDS determined that the plates contain carbon and zinc (Fig. 23.3d).

FTIR analysis of scrapings of efflorescent material revealed the presence of crystalline zinc carboxylates, confirmed by comparison with a reference sample of zinc stearate (Fig. 23.3e).[6] The crystalline species are characterized by a sharp COO^- band at 1538 cm^{-1} [3]. The sharp band observed at 1536 cm^{-1} in the spectrum of efflorescent material from *The Spirit of Light* indicates the presence of crystalline zinc carboxylates. Baij et al. propose a process in which free fatty acids in the binding medium combine with metal ions to form amorphous complexes, which eventually crystallize as metal-soap aggregates [3]. None of the carboxylate band characteristic of amorphous zinc soaps (1585 cm^{-1}) is discernable in the spectra collected from efflorescent material from *The Spirit of Light* [3].

The organic components of the efflorescence were identified with GC/MS as primarily stearic and palmitic acids, with some azelaic acid, present as metal carboxylates; trace levels of these same fatty acids were detected in their free form.[7] Phosphate was also identified in the efflorescent material.

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[5] A paint flake sample was coated with carbon and analyzed with FE-SEM/EDS in secondary electron mode.

[6] The zinc stearate reference sample (purum, 10–12% Zn basis) was purchased from Sigma Aldrich, St. Louis, MO.

[7] Interpretation of the GC/MS results for efflorescence scrapings supports the proposed composition of metal carboxylates of stearic acid, palmitic acid, and azelaic acid, inorganic phosphate, as well as small amounts of free fatty acids. The analysis began with a two-step extraction and derivatization procedure and was created to fit the sample size limitations using ideas in [9, 16],

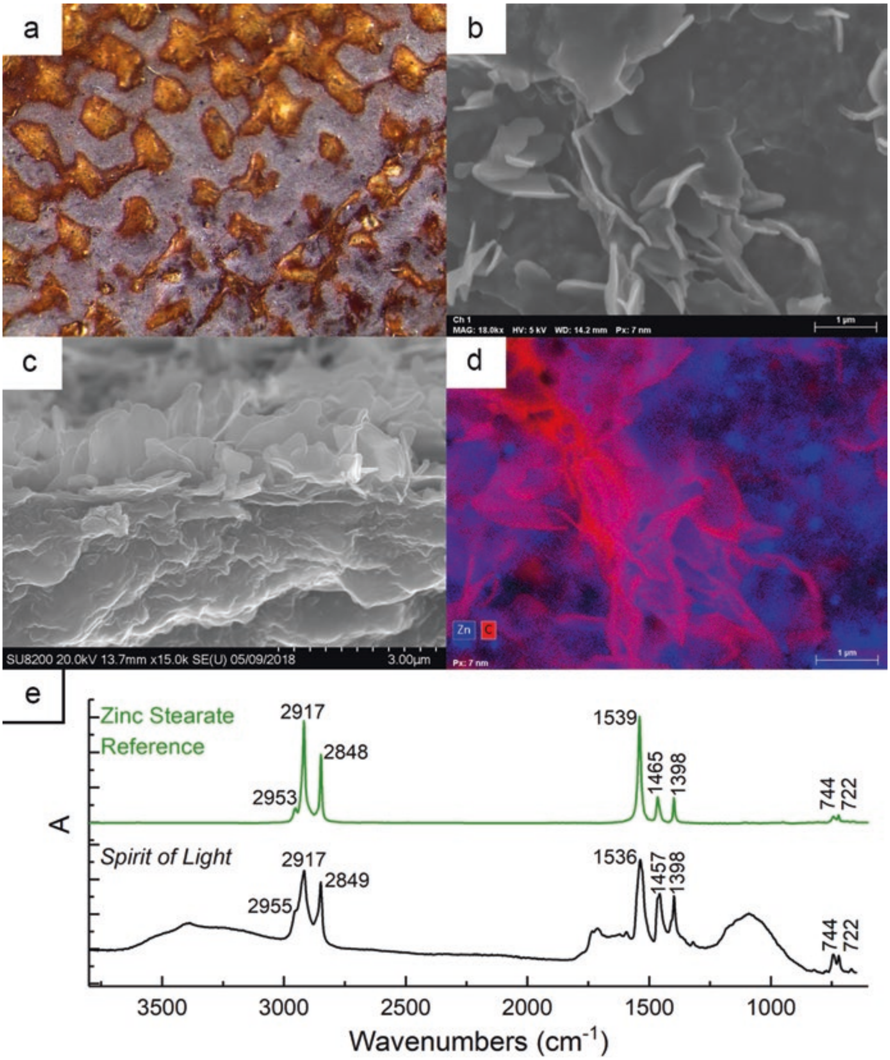

Fig. 23.3 (**a**) The efflorescence photographed at 16x (**b**) Secondary Electron Image (SEI) of efflorescence on top surface of paint chip, 15 kV, 18,000x (**c**) SEI of efflorescence in cross-sectional orientation, 20 kV, 15,000x (**d**) EDS false color elemental map of efflorescence plates, 5 kV, 18,000x: zinc = blue, carbon = red (**e**) FTIR spectra of zinc stearate reference sample (top) and efflorescence sample from *The Spirit of Light* (bottom)

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and [14]. Efflorescence samples were dissolved in a minimum volume of pyridine and analyzed for free fatty acid content. The remainder was then derivatized using a minimum volume of *N,O*-bis(trimethylsilyl)trifluoroacetamide for maximum conversion, and analyzed for trimethylsilylated carboxylates. The derivatized sample results included both the original free fatty acids and fatty acids present as metal carboxylates. An Agilent 7890A/5975C GC/MS system with a DB-5MS-UI column (30 m × 0.25 mm × 0.25 μm) and a CombiPAL autosampler was used for

23.4 Cleaning Approaches

The thin and underbound paint layers of *The Spirit of Light* are highly soluble and easily burnished, presenting a challenging surface from which to clean the disfiguring efflorescence. The paint layers are soluble in aromatic and aliphatic solvents, polar solvents, and aqueous solutions (pH between 6 and 8 with or without the inclusion of chelators). It is possible that the underbound nature of the paint layers contributes to *The Spirit of Light's* high solubility. This property may be exacerbated by the hydrolysis of the oil film as evidenced by the formation of metal soap and fatty acid efflorescence on the surface. The efflorescence appeared soluble in the aromatic solvents xylene and Shellsol A100, with both neat and gelled applications of these solvents greatly reducing the appearance of the efflorescence. Gelling Shellsol A100 and xylene with Carbopol increased the cleaning efficacy by achieving longer dwell times, enabling the removal of thicker efflorescence in the valleys of the canvas weave. However, this system was not safe for much of the delicate surface.

The silicone emulsifiers Momentive Velvesil Plus and Shin-Etsu KSG-350Z and the silicone solvents decamethylcyclopentasiloxane (D5) and octamethylcyclotetrasiloxane (D4) successfully reduced the appearance of the efflorescence without solubilizing the sensitive paint layers [5, 10]. Shin-Etsu KSG-350Z was preferred because of its wider commercial availability. The emulsifier was applied to the surface and agitated for at least 1 minute with a brush. This agitation step was crucial in evenly thinning the efflorescence in the valleys of the canvas weave (Fig. 23.4). The gel was cleared from the surface with D5 solvent. The addition of small percentages of aqueous chelating solutions and non-miscible aromatic solvents such as benzyl alcohol to form emulsions did not increase the efficacy of the cleaning.

This cleaning system is promising for paintings with sensitive surfaces and presents several advantages. Cleaning with the KSG-350Z emulsifier and silicone solvents allowed the original coatings to remain intact and restored subtle variations in gloss and saturation to the surface. The insolubility of zinc carboxylates in D5 solvent suggests that the cleaning will not mobilize soaps in the stratigraphy of the painting, although the mobility of metal carboxylates in silicone solvents has not yet been evaluated. This system also presents some challenges, such as the long evaporation time of the D5 solvent and the ease with which one can create tidelines during cleaning. When applied to the surface, the D5 saturates the efflorescence, making it difficult to differentiate between cleaned and uncleaned areas. The potential for silicone emulsions to leave residues, especially in porous paint surfaces, is yet to be fully explored.

The cleaning mechanism of the emulsifier during the removal of crystalline zinc soap efflorescence is uncertain. Crystalline zinc soaps have low solubility in D4 and

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these analyses. The procedure and results will be described in more detail in a forthcoming publication.

Fig. 23.4 Detail of *The Spirit of Light* during cleaning with Shin-Etsu KSG-350Z

D5; therefore, the effectiveness of the KSG-350Z emulsifier is surprising.[8] It is possible that the emulsifier works on a primarily mechanical level to loosen, lift, and suspend the efflorescence flakes from the surface. It is also possible that the emulsifier flattens and redistributes the flakes across the surface.

23.5 Discussion and Conclusions

The selective distribution of zinc carboxylate efflorescence on *The Spirit of Light* suggests that efflorescence formation is closely related to the painting materials and the ways in which they were applied by the artist. The lithopone-containing upper ground layer is a potential source of zinc carboxylates, aggregates of which are visible in the cross-sectional sample and on the surface of the paint layers. Zinc

[8] The solubility of crystalline zinc stearate and stearic acid in D4, D5, and the aromatic hydrocarbon Shellsol A100 were evaluated. Both compounds were determined to be minimally soluble in siloxane-based solvents, suggesting that solubility was not a main factor in the efficacy of the Shin-Etsu KSG-350Z cleaning. Both compounds were soluble in Shellsol A100.

carboxylates in ground layers have been previously linked to the formation of inorganic surface crusts, migrating aggregates, and free fatty acid efflorescence, but few studies have discussed the presence of efflorescence composed primarily of crystalline zinc carboxylates [7, 15].[9]

The potential presence of lead and zinc driers in the ground, paint, and coating layers may also be significant factors in efflorescence formation on *The Spirit of Light.* The mobile and reactive nature of these compounds has been noted in studies of nineteenth-century oil sketches executed on commercially primed canvases [19]. Archival research, visual observation, and analysis suggest that Abbey used commercially primed canvases and freely interlayered and mixed resins and driers into his paints. Mrs. Abbey noted that her husband "never went at it wet" and quoted him stating, "Sargent[ʻs] work [will] never stand" [11]. This may suggest that Abbey readily used driers to paint works that he believed would remain stable with age. His desire to use stable painting techniques, perhaps compensating for his lack of formal training, may have led Abbey to use unsound painting practices.

In contemplating the unique relationship between the coating and the efflorescence, it is possible that zinc soaps formed within and on top of the varnish layer as a result of the reaction of lead and zinc driers with each other and with migrating free fatty acids: a phenomenon that has been noted in primary sources and could explain the presence of lead sulfate in the coating layer.[10] The formation of the stable compound lead sulfate within the coating layer, thereby providing few available lead ions, could explain the surprising absence of lead soaps. It is also possible that zinc carboxylates identified in the ground layer were mobilized by the varnish solvent, resulting in their migration to the surface. The identification of phosphate, possibly from bone black pigments, in samples of efflorescent material suggest that the composition of the paint layers may also contribute to efflorescence formation [12]. The impact of potentially high moisture environmental conditions in Abbey's studio and in museum storage could also be a contributing factor in efflorescence formation [2].

Imaging of the efflorescence contributed to a greater understanding of the sensitive paint surface, and characterization of the efflorescence explained its solubility characteristics, thereby informing cleaning methodology and testing. The use of Shin-Etsu KSG-350Z and the silicone solvent D5 for the cleaning of efflorescent material has not been previously noted; their effectiveness at reducing the appearance of the efflorescence without solubilizing sensitive oil paint layers presents a promising avenue for future investigation. Future study will concentrate on imaging the surface of *The Spirit of Light* after cleaning and analyzing the composition of the emulsifier after cleaning. Further research is ongoing to establish the mechanism for the formation of crystalline zinc carboxylates on the surface of Abbey's paintings.

[9] A similar morphology was imaged by Jimenez and Chua, who identified hydroxychlorides, sulfates, and/or oxalates in efflorescent material on an oil painting containing zinc white [8].

[10] Field notes a phenomenon in which both lead acetate and zinc sulfate driers are incorporated into a coating and react to form lead sulfate and zinc acetate, with efflorescence eventually forming on the surface [6].

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