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Moisture and fungal degradation in fibrous plaster

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28 Fibrous plaster degradation has been a key concern over recent years, with ceiling failures
29 occurring suddenly in historic buildings, including the Apollo theatre in 2013. This rigorous
30 investigation explores fibrous plaster degradation through subjecting 290 specimens to a range
31 of moisture and fungal-related treatment conditions over periods of up to two years and
32 analysis using mechanical flexural tests, Fourier Transform Infrared Spectroscopy (FTIR),
33 Scanning Electron Microscopy (SEM) and Deoxyribonucleic Acid (DNA) sequencing. Using
34 FTIR peak ratios from spectra of hessian fibres and mechanical tests in conjunction, an original
35 methodology for identifying mechanisms and severity of fibrous plaster degradation through
36 moisture and fungal exposure was developed. Results showed defined clusters for differing
37 moisture and fungal treatments when two peak ratios are plotted together and compared with
38 mechanical data. Fungal exposure over two years, water submersion and wetting and drying
39 were particularly detrimental conditions for fibrous plaster. Fungal exposure resulted in
40 degradation of cellulose bonds in hessian fibres, with defined clusters on the extreme left of
41 peak ratio plots correlating with a pronounced reduction in fibrous plaster mean flexural
42 strength of 51%. Fungal species *Penicillium* and *Chaetomium* were identified on test samples.
43 Moisture affected plaster matrices significantly with wetting/drying and water submersion
44 treatments resulting in a 71% reduction in mean flexural strength for unreinforced plaster,
45 reducing to 26% with hessian-reinforced fibrous plaster. Many buildings containing fibrous
46 plaster are listed and removal of material is often minimised - the high impact of this research
47 stems from the ability to rapidly assess the mechanical integrity of a very small quantity of
48 harvested historic hessian fibres using FTIR. Identifying the location of weakened fibres in a
49 ceiling is highly important for effective restoration and conservation.

50

51

52 **Keywords**— Fibrous plaster, Hessian Fibres, Degradation, Fungi, Moisture, Fourier Transform
53 Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), Deoxyribonucleic Acid
54 (DNA).

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Glossary

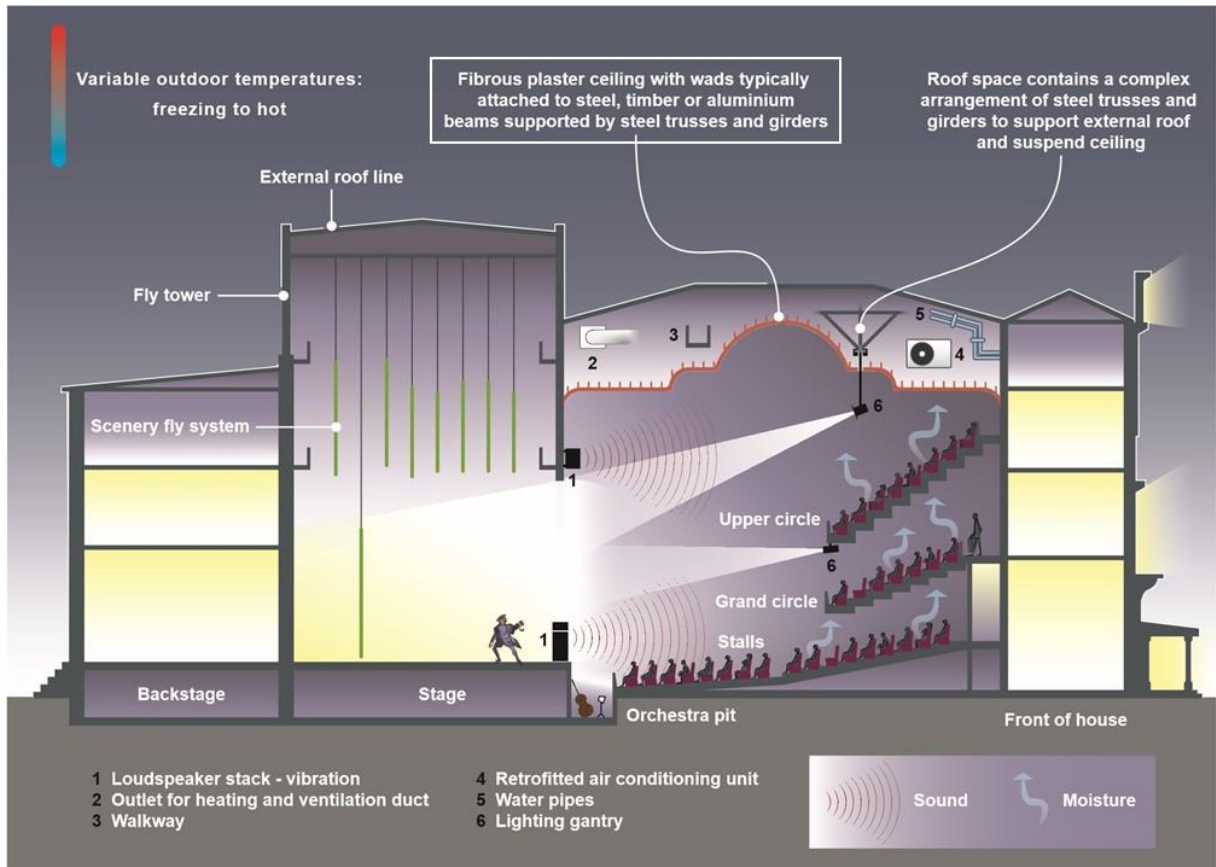
| | |
|-------|---|
| ANOVA | Analysis of Variance |
| BLAST | Basic Local Alignment Search Tool |
| DNA | Deoxyribonucleic Acid |
| FASTA | Used in DNA testing, a text based format representing sequences |
| FE | Fracture Energy |
| FTIR | Fourier Transform Infrared Spectroscopy |
| ITS | Internal Transcribed Spacer |
| LOP | Limit Of Plasticity |
| MOE | Modulus of Elasticity |
| MS | Maximum Stress |
| PDA | Potato Dextrose Agar |
| SEM | Scanning Electron Microscopy |

58

59

60 1 Introduction

61 Fibrous plaster has been used as a material in buildings for well over a century and is perhaps
62 best known for providing decorative interiors and ornate ceilings in buildings such as theatres,
63 hotels, civic buildings and private residences [1]. In a theatre setting, fibrous plaster ceilings
64 typically are found above the auditorium and form part of a large and complex building structure
65 as shown in Figure 1. Human occupation and activity can vary considerably within venues and
66 contribute to the fibrous plaster ceiling experiencing differences in temperature, relative
67 humidity, sound vibration and presence of micro-organisms in the air both below the fibrous
68 plaster ceiling and above in the roof-space, where fibrous plaster ceilings are also subject to
69 external weather conditions through the envelope of the roof structure.
70



71
72 *Figure 1 - Cross sectional diagram of a typical theatre layout, with the fibrous plaster ceiling hung above the auditorium and*
73 *subjected to varying environmental conditions resulting from human occupancy and external weather (Image courtesy of*
74 *Historic England)*

75
76 Failures have occurred in historic fibrous plaster applications during the 21st century which has
77 drawn more attention to fibrous plaster ceilings in particular, with the most notable and widely
78 publicised failure occurring during a performance at the Apollo theatre, London, in 2013 [2]
79 where 58 people were hospitalised through injury attained through fallen fibrous plaster debris
80 from a partial ceiling collapse [3]. The Savoy Hotel, London, also had a similar, but smaller-
81 scale, event in 2019 when a collapse happened during a charity auction event [4] and the
82 Piccadilly theatre also experienced a partial collapse in 2019 [5].
83

84 Failure incidents such as the above accentuate the importance of identifying and
85 understanding the mechanisms of degradation in fibrous plaster ceilings with a view to
86 assisting and informing the practice carrying out appropriate maintenance and repair work on

87 an ongoing basis; in the wake of the Apollo Theatre collapse, it is required for places of
88 entertainment to be inspected by industry specialist plaster companies and structural
89 engineers for deterioration on a regular basis [6]. Is it important to conduct research into
90 identifying the extent to which potential failure mechanisms resulting from differing
91 environmental conditions can affect and influence the degradation of fibrous plaster over time.
92

93 Decorative plaster was originally made from a combination of lime mortar and animal hair.
94 Gypsum plaster (also known as 'Plaster of Paris') subsequently became a popular alternative
95 to lime, which was ultimately quicker to set, lighter, and facilitated an increase in production
96 speed [2]. Hessian fibre scrim, typically comprising of bundled bast fibres from the jute plant
97 [1] became the most common reinforcement material within the gypsum plaster matrix.
98 Leonard Alexander Desachy patented the gypsum plaster and hessian fibre combination as
99 fibrous plaster in 1856 [7]. Gypsum plaster consists of three phases - calcium sulfate dihydrate
100 ($CaSO_4 \cdot 2H_2O$), calcium sulfate hemihydrate ($CaSO_4 \cdot 0.5H_2O$), and calcium sulfate anhydrite
101 ($CaSO_4$), with the proportions of each determining the gypsum plaster properties [8]. Traditional
102 gypsum plaster (known as 'beta' plaster) possesses an uneven crystalline structure [9], [8].
103 Gypsum plaster is a brittle material but the addition of fibrous reinforcement improves ductility
104 and durability [10].
105

106 To date, there has not been a large quantity of research conducted explicitly on fibrous plaster
107 and only a select group of specialist practitioners across the United Kingdom possess the
108 expertise to maintain fibrous plaster ceilings. Guidance written by Stewart et al., 2019 provides
109 a history of fibrous plaster, details forms of degradation and gives advice concerning methods
110 of care and repair [1]. Ireland, 2020 published guidance on the assessment and repair of
111 fibrous plaster ceilings [11]. Ngah et al., 2020 conducted research on the strength of gypsum
112 plaster, hessian fibres and quadaxial and continuous fibre mat glass fibre reinforcement as
113 potential modern substitutes for hessian fibres [8]. The Institute of Structural Engineers
114 published two articles providing a comprehensive overview of the potential causes of failure of
115 historic fibrous plaster ceilings, including a methodology for carrying out *in-situ* assessments
116 of condition [5], [12].
117

118 Moisture (including water ingress and variable humidity) and fungal growth have been
119 identified as important fibrous plaster degradation mechanisms, whether via biodegradation of
120 the hessian fibre scrim or compromising the integrity of the gypsum plaster matrix. Hessian
121 scrim is a natural fibrous material which does not bind strongly with gypsum plaster [8]; this
122 may promote degradation (and ultimately failure) by allowing fungal growth and/or moisture
123 ingress within cracks and voids. Moisture and fungi may even be introduced to hessian fibres
124 at the early stage of retting (fibre separation from plant stem) which uses several different
125 methods (plus possible treatment with caustic soda) involving moisture and microbes [13], [14].
126

127 Moisture may degrade gypsum by two possible methods; either gypsum will dissolve over time,
128 weakening the material and ultimately leading to failure, or moisture acts as a lubricant by
129 allowing gypsum particles to slide over each other. Gypsum crystals are randomly orientated
130 and are a mosaic of different textures [15] and dimensionally varied, therefore the dissolution
131 surface is uneven and unpredictable [16]. Gypsum dissolution by moisture over time could be
132 a factor which influences the rate of degradation [16] allowing not only moisture to affect
133 gypsum strength but also provide a shorter route for moisture reaching internal fibres (though
134 the research was not explicitly concerning fibrous plaster). With fibres situated inside a porous
135 gypsum matrix, moisture is transferred more easily to fibres through the interface [17]. Moisture
136 degrading flax fibres inside a resin epoxy matrix was noted by Assarar et al., 2011 who
137 concluded that matrix-interface weakening was the main cause of failure [17].
138

139 Moisture is known to affect natural bast fibres due to the high absorption ability of cellulose
140 [13]; hydroxyl groups within fibres attracting water molecules through the formation of

141 hydrogen bonds. Cellulose and lignin ratios of the reinforcement fibre determine the level of
142 water absorption; jute has a relatively high lignin content (11% - 26%) and lower cellulose (45%
143 - 71%) [18] [19] which promotes higher moisture absorption, whereas cotton possesses higher
144 cellulose levels (88% - 96%) [20] and lower lignin content (up to 1%) [21] and absorbs less
145 moisture. Since research explicitly on fibrous plaster is limited, bast fibres are relevant for
146 general comparison as jute and hemp are both types of bast fibres. Research conducted on
147 Flax fibres showed natural fibres are hydrophilic (attracted to water molecules) and composite
148 materials containing them lose strength when subjected to humid conditions [17]. Moisture can
149 infiltrate via several possible mechanisms; diffusion, imperfections or by capillarity in the fibres.
150 Fickian behaviour is likely and moisture equilibrium is reached rapidly in humid conditions and
151 is maintained whilst the humidity remains constant. A flax fibre composite material showed a
152 13.5% increase in weight when immersed in water whereas there was a 1.05% increase for
153 the glass fibre composite [17]. Bast fibres have high moisture absorption and poor dimensional
154 stability [22] and the swelling of fibres can cause microcracking in surrounding material which
155 in turn leads to degradation. This could be the case for the hessian in fibrous plaster, where
156 swelling due to moisture absorption cracks the plaster, as well as promoting fungal degradation
157 due to the high-humidity environment [22].
158

159 Indoor environments need to be carefully controlled and key factors which affect microbial
160 growth are lighting, heating, humidity and ventilation [23] – a challenging task in a venue such
161 as a theatre, which may alternate periods of dense occupation with periods of low or non-
162 occupation. Plaster is known to be affected by fungi within the built environment [23]; it is
163 composed of minerals (gypsum) and is susceptible to biodeterioration. Particulates in the
164 atmosphere are the food source for fungi and bacteria to grow within cracks or pores. Fungi
165 entering the plaster are classed as physical weathering due to filaments growing further into
166 the material. Plaster is classed as a mineral based material so biofilms from the fungi could
167 develop, and humidity could cause mineral dissolution.
168

169 Experiments concerning how different environmental conditions affect types of plaster give a
170 good perspective on biodegradation as a result of moisture or fungal-related mechanisms [24].
171 Fungal growth on gypsum plaster and hessian fibres is facilitated by the porous nature of the
172 materials, with hyphae penetrating the surface of gypsum on a microscopic level [25].
173

174 Hessian fibres are organic bast fibres and therefore susceptible to biodegradation by
175 extracellular enzymes [23]. Hessian (jute) fibres were not degraded by fungi when tested as
176 part of a polylactic acid (PLA) composite material, but the PLA was, leading to a gap between
177 the interface and resulting in a loss of strength [26]. Jute fibres by themselves have been
178 heavily degraded by fungi and are susceptible to *Macrophomina phaseolina* pathogens during
179 cultivation [27]. Cladosporium is one of the most densely populated fungi found in both interior
180 and exterior environments [28]. Cellulose is the principal component of bast fibres and provides
181 the basis of strength and stiffness [29]. It has a crystalline structure formed of linear polymer
182 units which in turn form microfibrils held together by hydrogen bonds, forming cellulose fibres
183 which provide tensile strength. However, there is also a varying extent of amorphinity in
184 structure, with enough heterogeneity in topology to allow susceptibility to cellulase - enzymes
185 which can be produced by fungi which decompose cellulose molecules with the mechanism of
186 hydrolysis [30]. Fungi harbour enzymes which break down cellulose into simpler forms (mostly
187 glucose) [31]. Both enzymes and water can be used for the fibre retting process and may affect
188 the natural material. Although retting is a deliberate process that is necessary for fibre
189 extraction, the negative effects of water and fungi in an uncontrolled environment (such as in
190 a ceiling) may be informed by analysing the controlled retting process. It was noted by [22] that
191 moisture combined with fungal growth caused fibres to degrade and lose strength [31]. There
192 are two mechanisms for fungal attack on hessian fibres; either the fungal spores existed on
193 the hessian before it was incorporated into fibrous plaster, or the fungus infiltrated the fibrous
194 plaster during its working life. In the first instance, plaster may appear in good working condition

195 but fail once the hessian on the inside has degraded. The second mechanism is also possible
196 and may occur alongside the first, causing different species of microbes to enter. One further
197 possibility is purely the plaster cracking and the degradation mechanism being bio-weathering.
198 Fungal spores or moisture already being present in the hessian would cause fibres to degrade.
199

200 This study focuses on experimentation and analysis of the degradation of fibrous plaster
201 caused by moisture and fungal growth with the aim of understanding these degradation
202 mechanisms and identifying conditions particularly detrimental to fibrous plaster integrity.
203 Experiments encompassed mechanical flexural testing of plaster specimens with and without
204 hessian fibres, Fourier Transform Infrared Spectroscopy (FTIR) conducted on hessian fibre
205 samples, Scanning Electron Microscopy (SEM) carried out for identification of fungal growth
206 and Deoxyribonucleic Acid (DNA) sequencing of fungal specimens to identify presence upon
207 historic fibrous plaster samples and using this to further inform FTIR analysis of the types of
208 fungi identified.
209

210 Improving understanding of degradation mechanisms and anticipating potential failure of
211 fibrous plaster is important for public health and safety, the economic and business operation
212 of the historic buildings in which fibrous plaster is present, and for the wider development of
213 understanding how historic and current construction materials behave when considering in-
214 situ assessment and repair.
215
216

217 2 Methodology

218 The four-stage investigation strategy of this study consisted of flexural tests of flat plate fibrous
219 plaster specimens, FTIR, DNA and SEM, which together formed a rigorous evaluation of the
220 effects of moisture and fungal growth on the integrity of fibrous plaster. Flexural tests consisted
221 of multiple fibrous plaster plate specimens, subjected to a variety of moisture and fungal-based
222 treatments, tested to failure along with statistical analysis of result variation. Full details of the
223 range of test treatments upon test specimens are presented in section 2.1 and the flexural test
224 method is elaborated upon in section 2.2. FTIR experimentation on hessian fibres subjected
225 to the range of moisture and fungal treatments used a peak ratio method to determine the
226 difference between degradation mechanisms and aid identification of differing moisture and
227 fungal effects; full details of the FTIR method are presented in section 2.5. DNA tests were
228 used to identify types of fungi growing upon exposed samples of hessian fibres, with the
229 methodology outlined in section 2.4 and SEM was used to observe the microstructure of fungal
230 growth upon test specimens as detailed in section 2.3.

232 2.1 Test sample matrices

233 Fibrous plaster samples were created and subjected to a variety of environmental treatments,
234 which have been separated into two core categories – moisture treatment and fungal treatment
235 - to assess degradation from both treatments.

236 2.1.1 Moisture treatments

237 Table 1 shows the three sub-categories of fibres - no fibrous reinforcement present (N),
238 hessian fibres used (H) and with glass fibres used (G), which were subjected to four moisture-
239 related treatments along with a control category with no moisture treatment process applied.
240 The abbreviations assigned to the combinations of fibre type and moisture treatment are
241 shown and the samples are referred to using these abbreviations hereafter.

242 *Table 1 - Sample matrices for FIBROUS PLASTER samples, both with and without fibres, subjected to moisture-based*
243 *treatments*

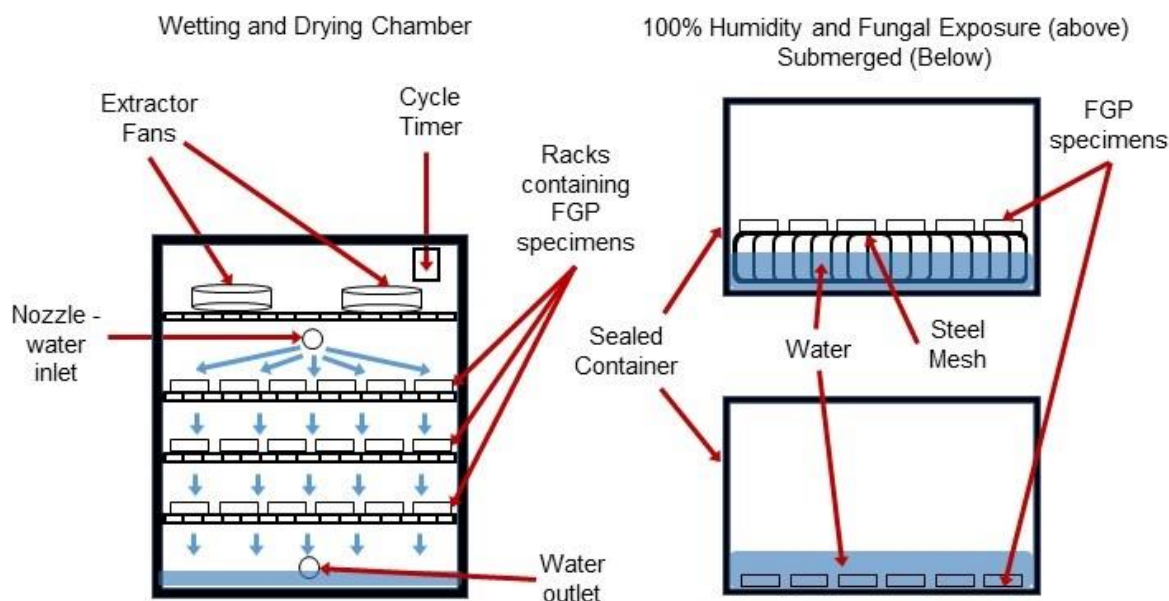
| Fibrous reinforcement | No treatment (N) | 100% RH (H) | Submerged in water (W) | Wetting and drying (D) | Freeze thaw (T) |
|--|-------------------------|--------------------|-------------------------------|-------------------------------|------------------------|
| None (N) | NN | NH | NW | ND | NT |
| Two Layers of Hessian (H) fibres (1 mm from sample base) | HN | HH | HW | HD | HT |
| Two Layers of Glass (G) fibres (1 mm from sample base) | GN | GH | GW | GD | GT |

244

245 For the 100% humidity tests, the sample specimens were stored for three months in a closed
246 plastic container. 4000 ml of water was inside the container, creating the humid environment
247 with a stainless-steel mesh holding the samples at a level of 100 mm above the surface of the

248 water. Submerged in water tests involved the samples being submerged in a closed container
249 with water for seven days [32]. Both methods are illustrated in Figure 2.

250
251 During the wetting and drying tests, samples were placed in a chamber which contained
252 shelving, an extractor fan, timer, and water nozzle. The samples were sprayed continuously
253 for 18 minutes, followed by a fan-drying phase lasting the remaining 23 hours and 42 minutes
254 of each day (Figure 2). This process was repeated 30 times overall [32].
255



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257
258 *Figure 2 - Diagrams illustrating the methodologies for the wetting and drying treatment (left) and the 100% Relative*
259 *Humidity, Fungal exposure and submerged methods (right)*

260
261 Samples subjected to the freeze-thaw conditions were first placed in tap water until a constant
262 weight was reached, indicating the pores were full of water, then the excess water was wiped
263 from the outside with a dry cloth. Following this, they were transferred to a freezer for 36 hours
264 before being dried on cotton fabric covers on top of a heated surface at 50°C until the weight
265 remained constant. This final sample weight was lower than the original weight due to some
266 of the gypsum dissolving during the process and this freeze thaw condition cycle was
267 completed once for each sample [32].
268

269 2.1.2 Fungal treatments

270 Table 2 shows the three sub-categories of specimens, both with hessian fibres (in two different
271 configurations) and without, which were subjected to three fungal-related treatments and a
272 control category with no treatment. The table shows the allocated sample abbreviations which
273 are used in the results sections.

274 For the treatment category of subjecting new fibrous plaster samples to fungi without a food
275 source, historic fibrous plaster samples obtained from the Hammersmith Apollo and KOKO
276 Theatre, (supplied by specialist plaster companies Hayles and Howe, Bristol, and Locker and
277 Riley, South Woodham Ferrers) were used for creating conditions in which fungal spores were
278 present. These historic samples were placed on a stainless-steel mesh with the new flexural
279 samples manufactured for testing within a sealed container as used for the 100% humidity

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tests, with 4000 ml water at the bottom of the container (Figure 2). The samples were then kept in these conditions for three months [32].

Table 2 - Sample matrices for FIBROUS PLASTER samples, both with and without fibres, subjected to fungal-based treatments

| Fibrous reinforcement | No treatment | Exposed to Fungal spores | | |
|---|------------------------------|------------------------------|----------------------------|----------------------------|
| | No food source (N) | 3 months | | 24 months |
| | | No food source (F) | food Source (Ff) | food source (F2) |
| None (N) | NN | NF | NFf | NF2 |
| Two Layers of Hessian fibres 1mm Away from sample base (A) | AN | AF | AFf | AF2 |
| Two Layers of Hessian fibres next to sample Base (B) | BN | BF | BFf | BF2 |

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Condition exposure for the samples exposed to fungus with a food source involved a similar method to those exposed without a food source, but with the addition of malt extract with 1 gram added per 100 ml water in the sealed container for three months [32]. The two-year samples (NF2, AF2 and BF2) were then kept under these conditions within a sealed container for a period of two years.

291

2.2 Three point flexural tests

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Fibrous plaster specimens for three-point flexural tests were manufactured using Siniat Prestia Classic Beta plaster. Hessian (jute fibre) scrim reinforcement with a variable mesh size of 5 mm x 5-10 mm (typically 7 mm) and a weight of 102 g/m² [33] was set inside the gypsum plaster matrix in two layers (distance from base surface as detailed in Table 1 and Table 2). For comparison, specimens were also made using continuous glass fibre mats with a weight of 210 g/m². The plaster matrix was cast in moulds with dimensions of 100 mm x 40 mm x 5 mm. Flexural strength tests on the samples involved a three-point bend test with a 50 kN load cell in an Instron 3366 Universal testing machine applying a central point load at a displacement rate of 0.5 mm/min (as shown in Figure 3, along with images of failure both with and without fibrous reinforcement). Displacements were recorded every 0.1 seconds until a 1.5 mm displacement was reached, with the rate then increasing to 5 mm/min until a total displacement of 10 mm had been applied. Under each condition shown in the sample test matrices in Table 1 and Table 2, there were twelve flexural samples manufactured and tested to identify variation in the results by statistical analysis.

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Four parameters were determined for comparison and evaluation – the maximum stress (MS), flexural Modulus of Elasticity (MOE), Limit of Plasticity (LOP) and Fracture Energy (FE). The parameters were calculated from each of the twelve samples from each combination shown in Table 1 and Table 2, following which a mean value was taken for that data set of twelve samples.

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From the load (kN) and displacement (mm) data, stress and strain were calculated. Maximum stress σ was obtained using equation (1) from International standards concerning flexural strength [34]:

$$\sigma = \frac{3PL}{2bd^2} \quad (1)$$

316 where P is the maximum load (kN), L is the length of the span from centre to centre of the
 317 support rollers (mm), b is the breadth of the test sample (mm) measured using digital callipers
 318 and d is the depth of the test sample (mm) measured using digital callipers. Flexural strain ε
 319 was calculated using equation (2):

$$\varepsilon = \frac{6Dd}{L^2} \quad (2)$$

320 where D is the deflection of the sample (mm), d is the depth of the sample (mm) and L is the
 321 length from centre to centre of the support rollers (mm). Limit of Plasticity (LOP) is taken as the
 322 point at which a material ceases to behave in the linear elastic range. Typically, in samples
 323 which have no fibrous reinforcement, this is the same as the maximum stress. Flexural
 324 Modulus of Elasticity MOE was calculated using equation (3):
 325

$$MOE = \frac{SL^3}{4000bd^3} \quad (3)$$

326 where S is the slope (or gradient) of the initial linear portion of the initial force (N) verses
 327 displacement (mm) curve and L , b and d are length, breadth, and depth as for equation (1)
 328 (mm). Fracture Energy FE (kJm^2) can be defined as the energy required to change a unit
 329 area of a fracture surface from its initial unloaded state to a state of complete separation and
 330 was calculated using equation (4) based upon the work of Petersson [35], [36] and Khalilpour
 331 et al. [37]:
 332

$$FE = \frac{E + Mg\delta_0}{b(d - a)} \quad (4)$$

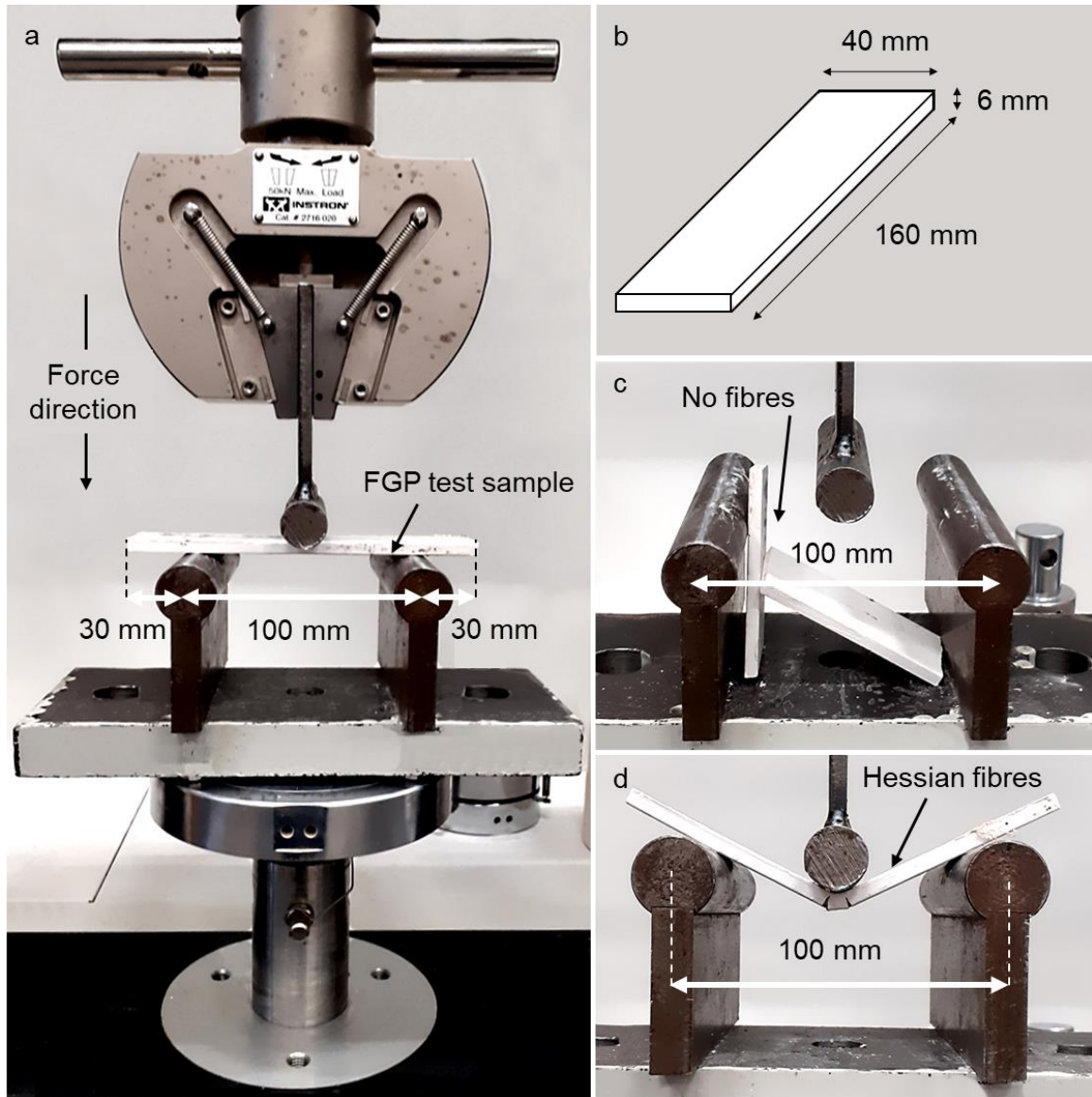
333 where E is the Fracture Energy (Nmm) calculated as the area under the stable load (N) versus
 334 deflection (mm) curve (not stress verses strain) until the point of deflection at maximum load;
 335 this was calculated using the trapezium method and approximated the areas between two sets
 336 of data points and summing to obtain the total area, b and d (mm) are breadth and depth
 337 respectively; a represents the notch cut (mm) if one was present (as there was no notch cut
 338 into these small fibrous plaster test samples, the value of a was zero) and $Mg\delta_0$ is a correction
 339 factor to allow for the mass of the beam; this would be required when dealing with a beam of
 340 notable mass such as concrete due to the length of the beam protruding over the end support
 341 of the rollers, being less than $\frac{1}{4}$ of the length of the beam [37]. M is the mass of the beam (kg),
 342 g is gravity taken as 9.81 ms^{-2} and δ_0 is the deflection of the test sample at maximum applied
 343 load (mm). However, in this study the mass of the fibrous plaster flexural samples led to this
 344 correction factor to be considered negligible, and therefore not applied.
 345

346 To provide statistical analysis of the flexural test results for samples subjected to moisture and
 347 fungal treatments, two statistical methods were chosen: Analysis of Variance (ANOVA) single
 348 factor and student t-test distributions (two-sample assuming unequal variances). These tests
 349 take the mean and variance into account, giving a more reliable result than using one statistical
 350 value. The null hypothesis was 'enough evidence to suggest the values are similar'. A value
 351 of 0.05 was used for the significance level alpha α for both statistical tests, meaning the null
 352 hypothesis was accepted if the comparison value exceeded 0.05.
 353

354 The ANOVA method has previously been used for analysing flax fibres and assessing how
 355 variations in chemical treatments impacted upon mechanical properties [38]. In this study,
 356 entire sample sets were initially tested using the ANOVA method, with sample size reducing

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(down to a minimum of three) to determine how many samples within a particular treatment set led to the null hypothesis being accepted. The t-test method was then applied to compare the similarity between two samples within a treatment category and assess whether the null hypothesis was accepted for the two samples.



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Figure 3 – Flexural test specimens and test set-up, a) a specimen loaded in the three-point test rig, b) Dimensions of the flexural test specimens, c) A typical failure of a specimen without fibrous reinforcement and d) a typical sample with hessian fibre reinforcement.

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2.3 Scanning Electron Microscopy (SEM)

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To obtain microscopic observation of the growth of the fungus on sample specimens, SEM imaging was undertaken on samples subjected to three different conditions:

- 100% relative humidity (HH)
- samples with historic hessian exposed to fungi with a food source (BFf)
- samples with historic hessian exposed to fungi without a food source (BF)

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All samples were placed on a 50 mm diameter aluminium mount and kept under vacuum for 48 hours prior to being covered in a 10 nm gold sputter coating in order to reduce charging before insertion into the microscope chamber. The SEM imaging was carried out to identify the fungus type and the pattern of growth on the gypsum plaster and hessian fibre scrim material. Images were captured with a JEOL SEM6480LV microscope at various magnifications.

382 2.4 DNA fungal identification

383 Historic fibrous plaster samples, with the historic hessian fibres exposed were used for the
384 identification of fungus growing within the reinforced fibrous plaster samples. The preparation
385 process involved the sample specimens being submerged for ten seconds in a 2.5%
386 hypochlorite solution, followed by placing for ten seconds in a 70% ethanol rinse and finally
387 being washed for forty seconds in distilled water.

388
389 Following this procedure, sample specimens were divided using forceps, whilst ensuring each
390 piece had internal hessian fibres exposed on at least one side and were each placed on a
391 section of a Potato Dextrose Agar (PDA) to promote fungal growth. Incubation time for the
392 samples was at least 3 days at a temperature of 25°C. Fungi which grew under the conditions
393 were then transferred to separate plates for analysis. Any fungi growing on test plates in areas
394 surrounding fibrous plaster sample specimens was not tested.

395
396 The most widely used DNA barcode region for fungus identification was amplified using Internal
397 Transcribed Spacer (ITS) 1 and ITS4 primers and this was sequenced using ITS1 for
398 undertaking Eurofins mix2seq sequencing. FASTA (a text-based format) sequences were then
399 trimmed and used with the Interactive Basic Local Alignment Search Tool (BLAST) on the
400 fungal ITS database for identification of the fungal species. The BLAST results were stored as
401 a text file which was then manually compared to the results on the database. Identification of
402 fungal species was based on the similarity to, or match with, DNA sequences already
403 contained in a sample database.

404
405 The most common fungi identified from the DNA tests were then grown in a petri-dish, with the
406 food source, and FTIR samples obtained for both mould and food source, the spectra of which
407 were compared to the hessian samples and similarity, or dissimilarity of spectra peaks
408 observed.

409 2.5 Fourier Transform Infrared Spectroscopy (FTIR)

410 Hessian fibres were extracted from a flexural test specimen for every different exposure
411 condition as described in Table 1 and Table 2. A Perkin Elmer Frontier FTIR instrument with a
412 diamond Attenuated Total Reflectance head was used for the scans. A Mercury-Cadmium-
413 Telluride (MCT) detector cooled by liquid nitrogen was used for the mid-infrared sensitivity and
414 provides a better response for the low levels of energy reaching the detector. The scan
415 resolution was 4 cm⁻¹ with a wave number range from 600 cm⁻¹ to 4000 cm⁻¹. 32 scans were
416 completed for each sample to obtain a high resolution, enabling significant peaks to be
417 identified. Extracted fibres were placed in a horizontal alignment on the crystal. Before each
418 scan of a different condition, a background scan was completed and the instrument was
419 cleaned with distilled water between every scan.

420

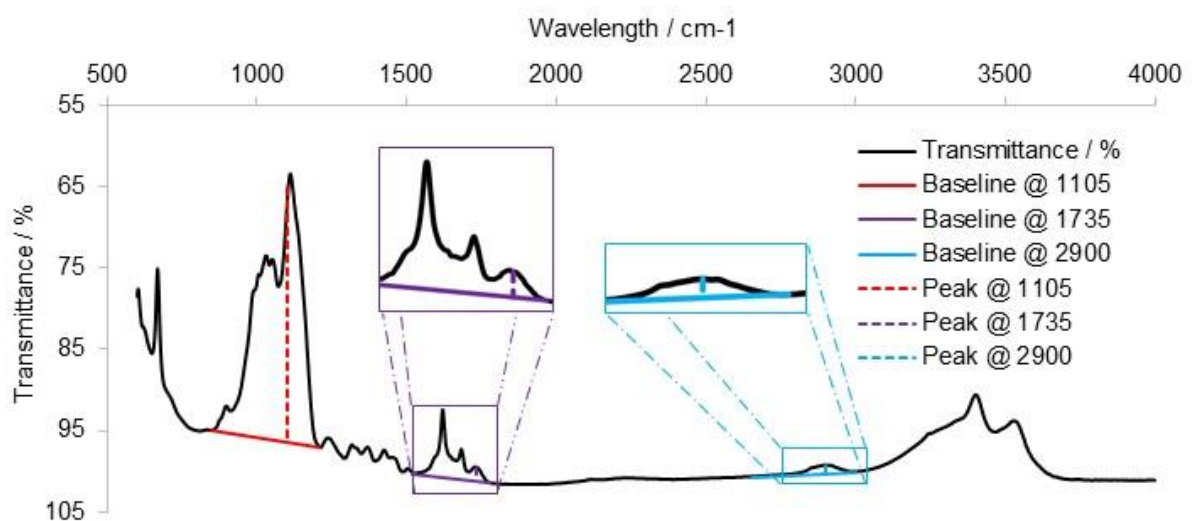
421 Based upon the FTIR peak ratio work of Garside and Wyeth [18], a method using FTIR peak
 422 ratios (R) (attained by the division of a transmittance peak value at a certain wavelength by
 423 the peak at another wavelength) was adapted for this study, with two peak ratios named R₁
 424 and R₂ plotted on an x and y axis to determine the identification of cellulosic fibres. It was
 425 aimed that multiple R₁ and R₂ results from different samples plotted on the same graph would
 426 indicate clusters or trends within the moisture and fungal treatment category sample sets with
 427 a view to differentiating the mechanisms of degradation. Peak ratios at the wavelengths shown
 428 in equations (5) and (6) were selected and used by this study:

$$R_1 = \frac{I_{1735}}{I_{1105}} \quad (5)$$

$$R_2 = \frac{I_{1735}}{I_{2900}} \quad (6)$$

429 Where I_{1105} is the wavelength peak (dotted line in Figure 2) at 1105 cm⁻¹ indicating the C-O-C
 430 glycosidic bond representing cellulose content and I_{2900} is the wavelength peak height at 2900
 431 cm⁻¹ denoting the C-H bond representing a measure of overall organic content [18]. The main
 432 constituents of fibrous plants are cellulose, hemi-cellulose, lignin and pectin. Pectin content
 433 determines the flexibility of fibrous plants; pectin is water soluble and degradable, which can
 434 cause fibres to lose overall strength [29]. The peak at wavelength 1735 cm⁻¹ representing
 435 pectin (C=O ester bond), was used in this study as the numerator I_{1735} in equations (5) and
 436 (6) rather than the peak at 1595 cm⁻¹ (C=C aromatic in-plane, representing lignin content) used
 437 in Garside and Wyeth, 2003 (which resulted in relative insensitivity as to whether samples
 438 were modern or aged). A wavelength of 1735 cm⁻¹ showing pectin is reported as being clearer
 439 in degraded materials from the carboxyl groups in oxycelluloses [18]. Therefore, in this study
 440 R₁ and R₂ represent pectin to cellulose and pectin to overall organic material ratios
 441 respectively.

442 For each chosen wavelength peak, a baseline was estimated, and the peak intensity was
 443 calculated using the transmittance at the peak top, as well as the transmittance at the
 444 wavelength along a linear baseline using $y = mx + c$. Figure 4 shows an example of an FTIR
 445 plot with peak transmittance for a sample set in the HN (top) treatment category showing the
 446 application of the linear baseline for the three peak wavelengths, calculated and applied to the
 447 peak ratios in equations (5) and (6).
 448



449 Figure 4 – Example FTIR plot showing peak transmittance and linear baselines applied for a fibre in treatment category HN.
 450

451 3 Results

452

453 3.1 Three point flexural tests

454 The three point flexural tests for both the moisture treated and fungal growth-treated samples
455 are shown in Figure 5 Part 1 (specimens with no fibres and glass fibres) and Part 2 (specimens
456 with hessian fibres) - note the differing vertical axis for glass fibre specimens and the different
457 horizontal axis for specimens with no fibres. Maximum Stress (MS, MPa), Limit of Plasticity
458 (LOP, MPa), Modulus of Elasticity (MOE, GPa) and Fracture Energy (FE, KJ/m²) are shown
459 for each moisture and fungal treatment category. Figure 6 shows a selection of the flexural
460 specimens subjected to the two-year fungal treatments after testing and reveals the extent to
461 which fungal growth can be observed on the exterior of the fibrous plaster matrix Particularly
462 AF2 specimens in part (c), and the variability from specimen to specimen, with black growth
463 being a clear visible sign of fungal attack [23].

464

465 Of particular note is the growth observed on AF2 specimens; sample specimens subjected to
466 fungi from historic samples (NF, AF and BF series) and samples subjected to 100% RH (NH,
467 HH, GH) also displayed fungal growth. Table 3 shows the t-test results for the flexural samples
468 and how two samples relate to one another within the treatment category, with green indicating
469 the acceptance of the null hypothesis and grey rejection of the null hypothesis. Running
470 ANOVA tests for each moisture and fungal treatment category in Table 1 and Table 2 results
471 in the null hypothesis largely being rejected for the full range of samples within the treatment
472 category. The most notable differences are noted in the mechanical property subsections
473 below.

474

475 3.1.1 Maximum Stress (MS)

476 For maximum stress (MS), the tests on fibrous samples were continued until a displacement
477 of 10 mm was reached. For the samples which do not possess fibrous reinforcement (NN,
478 NH, NW, ND, NT, NF, NFf, NF2) the limit of plasticity (LOP) equals MS. Unreinforced samples
479 were affected by the conditions in mechanical testing results. Samples which were submerged
480 in water (NW, 71.2% decrease compared to the no treatment samples with ANOVA analysis),
481 subjected to wetting and drying tests (ND, 43.3% decrease compared to no treatment) and
482 subjected to fungus for two years (NF2, 42.6% compared to no treatment) impacted the plaster
483 the most.

484

485 Hessian-reinforced plaster tests resulted in the samples exposed to historic fibres with fungi
486 and no food (BF) having a higher maximum stress than the no-treatment samples (NF) when
487 the reinforcement was located almost at the bottom of the sample. Otherwise, ANOVA analysis
488 reveals AF2 samples (51.4%), HW (25%) and HD (35%) were the most negatively impacted
489 with a decrease for each average maximum stress value compared to the no-treatment
490 samples.

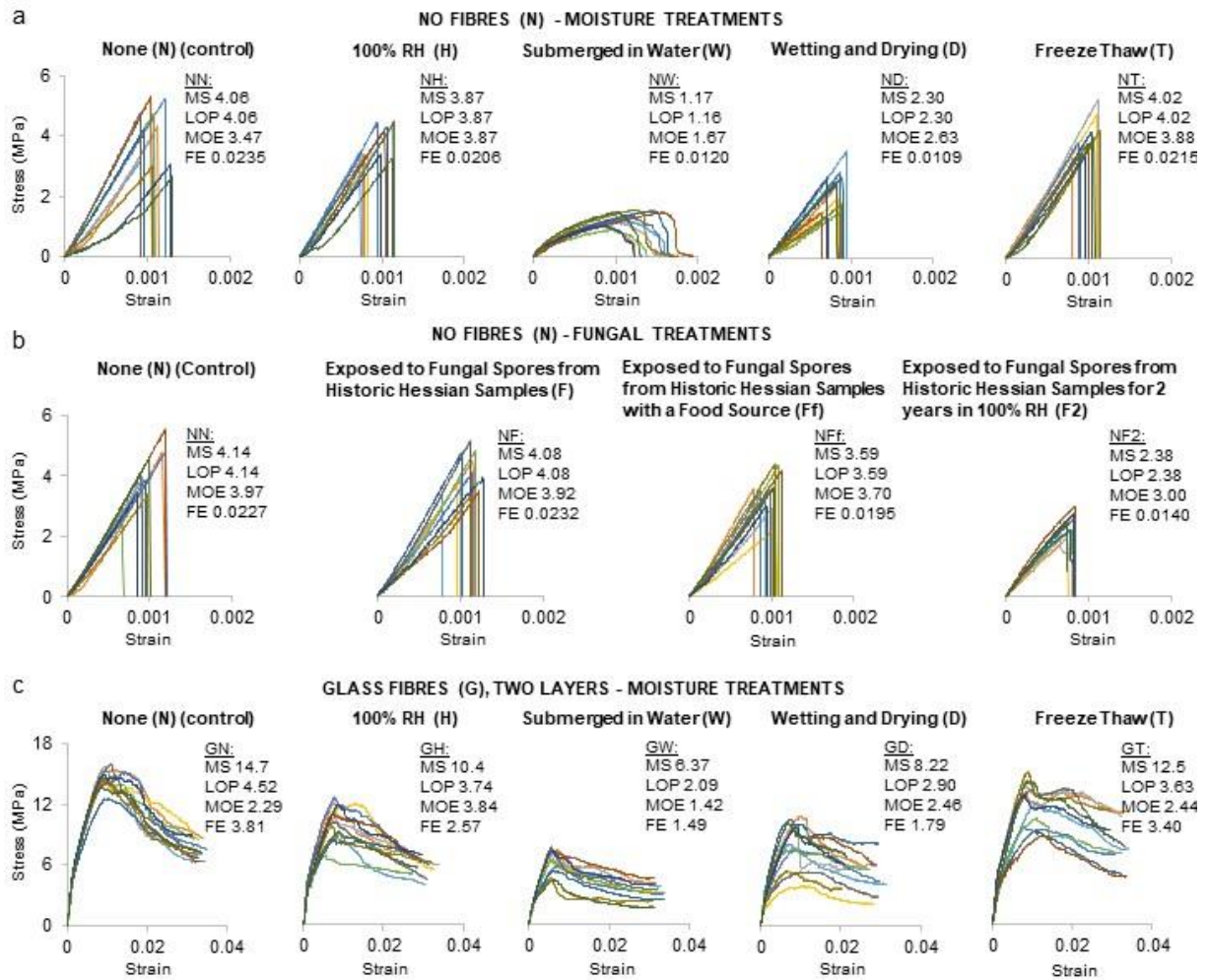
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492 The glass fibre samples (GN, GH, GW, GD, GT) were also all affected by the environmental
493 conditions, to the point of rejecting the null hypothesis, with submerged in water (GW) and
494 wetting and drying (GD) being the most affected with a 56.7% and 44.2% decrease in
495 maximum stress respectively compared to no treatment.

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Figure 5 Part 1 - Three point flexural test results – No Fibres and Glass Fibres, which shows a) the moisture treatment results for specimens with no fibres, b) fungal treatment results for specimens with no fibres and c) moisture treatment results for specimens with glass fibres. All three sample groups show results for Maximum stress (MS, MPa), Limit of Plasticity (LOP, MPa), Modulus of Elasticity (MOE, GPa) and Fracture Energy (FE, kJ/m²). Glass fibre specimens were not subject to fungal treatment.

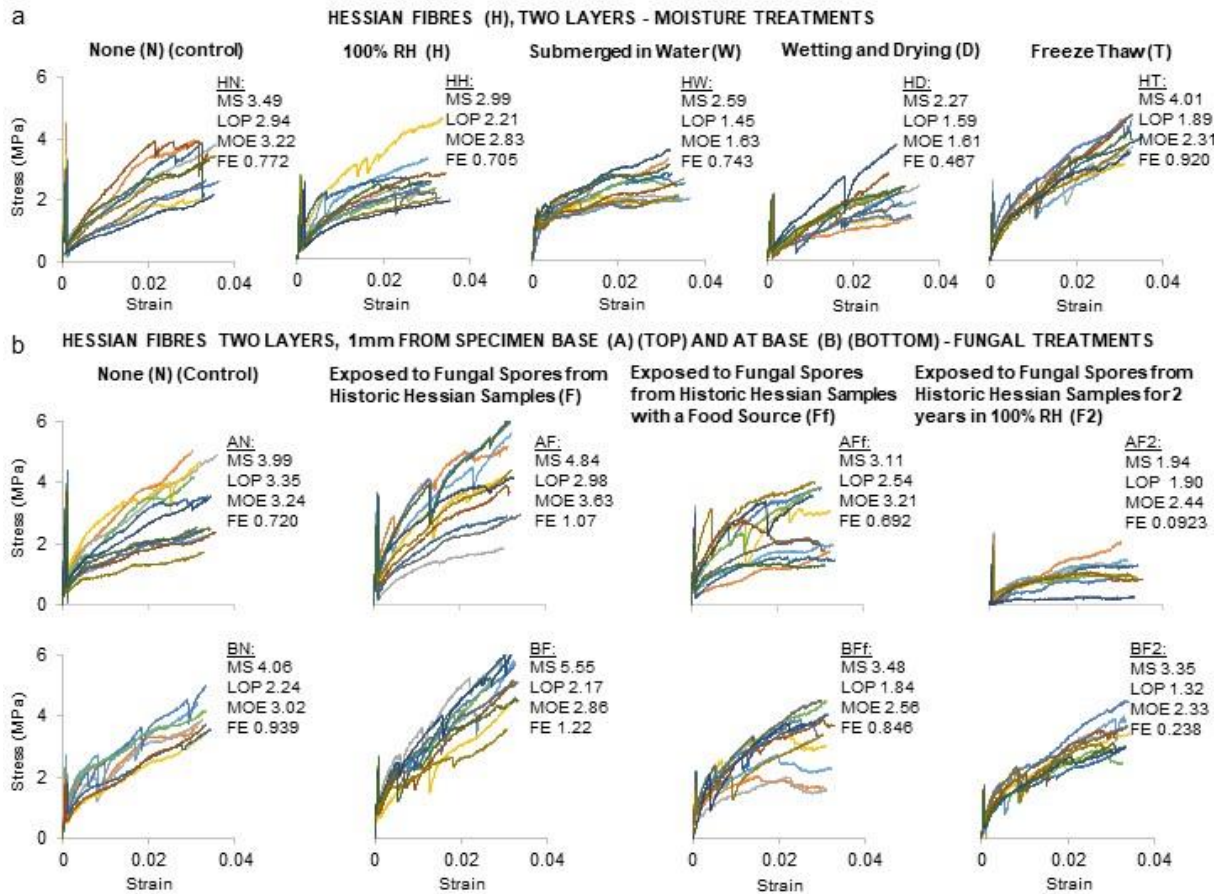


Figure 5 Part 2 - Three point flexural test results – Hessian Fibres, which shows a) the moisture treatment results for specimens with two layers of hessian fibres, b) fungal treatment results for specimens with two layers of hessian fibres 1mm from the bottom of the specimen bases (top) and two layers of hessian fibres right at the specimen bases (bottom). All three sample groups show results for Maximum stress (MS, MPa), Limit of Plasticity (LOP, MPa), Modulus of Elasticity (MOE, GPa) and Fracture Energy (FE, KJ/m²).



Figure 6 - Flexural test specimens subjected to the two-year fungal treatment showing the extent to which fungal growth can be observed on the exterior of the fibrous plaster, and the variation in extent within samples. a) NF2 – no treatment, no fungal growth evident. b) BF2 – subjected to fungal treatment, some growth evident. c) AF2 – subjected to fungal treatment and shows very significant fungal presence on the sample specimens.

520 3.1.2 Limit of Plasticity (LOP)

521 For the unreinforced samples, the results are the same as the maximum stress due to the
522 brittle failure of the gypsum and ensuing stress-strain profile. All conditions affected the limit of
523 plasticity in the samples with hessian reinforcement so the null hypothesis could not be
524 accepted for any conditions, with submerged in water being the most greatly affected (HW,
525 50.7% decrease from HN), wetting and drying (HD), freeze-thaw (HT) and then 100% humidity
526 (HH) the least affected (24.8% decrease) in comparison to HN. The addition of the food source
527 to historic fungi and keeping samples exposed to fungi for two years clearly impacted the
528 performance of the samples with hessian in mechanical testing, with samples subjected to
529 fungi for two years showing a reduce LOP (43% for AF2 and 41% for BF2) in comparison to
530 NF2. Again, for the glass fibre reinforced samples, the LOP was negatively affected compared
531 to the control for every environmental condition, with a 53.8% mean decrease for submerged
532 in water and 35.8% decrease for wetting and drying.

533 3.1.3 Modulus of Elasticity (MOE)

534 The modulus of elasticity for the no-reinforcement samples subjected to moisture treatments
535 was only affected by being submerged in water (NW), where the null hypothesis was rejected.
536 Without the submerged in water and wetting and drying (ND) values in an ANOVA statistical
537 test, the moduli of elasticity for (NN, NH, NT) all match within 63.5% which is much higher than
538 the required alpha value of 5%. The submerged in water test reduced the mean modulus of
539 elasticity by 51.7%. For samples without reinforcement subjected to fungal treatments, being
540 subjected for two years to fungi (NF2) resulted in the largest decrease, 24% in MOE from the
541 unreinforced NN. For the hessian-reinforced samples subjected to moisture treatments, the
542 submerged in water (HW) and wetting and drying (HD) displayed the greatest reduction in
543 MOE, but freeze-thaw (HT) testing also reduced MOE by 28.0%. With the two year fungal
544 exposure samples excluded, samples exposed to the historic fungal fibres all accepted the
545 null hypothesis for the ANOVA tests and student t-tests when comparing conditions for each
546 reinforcement location. When the two year fungal samples are included, the null hypothesis is
547 rejected for both hessian reinforcement locations combined, however when separated into the
548 two different reinforcement locations, AN, AF, AFf and AF2 reject the null hypothesis but BN,
549 BF, BFf and BF2 narrowly accept the null hypothesis. The glass-reinforced fibrous plaster
550 samples were negatively affected by being submerged in water (GW, 37.7% decrease),
551 although the mean MOE increased by 68% for the 100% humidity test (GH).

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Table 3 - Matrix of flexural samples T-test results for MS, LOP, MOE and FE, to demonstrate the variation in the flexural tests between two sets of samples. Green indicates acceptance of the null hypothesis (>0.05); therefore these two data sets are similar, and grey rejection of the null hypothesis (<0.05) which indicates significant difference between the two data sets. Note that MS and LOP are the same for specimens with no fibres. Values are presented to three significant figures.

| MAXIMUM STRESS (MS) | | | | | | | | |
|--|----|-------|-------|-------|-------|-------|-------|-------|
| MS - NO FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | NN | NH | NW | ND | NT | | | |
| NN | - | 0.540 | 0.000 | 0.000 | 0.912 | | | |
| NH | - | - | 0.000 | 0.000 | 0.492 | | | |
| NW | - | - | - | 0.000 | 0.000 | | | |
| ND | - | - | - | - | 0.000 | | | |
| NT | - | - | - | - | - | | | |
| MS - NO FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | NN | NF | NFf | NF2 | | | | |
| NN | - | 0.823 | 0.067 | 0.000 | | | | |
| NF | - | - | 0.000 | 0.088 | | | | |
| NFf | - | - | - | 0.000 | | | | |
| NF2 | - | - | - | - | | | | |
| MS - GLASS FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | GN | GH | GW | GD | GT | | | |
| GN | - | 0.000 | 0.000 | 0.000 | 0.013 | | | |
| GH | - | - | 0.000 | 0.016 | 0.033 | | | |
| GW | - | - | - | 0.024 | 0.000 | | | |
| GD | - | - | - | - | 0.000 | | | |
| GT | - | - | - | - | - | | | |
| MS - HESSIAN FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | HN | HH | HW | HD | HT | | | |
| HN | - | 0.140 | 0.002 | 0.000 | 0.054 | | | |
| HH | - | - | 0.208 | 0.033 | 0.004 | | | |
| HW | - | - | - | 0.183 | 0.000 | | | |
| HD | - | - | - | - | 0.000 | | | |
| HT | - | - | - | - | - | | | |
| MS - HESSIAN FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | AN | AF | AFf | AF2 | BN | BF | BFf | BF2 |
| AN | - | 0.051 | 0.000 | 0.004 | 0.803 | 0.000 | 0.022 | 0.139 |
| AF | - | - | 0.000 | 0.000 | 0.069 | 0.139 | 0.002 | 0.006 |
| AFf | - | - | - | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| AF2 | - | - | - | - | 0.002 | 0.000 | 0.335 | 0.259 |
| BN | - | - | - | - | - | 0.001 | 0.012 | 0.094 |
| BF | - | - | - | - | - | - | 0.000 | 0.000 |
| BFf | - | - | - | - | - | - | - | 0.682 |
| BF2 | - | - | - | - | - | - | - | - |

| MODULUS OF ELASTICITY (MOE) | | | | | | | | |
|---|----|-------|-------|-------|-------|-------|-------|-------|
| MOE - NO FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | NN | NH | NW | ND | NT | | | |
| NN | - | 0.330 | 0.000 | 0.053 | 0.276 | | | |
| NH | - | - | 0.000 | 0.000 | 0.964 | | | |
| NW | - | - | - | 0.001 | 0.000 | | | |
| ND | - | - | - | - | 0.000 | | | |
| NT | - | - | - | - | - | | | |
| MOE - NO FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | NN | NF | NFf | NF2 | | | | |
| NN | - | 0.848 | 0.000 | 0.335 | | | | |
| NF | - | - | 0.004 | 0.523 | | | | |
| NFf | - | - | - | 0.014 | | | | |
| NF2 | - | - | - | - | | | | |
| MOE - GLASS FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | GN | GH | GW | GD | GT | | | |
| GN | - | 0.000 | 0.000 | 0.164 | 0.336 | | | |
| GH | - | - | 0.000 | 0.000 | 0.000 | | | |
| GW | - | - | - | 0.000 | 0.000 | | | |
| GD | - | - | - | - | 0.896 | | | |
| GT | - | - | - | - | - | | | |
| MOE - HESSIAN FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | HN | HH | HW | HD | HT | | | |
| HN | - | 0.093 | 0.000 | 0.000 | 0.026 | | | |
| HH | - | - | 0.000 | 0.000 | 0.179 | | | |
| HW | - | - | - | 0.937 | 0.078 | | | |
| HD | - | - | - | - | 0.064 | | | |
| HT | - | - | - | - | - | | | |
| MOE - HESSIAN FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | AN | AF | AFf | AF2 | BN | BF | BFf | BF2 |
| AN | - | 0.078 | 0.001 | 0.857 | 0.322 | 0.154 | 0.003 | 0.030 |
| AF | - | - | 0.000 | 0.052 | 0.012 | 0.008 | 0.000 | 0.002 |
| AFf | - | - | - | 0.001 | 0.012 | 0.113 | 0.454 | 0.664 |
| AF2 | - | - | - | - | 0.401 | 0.190 | 0.004 | 0.038 |
| BN | - | - | - | - | - | 0.545 | 0.015 | 0.134 |
| BF | - | - | - | - | - | - | 0.065 | 0.369 |
| BFf | - | - | - | - | - | - | - | 0.333 |
| BF2 | - | - | - | - | - | - | - | - |

| LIMIT OF PLASTICITY (LOP) | | | | | | | | |
|---|----|-------|-------|-------|-------|-------|-------|-------|
| LOP - NO FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | NN | NH | NW | ND | NT | | | |
| NN | - | 0.540 | 0.000 | 0.000 | 0.912 | | | |
| NH | - | - | 0.000 | 0.000 | 0.492 | | | |
| NW | - | - | - | 0.000 | 0.000 | | | |
| ND | - | - | - | - | 0.000 | | | |
| NT | - | - | - | - | - | | | |
| LOP - NO FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | NN | NF | NFf | NF2 | | | | |
| NN | - | 0.823 | 0.067 | 0.000 | | | | |
| NF | - | - | 0.000 | 0.088 | | | | |
| NFf | - | - | - | 0.000 | | | | |
| NF2 | - | - | - | - | | | | |
| LOP - GLASS FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | GN | GH | GW | GD | GT | | | |
| GN | - | 0.001 | 0.000 | 0.000 | 0.020 | | | |
| GH | - | - | 0.000 | 0.005 | 0.736 | | | |
| GW | - | - | - | 0.006 | 0.000 | | | |
| GD | - | - | - | - | 0.065 | | | |
| GT | - | - | - | - | - | | | |
| LOP - HESSIAN FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | HN | HH | HW | HD | HT | | | |
| HN | - | 0.012 | 0.000 | 0.000 | 0.002 | | | |
| HH | - | - | 0.000 | 0.001 | 0.139 | | | |
| HW | - | - | - | 0.276 | 0.037 | | | |
| HD | - | - | - | - | 0.187 | | | |
| HT | - | - | - | - | - | | | |
| LOP - HESSIAN FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | AN | AF | AFf | AF2 | BN | BF | BFf | BF2 |
| AN | - | 0.156 | 0.000 | 0.004 | 0.000 | 0.000 | 0.000 | 0.000 |
| AF | - | - | 0.000 | 0.030 | 0.000 | 0.000 | 0.000 | 0.000 |
| AFf | - | - | - | 0.004 | 0.091 | 0.139 | 0.007 | 0.738 |
| AF2 | - | - | - | - | 0.075 | 0.013 | 0.000 | 0.000 |
| BN | - | - | - | - | - | 0.555 | 0.000 | 0.008 |
| BF | - | - | - | - | - | - | 0.000 | 0.005 |
| BFf | - | - | - | - | - | - | - | 0.001 |
| BF2 | - | - | - | - | - | - | - | - |

| FRACTURE ENERGY (FE) | | | | | | | | |
|--|----|-------|-------|-------|-------|-------|-------|-------|
| FE - NO FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | NN | NH | NW | ND | NT | | | |
| NN | - | 0.263 | 0.000 | 0.000 | 0.417 | | | |
| NH | - | - | 0.001 | 0.000 | 0.663 | | | |
| NW | - | - | - | 0.287 | 0.000 | | | |
| ND | - | - | - | - | 0.000 | | | |
| NT | - | - | - | - | - | | | |
| FE - NO FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | NN | NF | NFf | NF2 | | | | |
| NN | - | 0.852 | 0.003 | 0.283 | | | | |
| NF | - | - | 0.000 | 0.139 | | | | |
| NFf | - | - | - | 0.017 | | | | |
| NF2 | - | - | - | - | | | | |
| FE - GLASS FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | GN | GH | GW | GD | GT | | | |
| GN | - | 0.000 | 0.000 | 0.000 | 0.093 | | | |
| GH | - | - | 0.000 | 0.001 | 0.003 | | | |
| GW | - | - | - | 0.115 | 0.000 | | | |
| GD | - | - | - | - | 0.000 | | | |
| GT | - | - | - | - | - | | | |
| FE - HESSIAN FIBRES/ MOISTURE TREATMENTS | | | | | | | | |
| | HN | HH | HW | HD | HT | | | |
| HN | - | 0.434 | 0.677 | 0.000 | 0.047 | | | |
| HH | - | - | 0.588 | 0.003 | 0.006 | | | |
| HW | - | - | - | 0.000 | 0.001 | | | |
| HD | - | - | - | - | 0.000 | | | |
| HT | - | - | - | - | - | | | |
| FE - HESSIAN FIBRES/ FUNGI TREATMENTS | | | | | | | | |
| | AN | AF | AFf | AF2 | BN | BF | BFf | BF2 |
| AN | - | 0.014 | 0.000 | 0.795 | 0.042 | 0.000 | 0.000 | 0.251 |
| AF | - | - | 0.000 | 0.004 | 0.229 | 0.220 | 0.000 | 0.062 |
| AFf | - | - | - | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| AF2 | - | - | - | - | 0.004 | 0.000 | 0.000 | 0.081 |
| BN | - | - | - | - | - | 0.002 | 0.000 | 0.214 |
| BF | - | - | - | - | - | - | 0.000 | 0.000 |
| BFf | - | - | - | - | - | - | - | 0.000 |
| BF2 | - | - | - | - | - | - | - | - |

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559 3.1.4 Fracture Energy (FE)

560 Compared to the plaster samples with no reinforcement, the fracture energy (FE) for the plaster
561 samples submerged in water (NW) and wetting and drying (ND) tests decreased, with the null
562 hypothesis being rejected. Hessian-reinforced plaster subjected to moisture treatments was
563 affected by the wetting and drying (HD) and freeze-thaw conditions (NT). For the wetting and
564 drying test, FE decreased by 27.4%, conversely it increased for the freeze-thaw test by 19%.
565 For the samples exposed to the historic fibres, the hessian-reinforced samples rejected the
566 null hypothesis for the ANOVA tests, as indeed did the unreinforced plaster-only samples
567 subjected to fungal treatments. For both locations of hessian reinforcement without any food
568 source present, FE increased significantly enough for the null hypothesis to be rejected (49.3%
569 increase for 1mm away from bottom of sample and 30% increase for almost at the bottom of
570 the sample). Hessian fibre samples subjected to historic fungi for two years displayed a
571 significant reduction in FE, with ANOVA analysis showing AF2 reducing by 87.2% in
572 comparison to AN, and BF2 reducing by 74.7% in comparison to BN; therefore, the two year
573 treatment showed the greatest impact upon deteriorating the hessian fibres to impact FE.
574 Glass fibre samples had the biggest decrease in FE for 100% humidity (GH), submerged in
575 water (GW) and wetting and drying (GD) tests and the null hypothesis was again rejected.
576

577 To summarise the flexural tests, overall the conditions which affected the samples to the
578 greatest extent and consequently causing a deterioration in the mechanical properties were:

579 Moisture treatments:

- 580 • being submerged in water
- 581 • wetting and drying and

582 Fungal treatments:

- 583 • exposure to the historic fungi
- 584 • subjected to fungus for two years with a food source for the fungal treatments – this in
585 particular showed a very significant negative impact upon FE.
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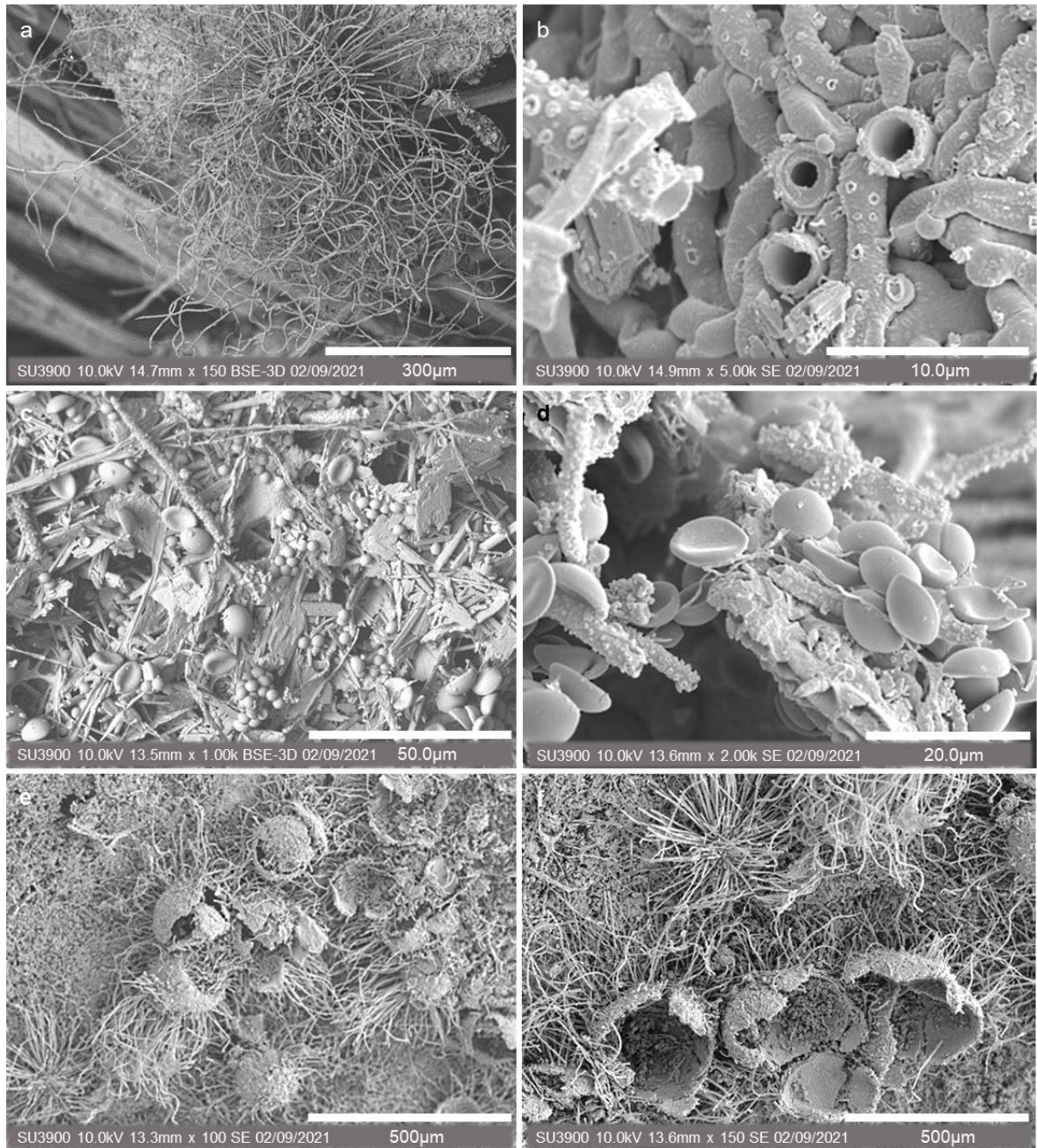
588 3.2 Scanning Electron Microscopy (SEM)

589 After having been exposed to the respective moisture and fungal-related environmental
590 conditions, by observation black-coloured fungal growth was evident on the sample specimens
591 in this study. This was most notable particularly on the 100% humidity samples for the moisture
592 treated conditions and samples which had been exposed to fungi growing on the historic
593 hessian fibres, especially where the fungi had a food source and the specimens were left in a
594 covered box for two years. SEM images of samples HH (100% RH) and BFf (samples exposed
595 to fungi with a food source) can be seen in Figure 7.
596

597 In contrast with the gypsum matrix and hessian fibre SEM images of newly manufactured
598 samples not subjected to moisture or fungal-related treatments illustrated in [8], which showed
599 no visual indications of degradation or fungal growth, entangled masses of hyphae (mycelium)
600 were observed in Figure 7a and b covering the hessian and gypsum plaster matrix. No fungal
601 spores from historic fibrous plaster samples were explicitly introduced in the 100% RH HH
602 specimens., Hence, the fungus is able to grow on the fibrous plaster samples, even when not
603 directly exposed to fungus on historic samples and fungi are able to grow on the gypsum
604 plaster matrix as well as hessian fibres. Some hyphae are broken, exposing the hollow interior
605 of the tubular structure; in addition, exterior surface nodules are visible showing a coarser
606 exterior hyphae surface. Fungal spores are not evident in this image though, suggesting the
607 level of fungal growth and expansion would be lower.
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Figure 7c shows an array of fungal spores on a BFf test sample, growing on the surface of the angular gypsum crystals and 5d reveals a closer look at the spores and hyphae. The malt extract food source is shown in 5e and 5f, being invaded by the fungus - both spores and hyphae are seen to be inside the hollowed-out shells of the food source, with hyphae growing from the food source.



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Figure 7 – SEM imaging showing fungal growth on test samples, a) x150 magnification of a sample in condition HH showing fungal hyphae, b) x5000 magnification of the hyphae in the HH sample, c) x1000 magnification of fungal spores in a sample subjected to the condition BFf, d) BFf spores observed at x2000 magnification, e) Fungal hyphae growing out of the hollow shells of the food source in the BFf sample and f) x150 magnification of hyphae and food source shells in the BFf sample.

622 3.3 DNA Fungal identification

623 Upon completion of SEM tests, DNA sequencing took place on the samples to accurately
624 identify the fungus type. Using BLAST paired up with the DNA sequences, which were found
625 from the samples collected on the agar plates, the closest matches to the fungi on the database
626 were *Penicillium rubens* and *Chaetomium globosum*. These were the only two fungal species
627 isolated from the inside of the samples and the match certainty for these specific fungus types
628 was high; manual assignment of species was based on both sequence similarity to the top hit
629 in the database and on the exclusivity of that hit. For example, a sequence would be considered
630 to represent a species if it had greater than 99% similarity to a named representative of 'that
631 species in the database, but not if it also had equal similarity to another named taxon. With *P.*
632 *rubens* and *C. globosum* identified, specimens of the fungi were grown in sealed petri dishes
633 in an ambient temperature of 20°C for a period of three weeks (with food source particles
634 included within the petri-dish to promote growth) and the ensuing *P. rubens* and *C. globosum*
635 mould growth formed part of the FTIR experimentation and analysis.

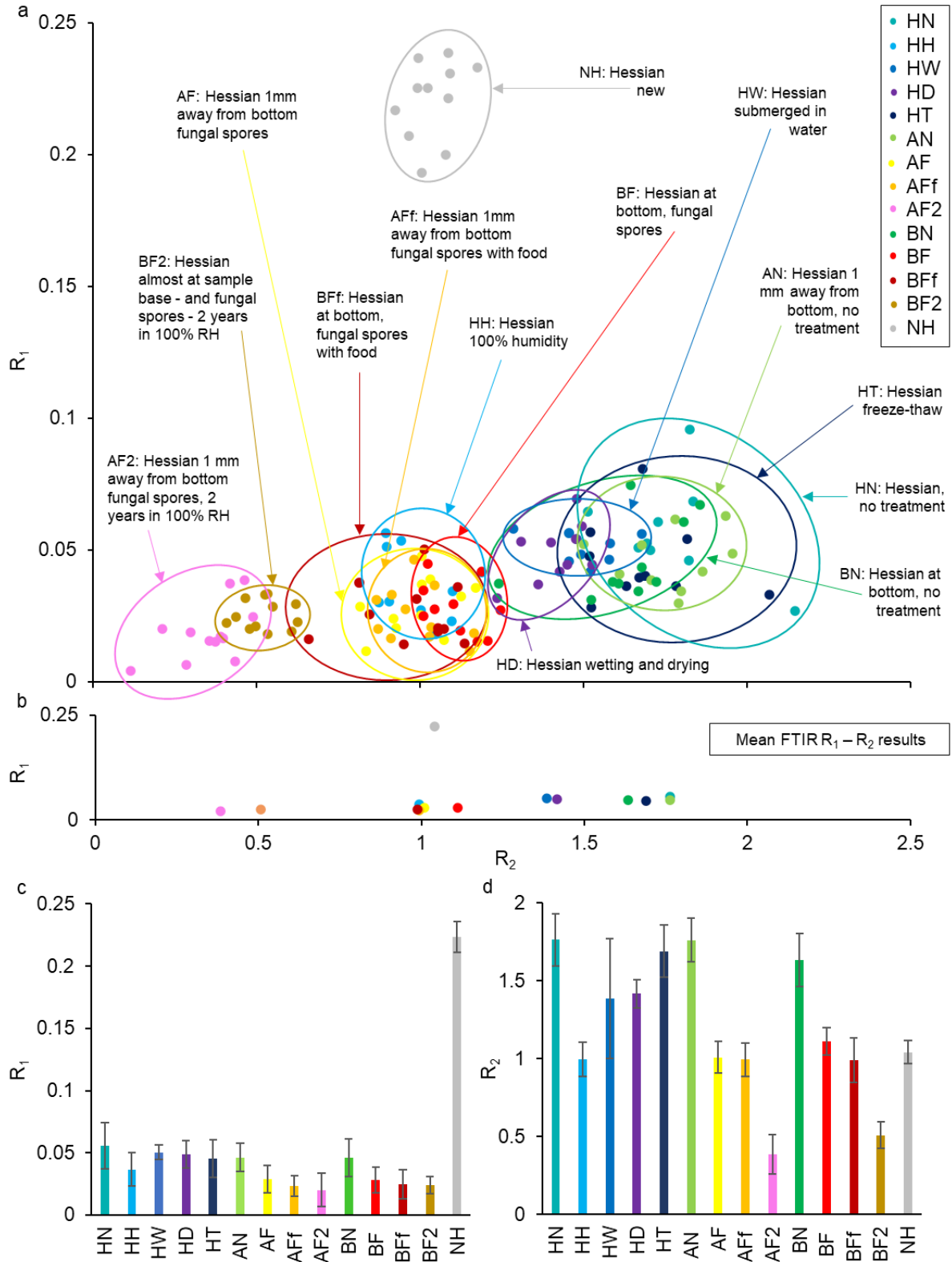
636 3.4 Fourier Transform Infrared Spectroscopy 637

638 The FTIR results for the hessian fibres obtained from the flexural samples are shown in Figure
639 8, with part *a* plotting R_1 versus R_2 for all samples tested to identify the formation of distinct
640 clusters for each treatment category – with multiple hessian fibre specimens from one sample
641 in each treatment category tested. Part *b* plots the mean R_1 versus R_2 values for each
642 treatment category, part *c* shows the mean R_1 values with the error bars denoting the standard
643 deviation and part *d* showing the mean R_2 values also with the error bars denoting standard
644 deviation. Sample conditions which are lower-left in the part *a* and *b* charts are dominated by
645 fungal treatment, with samples subjected to two years (AF2 and BF2 series) with defined
646 clusters in part *a* showing the lowest R_1 and R_2 values. Progressing along the axes, these are
647 followed by BFf and AFf, with the 100% humidity (HH) then following for the moisture treatment
648 samples and ultimately the other moisture treatments and no-treatment samples rightmost.
649 'New hessian' (NH) was not part of a fibrous plaster flexural sample, but new hessian fibres
650 tested for comparison – this group is most distinct at the top of parts *a* and *b*.

651
652 Figure 9 shows the FTIR spectra for the grown *P. rubens* and *C. globosum* moulds, along with
653 the food source and examples of FTIR spectra for the hessian fibres, to show the differences
654 observed in spectra peaks. In part *a*, the fungal treatment category 'A' is shown, with AN (no
655 treatment) and AF2 (exposed to fungi for two years), with fungal growth visually evident on the
656 flexural samples. Treatment category A was chosen to visualise as this represents a wide
657 range of results both in flexural strength and position on the $R_1 - R_2$ plots, with AN performing
658 well in strength tests ranging through to AF2 performing less well. In part *b*, pure *P. rubens*
659 mould is shown, along with new hessian fibres and the new hessian fibres brushed with *P.*
660 *rubens*, to represent an in-situ scenario in a period building where fungi might be present on
661 partially exposed hessian fibres. In part *c*, pure *C. globosum* is shown along with new hessian
662 (untreated) and new hessian brushed with *C. globosum*. The wavelengths used for the peak
663 ratios 1105, 1735 and 2900 cm^{-1} are indicated. At wavelength 1105 cm^{-1} , small shoulder peaks
664 in transmission are more evident for the new hessian fibre samples than on the pure mould
665 spectra. With the 'A' treatment category samples, there are pronounced peaks at 1105 cm^{-1} ,
666 with a reduction in peak intensity for AF2 in comparison to AN. At wavelength 1735 cm^{-1} , the
667 new hessian samples and hessian brushed in mould show small shoulder peaks which are not
668 evident on the pure mould samples. The 'A' treatment category hessian shows small peaks
669 which are quite uniform for the different samples. At 2900 cm^{-1} , the 'A' treatment category
670 samples show a small peak, with variation in the AF2 spectra between 1735 and 2900 cm^{-1}

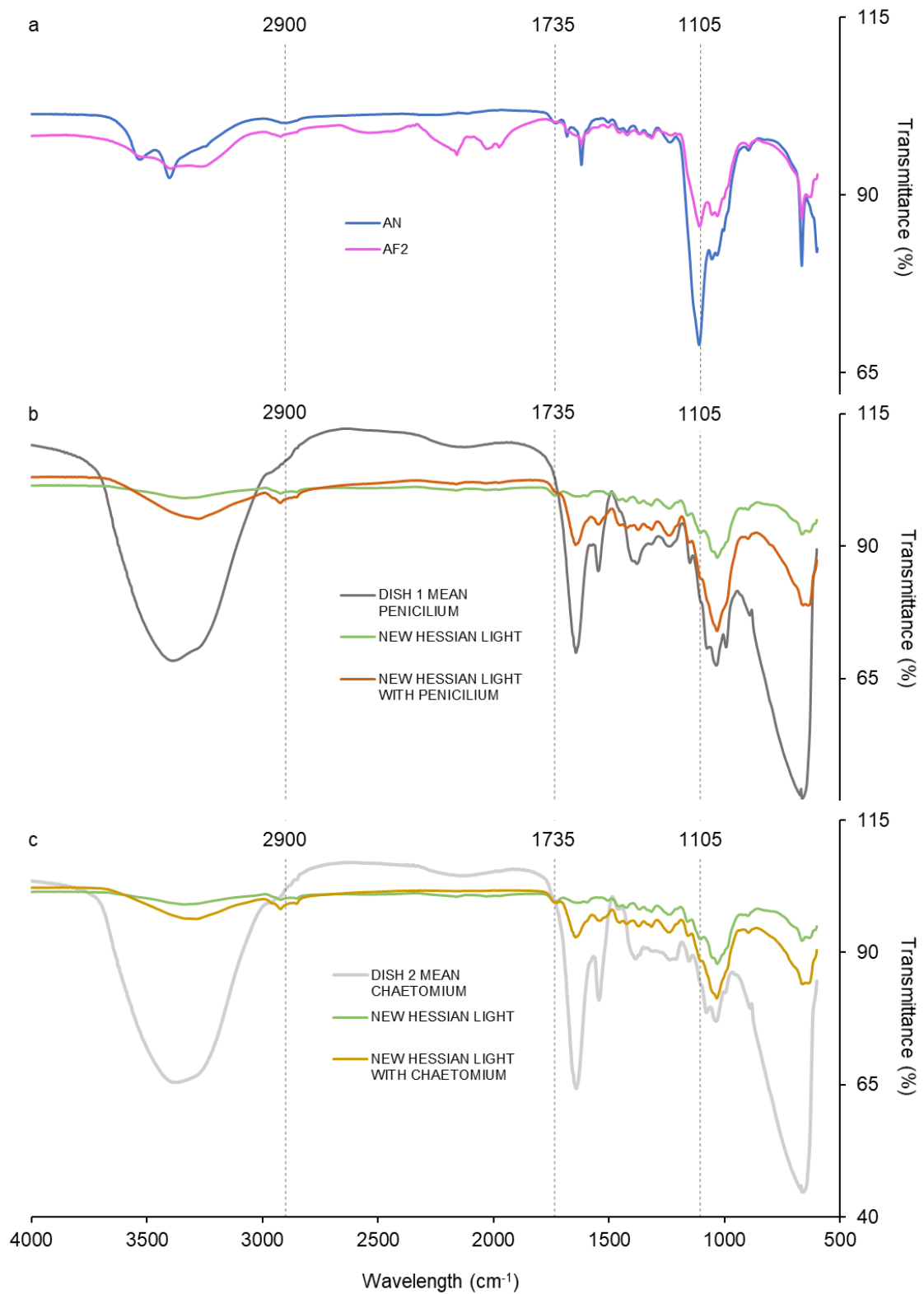
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and from 2900 to 4000 cm^{-1} in comparison to AN. Small peaks are in evidence with the new hessian and new hessian brushed with mould; no peaks are evident with pure mould samples.



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Figure 8 - FTIR peak ratios for hessian fibres taken from the range of moisture and fungal treatment samples a) R_1 and R_2 plotted against each other – at least ten specimens of hessian fibre from one sample from each sample set, b) Mean R_1 and R_2 values from the sample sets, c) Mean R_1 values for each sample set with the standard deviation for each sample set, d) Mean R_2 values with the standard deviation for each sample set.



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Figure 9 – Comparison of FTIR spectra for *Penicillium* and *Chaetomium* moulds along with the food source, hessian fibres from the 'A' range of fungal treatments, new untreated hessian and new hessian brushed in mould to represent an in-situ scenario in a period building of fungi present on fibres. a) Hessian from flexural samples in the 'A' treatment range with AN (no treatment), AF2 (exposed to fungi for two years). b) Pure *Penicillium rubens*, new hessian and new hessian brushed with *P. rubens*. c) Pure *Chaetomium globosum*, new hessian and new hessian brushed with *C. globosum*. The wavenumbers at 1105, 1735 and 2900 used for the R_1 and R_2 peak ratios are indicated by dashed vertical lines.

687 Different mechanisms that affect and degrade fibrous plaster can be evaluated by comparison
688 of the results from the mechanical test data, FTIR results and R_1 versus R_2 plots derived from
689 the FTIR data. The results of three-point bending tests reflect the pre-conditioning by
690 submersion in water, wetting and drying, exposure to fungi with food, and exposure to fungi
691 with food for two years. Unreinforced gypsum was affected by the treatment conditions as well
692 as reinforced plaster, which emphasises the observation that gypsum itself is also vulnerable
693 to experiencing degradation and it is not just an issue for reinforcing hessian fibres. The FTIR
694 data shows defined 'clusters' corresponding with treatment conditions and this, in conjunction
695 with the flexural test results, demonstrates the potential of using FTIR peak ratios for identifying
696 degradation mechanisms of hessian fibres in fibrous plaster and the extent to which the
697 mechanism has caused degradation.

698
699 There is a distinction between moisture conditions and fungal conditions, with the 100%
700 humidity test in the moisture conditions being closest to the fungal conditions in terms of the
701 FTIR R_1 versus R_2 charts in Figure 8. The submerged in water and wetting and drying tests
702 are situated around the median of the sets, so are performing less well than the untreated
703 samples as would be expected from reviewing the flexural data results. There is also less
704 variance in submerged in water and wetting and drying tests data in the FTIR data, so the
705 fibres are affected to approximately the same degree each time.

706
707 Moisture has shown to decrease the strength of natural fibres such as flax by saturation
708 following Fickian diffusion behaviour. Natural fibres absorb more water than glass fibres which
709 is expected due to the material types; the saturation leads to water ageing of the fibres [17].
710 For the submerged in water tests and wetting and drying, this saturation, and therefore loss of
711 strength is likely occurring. *Penicillium rubens* is a fungus commonly found indoors [39] and
712 *Chaetomium globosum* is usually found both indoors and outdoors, so it was not surprising for
713 them to be present on the samples tested. As slow growing species, both types may not be
714 damagingly invasive in the short-term, but there could be potential for degradation over a long
715 period of time. For *P. rubens* to grow quickly, there is a need for a relative humidity level above
716 90% [40]. Indeed, the 100% humidity treatment and fungal treatment test specimens have
717 black mould visible (Figure 6); The photographs are of the most visibly affected samples from
718 the fungal attack as well as those with no reinforcement to demonstrate the difference.

719
720 It can be observed that the FTIR peak ratio approach displays clearly defined clusters of data
721 points for the different fungus and moisture treatments. Working from the left of the R_2 x-axis
722 and moving to the right, it can be observed that fungal treated samples are placed furthest
723 left, followed by the moisture categories of wetting and drying, submerged in water and 100%
724 RH and finally the untreated samples and freeze-thaw to the furthest right. Comparing
725 these FTIR ratio results to the flexural results, it can be broadly reasoned that the most extreme
726 case is the two-year fungal category, followed by the other fungal categories and the moisture
727 categories wetting and drying, submerged in water and 100% RH and then finally the least
728 onerous, along with no treatment at all, being freeze-thaw.

729
730 With the use of peak ratios in this study, it was aimed to demonstrate that the changes in R
731 values observed are due to fibre degradation, rather than just confirmation of the presence or
732 type of fungi. The FTIR spectra show that 'pure' *P. rubens* and *C. globosum* mould is distinct
733 from hessian fibres, with the fibres containing mould applied to the surface using a fine brush
734 (simulating a more realistic in-situ/roof space occurrence with fungi on hessian) showing
735 spectra more in line with hessian fibres non-brushed rather than pure mould. For example, the
736 intense peaks at 1105 cm^{-1} (C-O-C bonds, representing cellulose content) in the fungal

737 treatment specimens 'A' in Figure 9a are not evident in the mould or new (untreated) Hessian
738 spectra in parts *b* and *c*. Therefore, it can be postulated that the FTIR peak-ratios approach
739 can demonstrate a degree of identification of how different fungal and moisture treatments
740 affect hessian fibres.

741
742 The C-O-C bond shows greater absorption at 1105 cm^{-1} in the AN specimens than in the AF2
743 specimens and it is the bond which shows the most notable variation from new hessian. It is
744 reasoned the degradation of cellulose (and possible change in cellulose structure due to fungal
745 attack) in the specimens subjected to fungi for two years contributed to the reduced
746 performance in strength. Penicillium species are capable of secreting multi-enzyme systems
747 capable of degrading cellulose [41] and breaking glycosidic bonds resulting in hydrolysis and
748 the formation of sugar molecules [42]. Hulleman et al. 1994 state that when cellulose changes
749 between different degrees of crystalline to amorphous structure, spectra peaks can alter at
750 numerous wavelengths including 1105 cm^{-1} (due to anti-symmetric in-phase ring stretching at
751 that wavelength) [43]. The difference in peaks between the 'A' treatment samples, new hessian
752 and mould can be reasoned to demonstrate degradation of the cellulose in aged hessian due
753 to the prolonged fungal treatment, resulting in a loss of strength. At 1735 cm^{-1} , the C=O bond
754 showing Pectin does not show a reduction in absorption from AN to AF2 and is not visually
755 notably different to new hessian. New hessian and untreated AN specimens were expected to
756 differ to an extent due to AN specimens being present within a gypsum plaster matrix for the
757 duration of laboratory preparation and testing.

758
759 As discussed by Majumber et al. [27], genes associated with jute varieties affect their ability to
760 resist degradation during cultivation, which could be a reason for fungal degradation in some
761 cases. In Figure 7, hyphae, and fungal spores coat both the gypsum and jute fibres. Gypsum
762 inoculated with *P. rubens* formed mycelium and hyphae on its surface as well as germinating
763 in water [44]. This species grows readily on indoor surfaces and was found to grow in humid
764 conditions on the gypsum [25]. *C. globosum* is known to have degraded jute fibres in storage
765 conditions, which gives a strong suggestion that the fungus could be slowly affecting the
766 strength of the fibres. However, treatment of the fibres was found to reduce its growth
767 significantly [45], meaning fungal growth could be greatly reduced by using chemicals to treat
768 hessian fibres prior to incorporation into fibrous plaster as part of the manufacturing process –
769 though this would naturally introduce cost, time and resource considerations.

770 Moisture and fungal conditions for fibrous plaster degradation are linked, with moisture being
771 present and starting an ageing process (through being submerged in water or wetting and
772 drying for example), which then leads to the growth of fungus which will in turn cause further
773 degradation.

774 Historic fibrous plaster ceilings, in close proximity to external roofs, are vulnerable to water
775 leaks from either the roof or pipes located between the ceiling and the roof, with moisture
776 capable of filtering through to the plaster and hessian. Fluctuations in temperature would also
777 be greater closer to the roof which in extremely cold weather could potentially lead to the
778 freeze-thaw cycles occurring in an uninsulated, non-airtight aged roof space. According to
779 research undertaken in Denmark, the attic of a house had large temperature variations due to
780 the roof having the lowest U-value. In cold, moist outdoor temperatures, there was a higher
781 moisture content inside the roof space, so the historic fibrous plaster ceilings would be exposed
782 to moisture from the roof space above [46].

783
784 Fungal spores are also more likely to be present in these areas due to their proximity to the
785 outside elements from the roof. A roof space is not likely to be cleaned as often as a living or
786 occupied space (even though there can be walkways in the roof spaces of period buildings),
787 leading to the potential for heavy reproduction of fungal spores and food sources for the fungi

788 potentially building up. Interestingly, fungi are often found to attack timber building materials;
789 considering historic fibrous plaster is often suspended from or bound to wooden battens, the
790 presence of fungi is to be expected as wooden structures are often coated in a biofilm of micro-
791 organisms [47].
792

793 The distinctive nature of the flexural graphs for plaster without reinforcement demonstrates its
794 brittle nature and tendency to fail suddenly. Gypsum plaster can be modelled as a lattice
795 structure filled with pores. The pores mean it will have a tendency to let water in which may
796 cause problems, combined with its brittle nature, which then leads to failure problems with the
797 gypsum plaster [48]. The unreinforced plaster during the degradation testing was clearly
798 affected the most by being submerged in water and during wetting and drying tests. Moisture
799 is known to increase the subcritical crack growth and creep within plaster. The needle-shaped
800 gypsum crystals have a weak interface where water decreases the strength of the bonds by
801 thickening the adsorbed water layers, causing crystals to slide over each other [49]. For the
802 samples which were submerged in water, the pores between the gypsum crystals would be
803 fully saturated, leading to the adsorbed water layer becoming over-saturated. Once removed
804 from the water, the pores would still contain the water, which would act as a lubricant to the
805 gypsum particles when the load was applied.
806

807 Material dissolution could be the other potential cause of gypsum weakness, though this cause
808 of failure is much less likely due to the studies undertaken by Reynaud et al. [49]; tests were
809 completed to determine if lubrication or dissolution was the issue by using water and ethanol
810 for comparison. Ethanol will not dissolve gypsum, yet when applied to the plaster, a similar
811 graph to that of water being added presented itself, concluding the lubrication mechanism is
812 the main issue [49]. Wetting and drying tests were the other degradation issue causing
813 problems for all the samples, although to a lesser extent than the submerged in water tests.
814 The drying time allows the surface water to escape, rather than having permanent porosity
815 saturation. Water does become trapped within gypsum plaster once filling up the inner pores
816 [50]. This trapped water then has the same effect as the samples submerged in water with the
817 gypsum crystals sliding.
818

819 An important finding was that the unreinforced plaster was affected by the water as this
820 suggests the plaster, as well as reinforcement, is vulnerable. The mechanism involving the
821 saturated water layer explains the reasoning for failure of the gypsum. Originally the hessian
822 scrim was deemed to be a potential cause of failure with the rotting or degradation of the fibres
823 being a main concern. Stamboulis et al., 2000 discussed fibres swelling from water causing
824 further cracks within plaster, leading to the weakening of the plaster [22]. There is still a
825 possibility that hessian degradation is occurring within the sample, in addition to the
826 aforementioned plaster degradation, but results of this study suggest plaster itself is also a
827 concern. Comparing the difference between the plaster affected by moisture with and without
828 hessian reinforcement can determine the answer; submerged in water sample sets (NW and
829 HW) have similar LOP and MOE, but the MS and FT is much higher as expected because of
830 the hessian reinforcement.
831

832 From this comparison, it can be concluded that a likely cause of failure in historic fibrous plaster
833 through a moisture-related mechanism is the gypsum plaster failing, confirmed by the LOP and
834 MOE being similar, rather than just the hessian fibres degrading. For the samples subjected
835 to fungal treatment, the hessian 1 mm away from the bottom surface of the samples had inferior
836 mechanical properties to specimens with hessian nearly touching the bottom surface. Hence,
837 in flexure there seems to be no discernible benefit in situating the hessian fibre further away
838 from the external surface of the fibrous plaster, due to the hygroscopic properties of gypsum
839 plaster and inherent vulnerability to moisture, and fibres further from the surface being affected
840 by absorbed and retained moisture to a greater extent than fibres closer to the surface.

841

842 It is important to remember the results of this study are based on laboratory experiments, and
843 in real world in-situ applications each fibrous plaster ceiling is an individual creation in a unique
844 environment. However, laboratory experiments can determine material properties and
845 behaviour under stated conditions and contribute towards a scientific basis to the
846 understanding of how a historic fibrous plaster element in-situ may degrade over time and the
847 linked moisture and fungal mechanisms of degradation. The FTIR peak ratio approach
848 demonstrates the potential for identifying the mechanisms of degradation in fibrous plaster
849 elements displaying evidence of aged-related deterioration in real-world applications and
850 historic buildings. Using the R_1 versus R_2 plot approach demonstrated, an FTIR-based
851 approach may be used to analyse historic fibrous plaster specimens. Subject to the consent
852 of building owners, an exposed fibre of in-situ hessian material suitable for FTIR may be taken
853 for laboratory analysis and the peak ratio plot used to determine potential causes of
854 degradation (or lack of degradation if it is in the region of the new fibres on the $R_1 - R_2$ plot).
855 This would provide information on the conditions within the roof space and perhaps identify
856 fungal presence. It was determined by the investigation following the Apollo Theatre collapse
857 in 2013 that failure of the ceiling was due to ageing in the fibrous plaster material rather than
858 direct evidence of any liquid water ingress or action [51]. Hessian wadding tie material is
859 considered to have a finite life [51] which has been postulated as 80 years [52] – therefore
860 degradation of the material is of prime importance. The contribution of this study to
861 demonstrating the influence of fungi and water vapour levels will aid understanding of how
862 vulnerable fibrous plaster is in the environments of theatres and other buildings with ageing
863 building envelopes and high (and inconsistent) occupation levels. Many buildings containing
864 fibrous plaster are listed and therefore protected, with the removal of material often being
865 minimised; identifying degraded areas and harvesting very small amounts of material for
866 analysis would promote effective restoration and conservation of historic and culturally
867 significant buildings.

868

869

870 5 Conclusions

871 This study has demonstrated through a programme of laboratory tests – flexural, SEM, DNA
872 and FTIR - that moisture ingress and fungal attack have a detrimental effect on the mechanical
873 properties of fibrous plaster and the results contribute to evidence that moisture and fungi are
874 major causes of degradation. While experiments took place in a controlled laboratory
875 environment and fibrous plaster elements are typically installed in historic buildings, each of
876 which may be considered a prototype, this study provides a scientific base to add to the
877 empirical understanding of fibrous plaster and its performance and behaviour by providing:

- 878
- 879 • Quantification of the effects of a range of moisture and fungal treatments on the
880 mechanical properties of fibrous plaster
- 881 • The identification of fungi present on historic fibrous plaster samples
- 882 • The use of FTIR and an adapted peak ratio method to identify and analyse the effects
883 of fungi and mechanisms of degradation on hessian fibres, with the breaking of
884 cellulose glycosidic bonds within hessian as a result of fungal exposure identified as a
885 potent degradation mechanism. Test specimens subjected to different moisture and
886 fungal treatments can be identified by defined clusters on an FTIR peak ratio plot and
887 compared with flexural strength results.
- 888

889 This study highlights the importance of fibrous plaster ceilings being surveyed and monitored
890 for signs of moisture ingress or fungal degradation to prevent potentially dangerous failures
891 from happening in the future. Evidence of moisture ingress or fungal damage needs to be
892 addressed and a very small sample of fibre could be harvested in-situ and taken for laboratory
893 analysis. Application of the FTIR-based approach in this study could determine the degradation
894 mechanism and promote efficient restoration and conservation.

895

896 From the research and data analysis in this study, it can be concluded that the highest risks
897 posed to the properties and behaviour of historic fibrous plaster with the moisture treatments
898 are water submersion (which is particularly detrimental to the plaster matrix) and repeated
899 wetting and drying cycles, plus fungal attack with a food source and prolonged exposure over
900 a period of years. Results – in particular the reduction of flexural strength - suggesting attack
901 from fungi over a long time period as being the most onerous degradation mechanism of all
902 for fibrous plaster.

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