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The *Dalle de Verre* Lantern of Liverpool Metropolitan Cathedral: Composition and In-service Behaviour of the Epoxy Resin Mortar

ABSTRACT

Arising from questions concerning the long-term stability of the mortar, this research establishes the composition of the epoxy resin mortar used for the construction of the dalle de verre lantern of Liverpool Metropolitan Cathedral. Water ingress had manifested itself early in the life of the lantern as a result of flaws in the mortar. These defects are described and illustrated herein. The paper also describes the process of characterising the epoxy resin, quartz flour, and carbon black in the lantern mortar in order to prepare mock-ups for weathering assessments to gauge the mortar's future behaviour. The mortar specification was accomplished by archival research and personal interviews, complemented by laboratory analyses of lantern mortar samples using Fourier-transform infrared spectroscopy, Raman spectroscopy, scanning electron microscopy, and thermogravimetric analysis. The difficulties in sourcing sufficiently detailed documentary information on the original mortar components are discussed.

KEYWORDS

Dalle de verre • Stained glass • Liverpool Cathedral lantern • Epoxy resin mortar • Degradation

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INTRODUCTION

This paper presents research conducted to identify the precise composition of the epoxy resin mortar used for the glass lantern of Liverpool Metropolitan Cathedral. The cathedral was constructed between 1962 and 1967 to architect Sir Frederick Gibberd's 1959 competition-winning design. Its roof is crowned by a tapering lantern consisting of large *dalle de verre* panels, with coloured blocks of glass held together in a reinforced concrete structure by a mortar comprising epoxy resin, sand, and carbon black. The present study was motivated by the need for detailed information on each of the components of the mortar in connection with questions over its long-term, in-service stability and uncertainty about the possibility of deleterious consequences, including structural concerns. Ingress of rainwater through cracks in the mortar joints became evident soon after the construction of the lantern and has been of concern for much of its lifetime. This has given rise to the question of whether the problem is primarily due to defects in the mortar present from the outset or is exacerbated by degradation due to weathering. The need to address this issue by conducting accelerated ageing tests was thus a component of an extensive programme of research, surveying, and monitoring for the cathedral (Purcell UK 2022), supported by the Getty Foundation's 'Keeping it Modern' programme. An important prerequisite to the accelerated weathering experiments was the need to reproduce the original mortar composition as precisely as possible. This paper reports on the combination of archival research, personal interviews, and laboratory analyses undertaken to establish a recipe closely replicating the original mortar formulation.

CATHEDRAL AND LANTERN DESIGN AND CONSTRUCTION

The main structure of the cathedral and lantern is a reinforced concrete frame comprising 16 concrete ribs bound by three ring beams; a lower ring beam at the junction of the nave wall and roof, a middle ring beam at the junction of the roof and lantern, and an upper ring beam at the lantern roof level. The overall form of the lantern is that of a tapered drum sitting on the cathedral roof, surmounted by a crown (Figure 1). The lantern is approximately 21 m in diameter and 22.5 m in height.

Figure 1. The *dalle de verre* lantern crowning Liverpool Metropolitan Cathedral, viewed from the north. Courtesy of Purcell UK

From the middle ring beam, the ribs extend to an upper ring beam clad in white-grey Portland limestone, recessed from the face of the ribs and lighter in appearance. At lantern level, the 16 voids, or bays, between the ribs are infilled with panels comprising resin-bonded *dalle de verre* glass within a pre-cast concrete tracery overlaid with epoxy resin. Each bay is approximately 3.6 m in width, rising to a reinforced shallow-domed concrete roof. Twelve bays contain nine panels each, while the four sections that brace the tower contain 12 panels each. The panels vary in height between 1.2 and 2.4 m. Designed by John Piper and Patrick Reyntiens, the lantern's decorative scheme consists of 16 vertical sections with 30-mm-thick glass in red, yellow, and blue hues representing the Trinity, comprising a total area of approximately 1120 square metres (Figure 2).

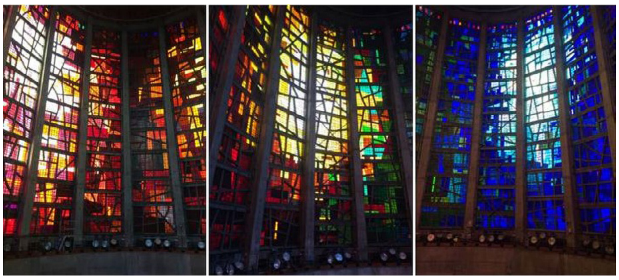

Figure 2. The lantern glass, showing the red, yellow, and blue zones, viewed from the internal rim balcony at the foot of the *dalle de verre* edifice. Courtesy of Norman Tennent

In the early 1960s, engineers and chemists collaborated to produce an epoxy resin to bond the glass slabs that would be strong enough to withstand the wind pressures the tower would experience. Tests were arranged at the National Physical Laboratory to prove that the resin was more permanent and stronger than concrete for cementing the glass. Testing informed the design and led to a decision that no area of cemented glass should exceed 1.5 m^2, resulting in each panel being subdivided by 10 cm square concrete ribs. The design was subject to further structural assessment at the cartoon stage, with the engineers working with Piper and Reyntiens to modify the structure of each panel to ensure stability. This process is observable in the film *Crown of Glass* (The Shell Film Unit 1967) which clearly shows the impact the engineers had on the original full-size cartoon layout, with Piper drawing in any extra ribs suggested by the engineering team.

The concrete panel frames were fabricated on steel tables overlaid with a polystyrene framework covered with polyethylene sheeting into which concrete was poured and the table vibrated to compact it. The glass *dalles de verre* were laid to the artists' design for each panel within the frame, and a resin mixture comprising epoxy resin, sand, and carbon black pigment was then applied in the gaps between the glass using a piping bag and over the concrete framing using a trowel. The resin between the glass *dalles* was applied in two layers and reinforced with strands of fibreglass, while the thick layer on the concrete was applied without reinforcement. The epoxy resin was finished with slate dust applied to the face before it had set to give the joints a matte appearance. Once complete, the polystyrene and plastic backing was removed. No cleaning or finishing was carried out to the back of the panels before they were lifted by tower crane and fitted within the main frame of the lantern (The Shell Film Unit 1967).

IN-SERVICE BEHAVIOUR OF THE EPOXY MORTAR

To date, the in-service behaviour of the epoxy has been unproblematic save for the ingress of rainwater through defects in the mortar at minor cracks, more significant gaps within the mortar, and, potentially, at voids at the epoxy/glass interface. Cracks and gaps within the mortar are readily visible from the building's exterior (Figures 3-5) and have, therefore, been well documented and quantified during the Purcell UK survey (2022). Any lack of epoxy-glass adhesion sufficient to allow water to penetrate into the cathedral is extremely difficult to observe and, as a result, the contribution of bonding defects to water ingress remains unquantified. The integrity of the individual *dalle de verre* slabs is excellent, so cracks in the glass are not contributing to water ingress.

Unfortunately, since this problem became apparent only a short time into the life of the cathedral, water management at the foot of the interior of the glass edifice has been a troublesome reality for several decades. In the 1990s, a polymer coating was applied extensively to the exterior of the lantern in order to seal defects in the mortar, but this attempt at remediation has had no long-term benefit. Figures 3-5 show the matt grey residues of this coating, thought to be a polyurethane elastomer. Whilst this failed polymer treatment merits further investigation, the primary concern which led to the current research was the possibility that degradation of the epoxy mortar and concomitant loss of adhesion at the epoxy-glass interface could eventually compromise the lantern's structural integrity. Accordingly, accelerated ageing tests, using mock-ups to explore long-term, in-service behaviour, were put in train. This paper is devoted to the research carried out to replicate the original mortar composition, a prerequisite to the weathering experiments. The results of these accelerated ageing assessments will be published in due course (Poulis et al. forthcoming).

Figure 3. Detail of the lantern's exterior showing one *dalle de verre* slab with a residual coating of the 1990s remedial polymer treatment and, surrounding it, the embedding epoxy mortar with a prominent gap at the upper right. Courtesy of Norman Tennent

Figure 4. Weathering of the 1990s polymer coating on the exterior of one *dalle de verre* slab. Courtesy of Norman Tennent

Figure 5. Exterior view of the glass-mortar interface at one *dalle de verre* slab, with a large adjacent mortar crack. Courtesy of Norman Tennent

HISTORICAL RESEARCH TO IDENTIFY THE COMPONENTS OF THE EPOXY MORTAR

The precise laboratory identification of epoxy resins used in past cultural heritage projects has been shown to be a very difficult scientific undertaking (Tesser, Lazzarini, and Bracci 2018; Tennent, de Groot, and Koob 2019). Therefore, in order to replicate the epoxy mortar accurately, extensive enquiries were made to locate documentation on the formulation used by Patrick Reyntiens, starting with the *Crown of Glass* film. In addition to the visual record of the viscous black epoxy being poured into the gaps between the *dalles de verre*, the film's commentary stated that an Epikote epoxy resin was used. Epikote is the brand name coined for Shell's epoxy resin range; however, the film does not refer to a specific product from within the range. Shell subsequently divested of its epoxy business and further company takeovers have taken place since, adding to the difficulty of locating further information on the purported use of Epikote epoxy resin for the lantern construction. Hexion is the company currently marketing the Epikote brand, but the possibility of locating information on the precise Epikote resin mentioned in the *Crown of Glass* film, by means of what was likely to be a tortuous sequence of investigations within international company records, was considered so unlikely to be successful that this route was not explored further. Searches in the archives of Liverpool Metropolitan Cathedral and of Gibberd Architects, the firm founded by Sir Frederick Gibberd, proved fruitless. However, with the knowledge that Patrick Reyntiens was still fit and active in 2019, contact was established with his daughter Edith and then, through her, with his son John, also a stained-glass artist. John Reyntiens was certain that his father would have no records or a precise recollection of the epoxy formulation used but suggested contacting David Kirby, project manager for the lantern construction, who indeed readily not only provided information on the resin, but was also able to locate photographs showing the epoxy resin drums on site during preparation of the *dalle de verre* panels (Figure 6). These photographs demonstrated that the brand name was Kollercast, not Epikote, and David Kirby produced documentation that it was ordered from James Beadel & Co Ltd.

Figure 6. Drum of Kollercast 332 epoxy resin, photographed on site during the lantern's construction. Courtesy of David Kirby

Despite this helpful information, attempts to specify the Kollercast 332 resin and the particular hardener used with it were to no avail. Synthetic Resins Ltd., a Unilever subsidiary at the address in Edwards Lane, Speke, Liverpool, formerly occupied by James Beadel & Co Ltd., was taken over in the 1980s by Scott Bader and essential parts of the business transferred to their Northamptonshire operation, but this trail led no further. In the past, Beck Koller & Co (England) Ltd. had a Kollercast Division at 103 High Street, Thane, Oxon, but an attempt to track down the identity of Kollercast 332 through their current European headquarters was not pursued in view of the helpful spectroscopic information from samples of the lantern epoxy which were obtained at this stage in the quest for historical documentary evidence.

During the laboratory examination of lantern samples at Delft University of Technology (de Bie 2019), supportive information was gathered on each of the mortar constituents by means of Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy, scanning electron microscopy (SEM), and thermogravimetric analysis (TGA), as described in the following sections.

EPOXY RESIN CHARACTERISATION BY FTIR AND RAMAN SPECTROSCOPY

Prior to this project, previous attempts to characterise epoxy resins by means of FTIR spectroscopy (Tennent, de Groot, and Koob 2019) had demonstrated that this common method of polymer identification – see, for example, Picollo et al. (2014) – was not able to give a fully definitive characterisation of amine-cured epoxy resins. Nonetheless, the ubiquity of FTIR instrumentation as opposed to more comprehensive methods such as gas chromatography-mass spectrometry (GC-MS) and nuclear magnetic resonance (NMR) spectroscopy led us to FTIR as the first option. The initial FTIR results showed that, with the instrumentation available, the epoxy spectrum was dominated by the very broad, strong band centred at 1050 cm^{-1}, arising from the quartz particles in the mortar, which obscured the most diagnostic epoxy bands in the range of 950-1150 cm^{-1}. While the current lateral spatial resolution of both FTIR and Raman microscopes would be sufficient to overcome this problem by focusing solely on the epoxy between the quartz grains, we opted for Raman spectroscopy by means of a Renishaw inVia™ confocal Raman microscope with Renishaw WiRE™ software.

Raman has the added advantage that, in contrast to FTIR, the spectrum of quartz has no bands which obscure the most important diagnostic bands of epoxy resins. As illustrated in the upper spectrum shown in Figure 7, the major Raman-active band for quartz is a sharp feature at 466 cm^{-1} which does not interfere problematically with the interpretation of epoxy resin spectra – in this case, the replica sample prepared with Araldite 2020. The lantern epoxy spectrum (Figure 7 below, light blue) is in this case from a spot with no quartz and, thus, does not include the quartz signature band at 466 cm^{-1}. The similarity of the two spectra in Figure 7 indicates that they are both amine-cured epoxies derived from the diglycidyl ether of Bisphenol A, the characteristic FTIR vibrations of which are also Raman-active and have been discussed previously (Tennent, de Groot, and Koob 2019). Though there are small discrepancies between the two spectra (Figure 7), indicating that the epoxy resin match is not exact, the compositions were considered sufficiently close for preparation of mortar replica samples using Araldite 2020.

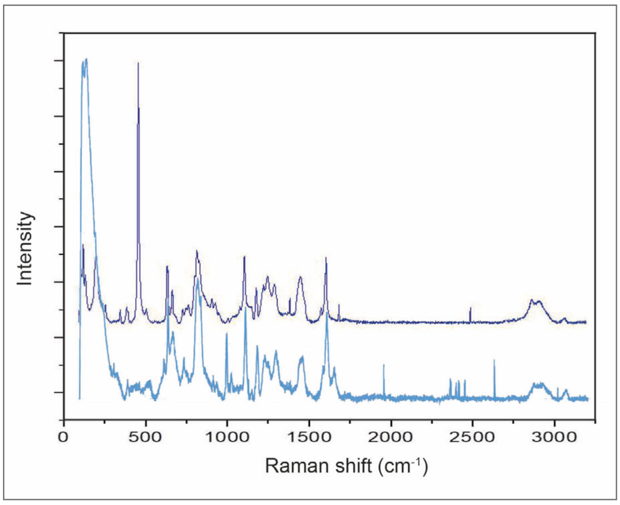

Figure 7. Raman spectra of the lantern mortar epoxy resin (below, light blue) and Araldite 2020 epoxy resin used in the replicated mortar sample (above, dark blue)

TGA OF THE LANTERN MORTAR

Mortar core samples removed from the *dalle de verre* edifice were analysed by TGA to determine the proportion of carbon black and sand in the mix. Experiments were performed using a Perkin Elmer TGA instrument at a constant heating rate of 20 °C per minute from 20-900 °C. From 20-600 °C, the test was performed under a nitrogen atmosphere; above 600 °C, the purge gas was switched to air. Additionally, three replicate mortar sample mixes were prepared containing 0%, 0.05%, and 3% carbon black, each with a 1:2 ratio of Araldite 2020 epoxy to fine-grain quartz flour.

The TGA results (Figure 8) comprise the plots of the two lantern samples from core 9, retrieved from a section designated Panel C on the east side of the cathedral, and the three replicate mixes. The TGA trace profiles are interpreted as follows: decomposition of the epoxy takes place below 600 °C under the inert nitrogen atmosphere. Above 600 °C, the three remaining components are the incombustible sand, the carbonised epoxy, and the carbon black. The decomposition step at around

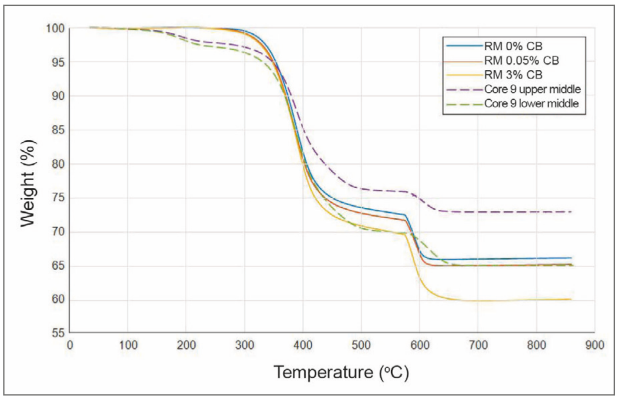

Figure 8. TGA plots of two samples of mortar from core 9 removed from the lantern and three replicate mortar samples (RM) with different amounts of carbon black

600 °C is ascribed to the combustion of both the carbon black present in the mortar and the residue of the carbonised epoxy. The residual material remaining above approximately 650 °C is the sand.

The lantern samples show a first weight loss step around 200 °C. As the replica samples do not show this step, we propose that this indicates the presence of volatile products in the material, very possibly including bound water from half a century of exposure to the elements. The onset of the second step in all samples is around 300 °C. This step is linked to the decomposition by pyrolysis of the epoxy present in the samples. The third and final step involves combustion of all the organic material. For the two samples from core 9, this results in a weight loss of approximately 3% and 5%, reflecting the different proportions of epoxy resin therein. The difference in epoxy to sand ratio from the different locations in the same mortar core is also indicated by the amount of sand ultimately remaining in these samples, namely 73% and 65%, respectively.

Each of the three replicate samples also shows a distinct step at around 600 °C, regardless of the carbon black content. In this step, the weight losses of the 0% and 0.05% carbon black samples are approximately equal at 7%, while the 3% carbon black sample shows a distinct increase in weight loss to 10%. This step represents the combustion of both the carbon black and the carbonised epoxy residue after pyrolysis. Taking into account the contribution of the epoxy combustion in this step, the results are consistent with the proportion of carbon black, with the differential in the step height between the 0% and 0.05% carbon black samples being marginal. The TGA experiments thus proved of value for two reasons: as confirmation of the information provided by David Kirby on the 1:2 epoxy : sand ratio used for the lantern mortar, and as a reliable guide to the appropriate amount of carbon black required for preparation of the accelerated ageing sample mixes. In support of the TGA findings, a mix prepared with 0.05% carbon black corresponded to the colour of the original mortar.

SPECIFICATION OF THE SAND IN THE LANTERN MORTAR MIX

David Kirby was also able to provide information that, as a filler for the mortar, grade M3 quartz flour, a designation which corresponded to a very fine grain size of less than 125 μm, had been supplied by British Industrial Sand (Scotland) Ltd. SEM of a sample of the lantern mortar (Figure 9) confirmed that the general particle size range was of this order. For the mortar replication, a small quantity of a commercial quartz flour with a similar particle size range, 'Sand, white quartz > 230 mesh', was sourced from Sigma Aldrich. For the final mortar recipe, this grade was mixed into the epoxy resin in a ratio of 1:2 epoxy : sand, together with carbon black at 0.05 percent by weight. SEM images of the replicated mortar attested to a similar quartz flour dispersion in the resin and a grain size distribution only marginally greater than the original.

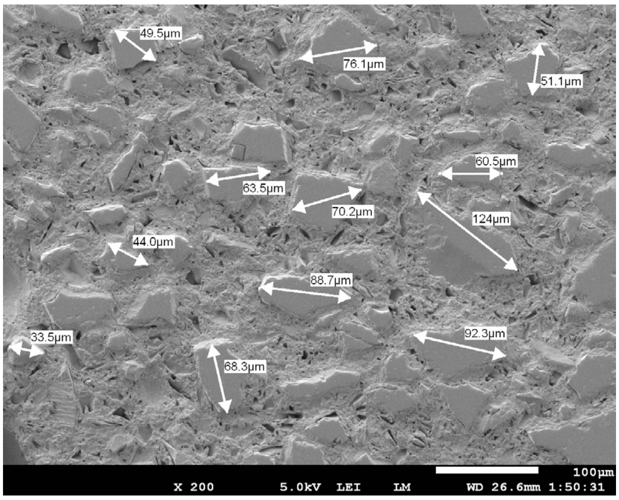

Figure 9. SEM micrograph at 200x magnification of the lantern mortar showing the quartz flour grains and a representative range of the grain dimensions

CONCLUSIONS

The research described in this paper successfully achieved its main goal; to enable the preparation of samples corresponding to the original lantern mortar composition in order that valid accelerated ageing tests could be undertaken with confidence. The lantern mortar components were sufficiently precisely defined by means of archival research, personal interviews, and laboratory analyses to permit a replica mortar to be prepared which mirrors closely the original formulation.

In accomplishing what was initially imagined would be a relatively straightforward task, several unanticipated difficulties were encountered. The principal issues complicating the quest for documentary evidence which would enable a precise mortar characterisation included:

- the span of intervening time, more than five decades, limiting the possibility of personal reminiscences;
- the absence of relevant technical records in the cathedral and other archives;
- the conflicting evidence from the *Crown of Glass* film and the on-site photographic record on the identity of the commercial epoxy resin used;
- the absence of online information on the commercial epoxy product, the name of which was depicted in the photograph of the resin drum; and
- the commercial firm closures and takeovers which made tracing suppliers' and manufacturers' product specifications prohibitively laborious.

Though specific to this project, these obstacles resonate with the difficulties encountered in other attempts to undertake retrospective investigations requiring the need for specificity in the materials used in the creation or conservation of artistic works of art in glass. As has been found in projects involving museum glass artefact conservation (Tennent, de Groot, and Koob 2019) and the creation and conservation of outdoor monumental appliqué stained glass (Tennent 2006), a span of time as short as a few decades is often sufficient to make personal recollections dubious, the retrieval of commercial product data extremely difficult, the acquisition of new batches of commercial products impossible, and the location of documentation that might be held by cultural and religious bodies an entirely fruitless task. The last of these issues is frequently exacerbated by the fact that detailed documentation of technical information on methods and materials – essential for an effective retrospective investigation – was not originally considered important enough to be properly recorded.

In this project, these difficulties were counterbalanced, fortuitously, by the good personal record-keeping of the project manager who had overseen the lantern construction. Thanks to the serendipitous discovery of documentation for the materials used, combined with laboratory analyses, a satisfactory outcome for the replication of the original mortar formulation – the fundamental prerequisite for assessment of its longevity by means of subsequent accelerated ageing tests – was achieved.

Encouragingly, the results of the various tests to quantify the in-service changes in the epoxy resin mortar give confidence for the future integrity of the lantern structure (de Bie 2019); minimal changes have been observed in the intrinsic strength-related properties of the resin system during the lifetime of the cathedral. Combined with this, the good replication of the cathedral mortar formulation described above enabled accelerated ageing tests to be performed (Poulis et al. forthcoming) which give no reason to believe that there should be any acceleration of the degradation process beyond the slow changes that have occurred to date.

ACKNOWLEDGEMENTS

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