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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

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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

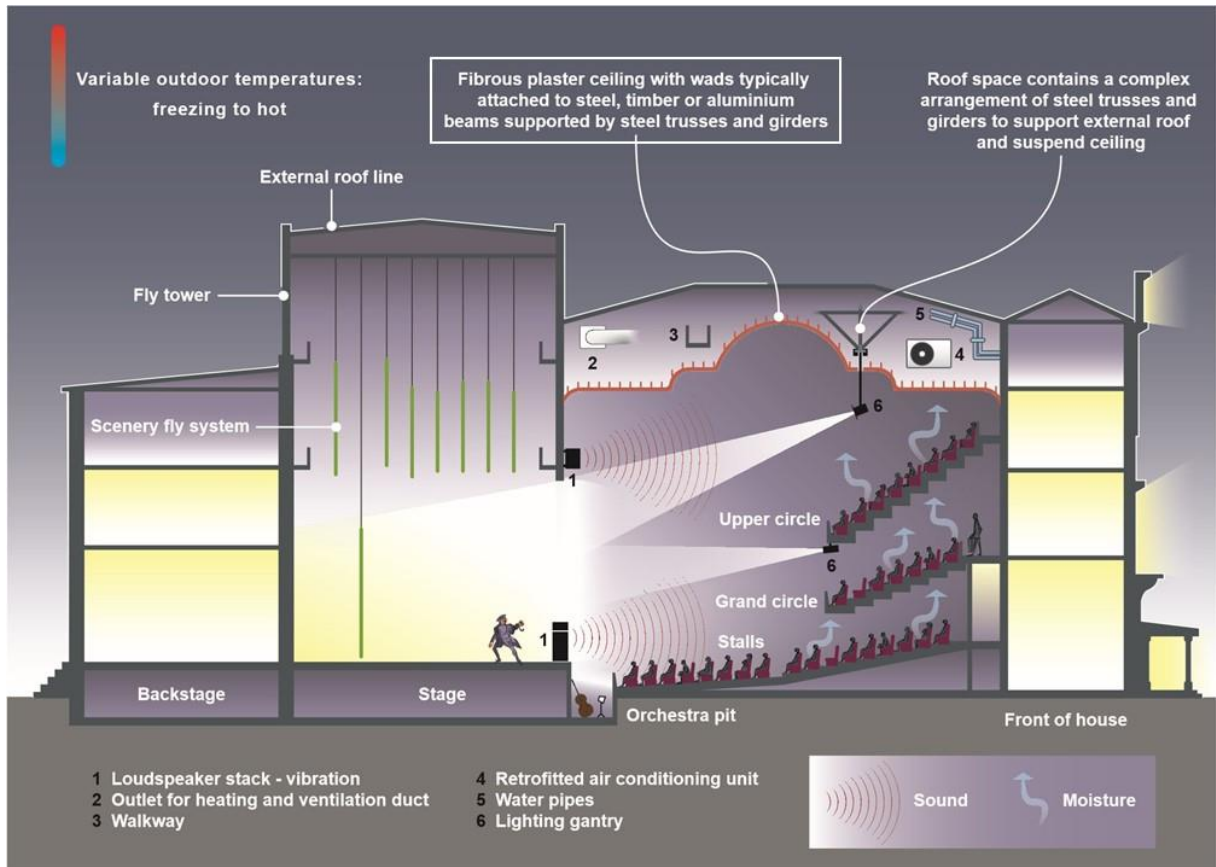
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60 1 Introduction

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 21<sup>st</sup> 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.  
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## 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*

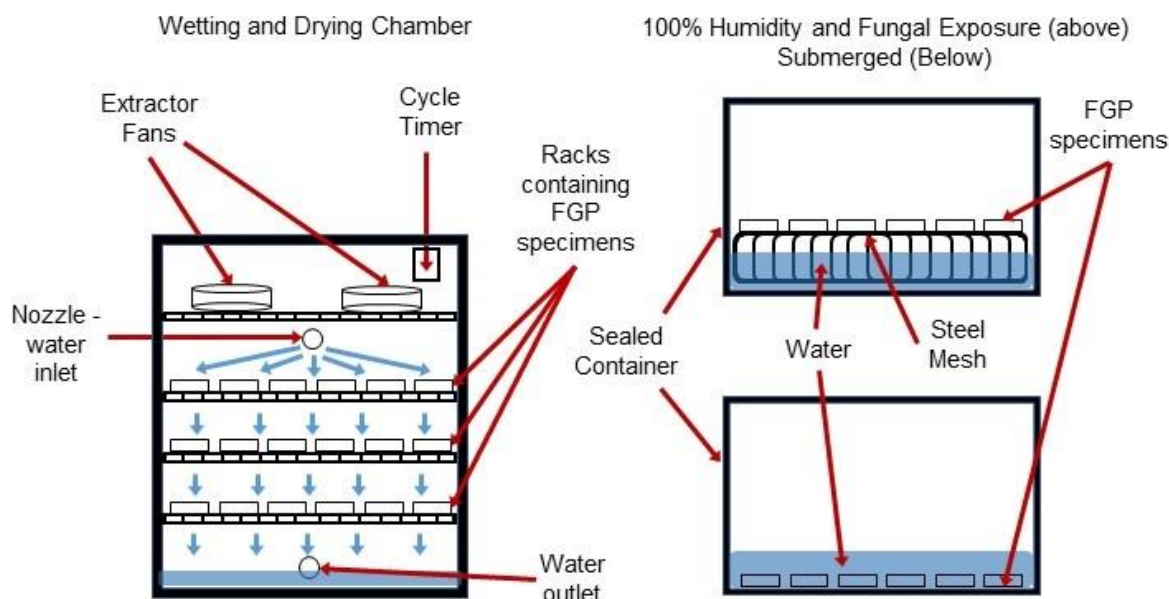
<b>Fibrous reinforcement</b>	<b>No treatment (N)</b>	<b>100% RH (H)</b>	<b>Submerged in water (W)</b>	<b>Wetting and drying (D)</b>	<b>Freeze thaw (T)</b>
None (N)	NN	NH	NW	ND	NT
Two Layers of Hessian (H) fibres (1 mm from sample base)	HN	HH	HW	HD	HT
Two Layers of Glass (G) fibres (1 mm from sample base)	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].  
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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	3 months		24 months
		No food source	food Source	food source
(N)	(F)	(Ff)	(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.

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## 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/m<sup>2</sup> [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/m<sup>2</sup>. 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.

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.

From the load (kN) and displacement (mm) data, stress and strain were calculated. Maximum stress  $\sigma$  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  $\varepsilon$   
 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  $FE$  ( $\text{kJm}^2$ ) 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]:  
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$$FE = \frac{E + Mg\delta_0}{b(d - a)} \quad (4)$$

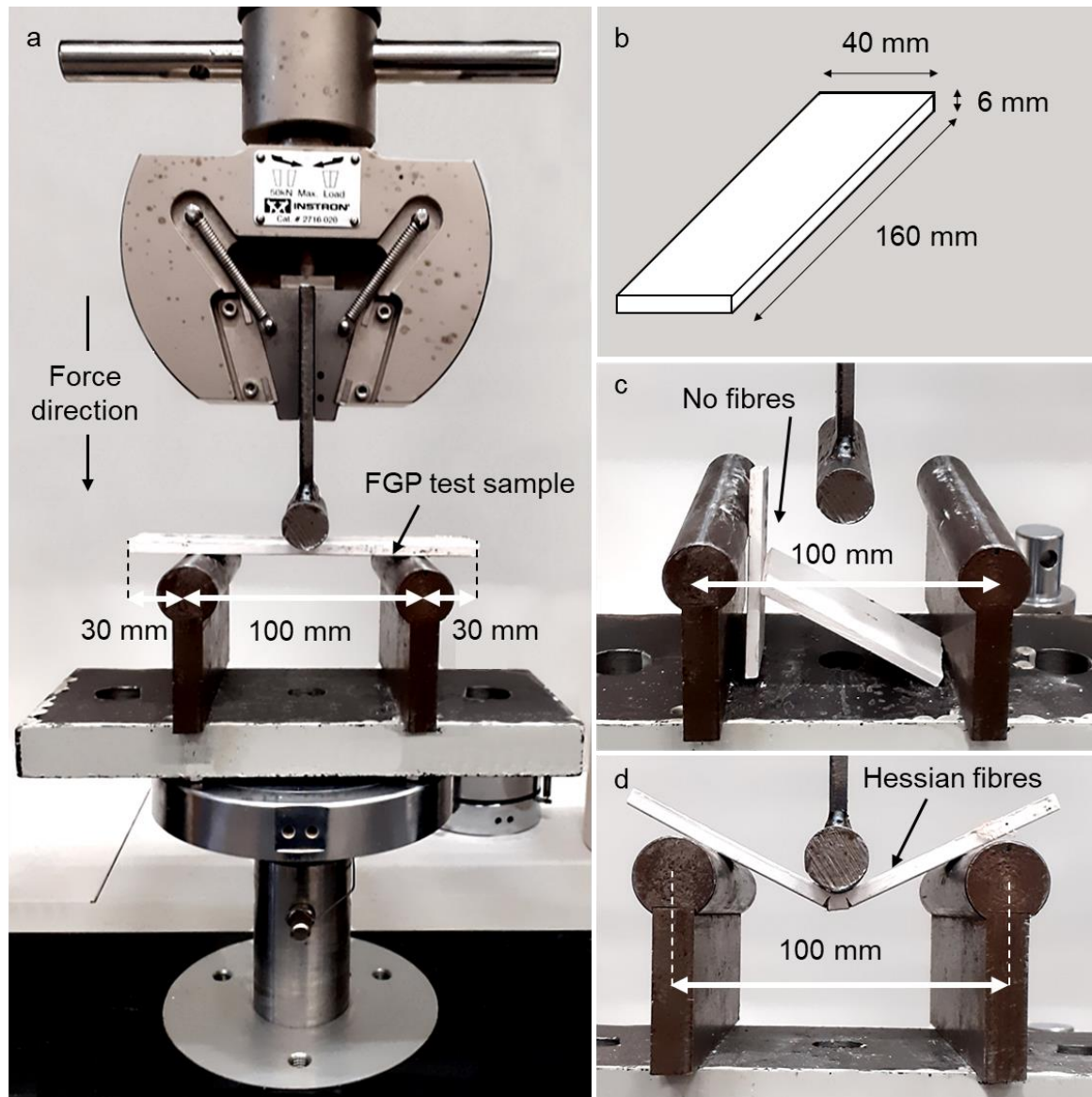
333 where  $E$  is the Fracture Energy (Nmm) calculated as the area under the stable load (N) versus  
 334 deflection (mm) curve (not stress verses strain) until the point of deflection at maximum load;  
 335 this was calculated using the trapezium method and approximated the areas between two sets  
 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  $9.81 \text{ ms}^{-2}$  and  $\delta_0$  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  $\alpha$  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<sup>-1</sup> with a wave number range from 600 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>. 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



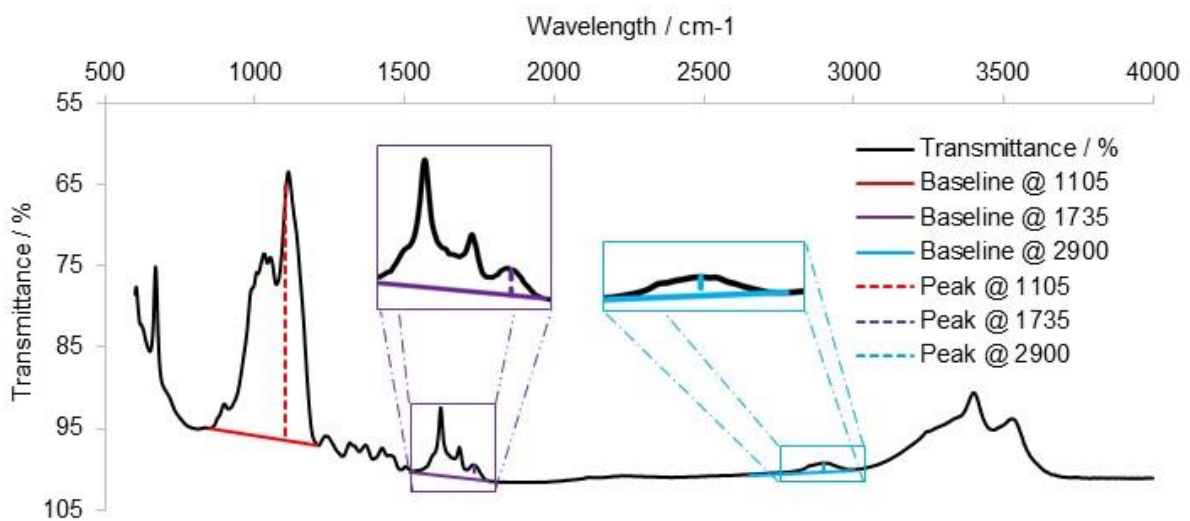
421 Based upon the FTIR peak ratio work of Garside and Wyeth [18], a method using FTIR peak  
 422 ratios (R) (attained by the division of a transmittance peak value at a certain wavelength by  
 423 the peak at another wavelength) was adapted for this study, with two peak ratios named R<sub>1</sub>  
 424 and R<sub>2</sub> plotted on an x and y axis to determine the identification of cellulosic fibres. It was  
 425 aimed that multiple R<sub>1</sub> and R<sub>2</sub> 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<sup>-1</sup> indicating the C-O-C  
 430 glycosidic bond representing cellulose content and  $I_{2900}$  is the wavelength peak height at 2900  
 431 cm<sup>-1</sup> 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<sup>-1</sup> 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<sup>-1</sup> (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<sup>-1</sup> showing pectin is reported as being clearer  
 439 in degraded materials from the carboxyl groups in oxycelluloses [18]. Therefore, in this study  
 440 R<sub>1</sub> and R<sub>2</sub> 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/m<sup>2</sup>) 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.

491

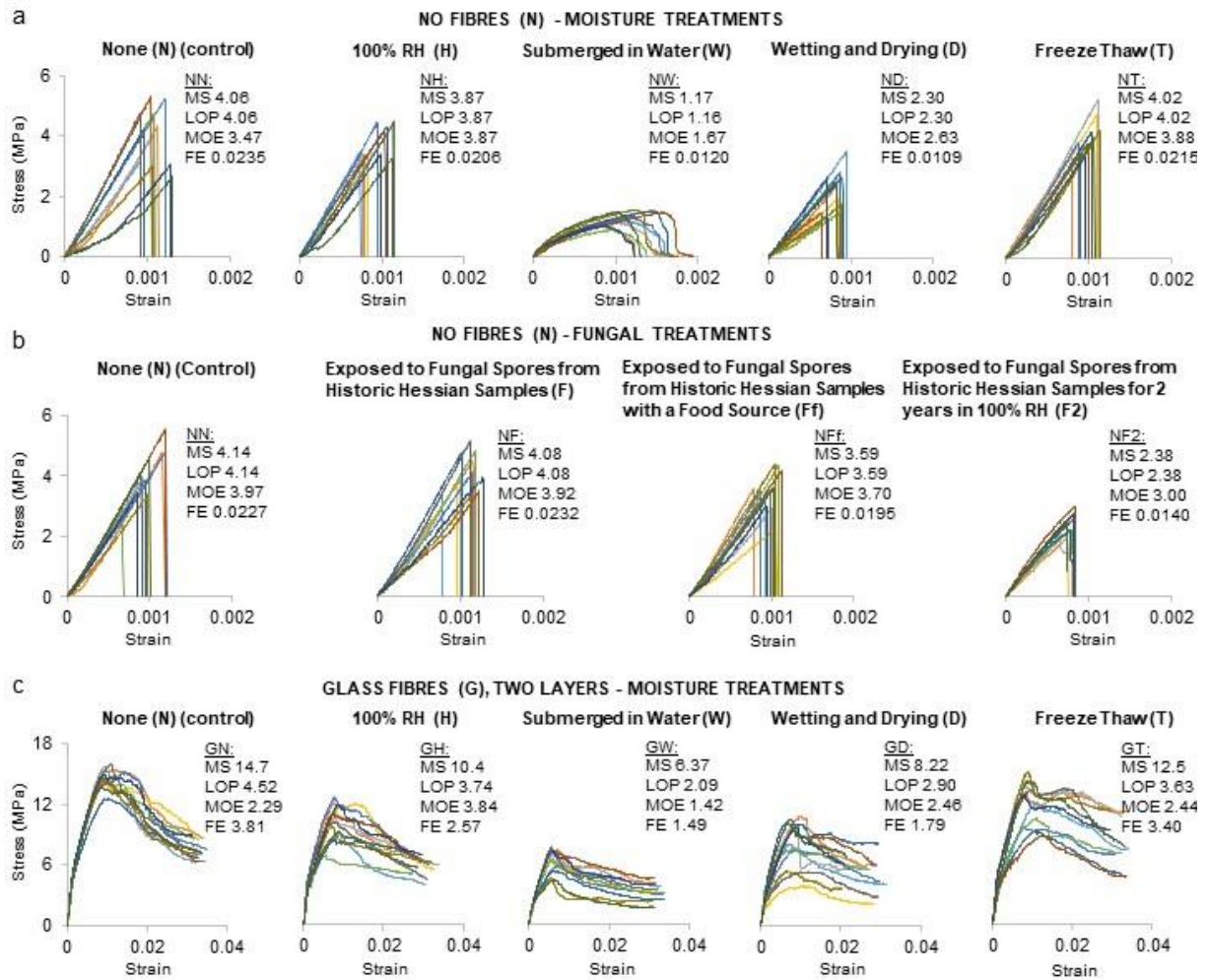
492 The glass fibre samples (GN, GH, GW, GD, GT) were also all affected by the environmental  
493 conditions, to the point of rejecting the null hypothesis, with submerged in water (GW) and  
494 wetting and drying (GD) being the most affected with a 56.7% and 44.2% decrease in  
495 maximum stress 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/m<sup>2</sup>). Glass fibre specimens were not subject to fungal treatment.

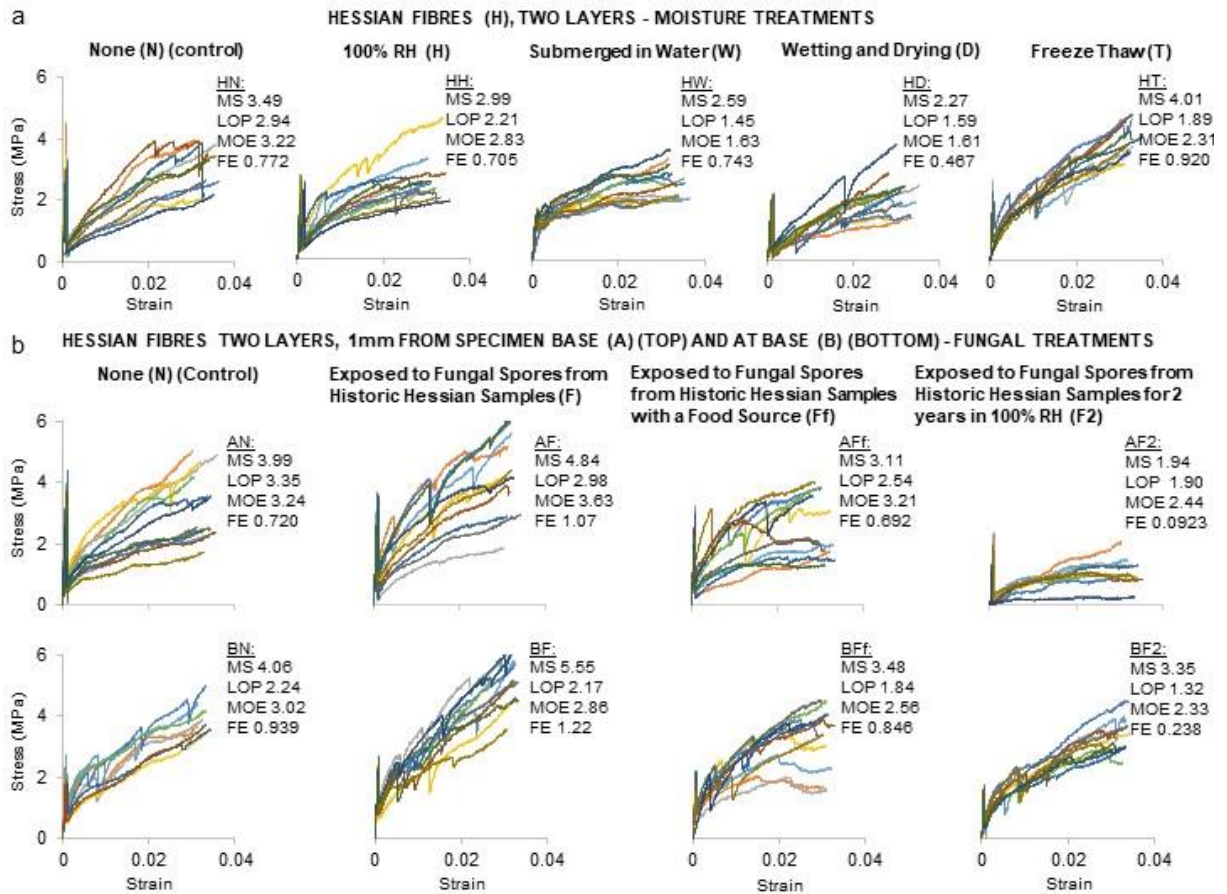


Figure 5 Part 2 - Three point flexural test results – Hessian Fibres, which shows a) the moisture treatment results for specimens with two layers of hessian fibres, b) fungal treatment results for specimens with two layers of hessian fibres 1mm from the bottom of the specimen bases (top) and two layers 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/m<sup>2</sup>).

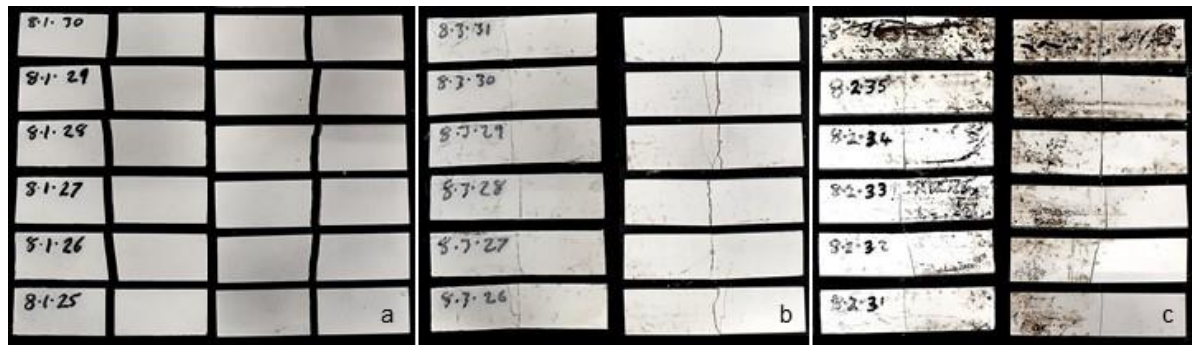


Figure 6 - Flexural test specimens subjected to the two-year fungal treatment showing the extent to which fungal growth can be observed on the exterior of the fibrous plaster, and the variation in extent within samples. a) NF2 – no treatment, no fungal growth evident. b) BF2 – subjected to fungal treatment, 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.
- 586  
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### 588 3.2 Scanning Electron Microscopy (SEM)

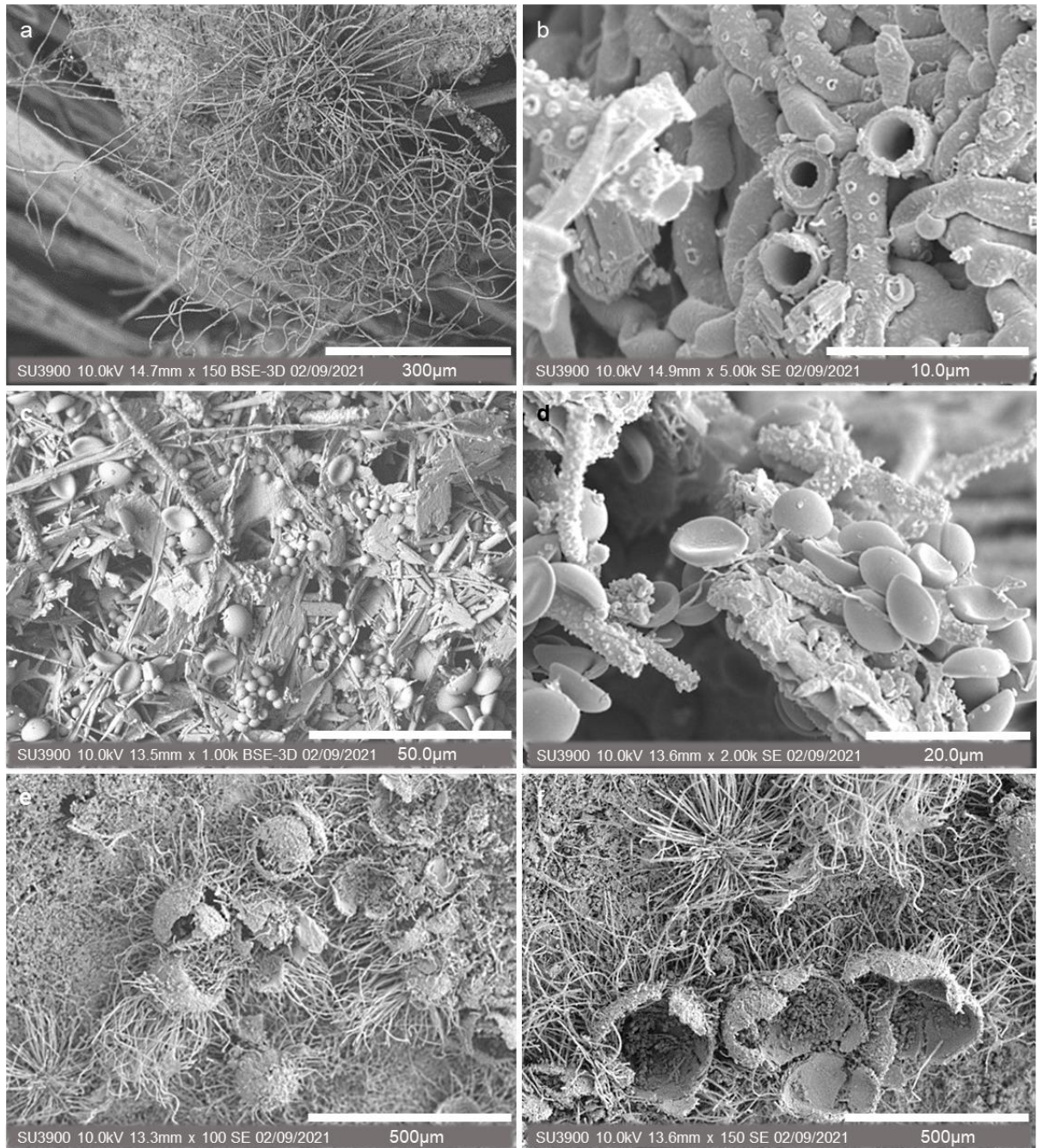
589 After having been exposed to the respective moisture and fungal-related environmental  
590 conditions, by observation black-coloured fungal growth was evident on the sample specimens  
591 in this study. This was most notable particularly on the 100% humidity samples for the moisture  
592 treated 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.

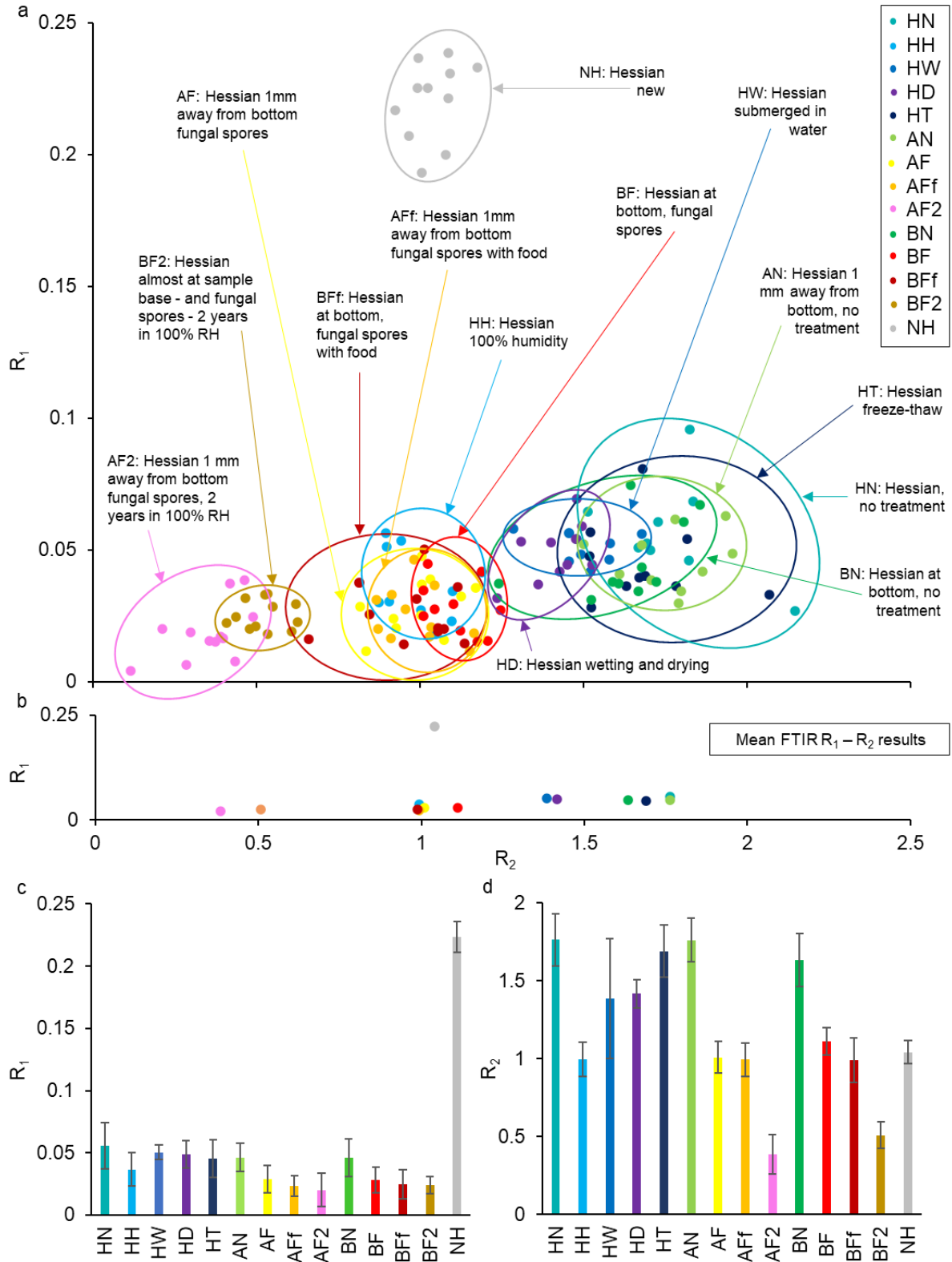
### 636 3.4 Fourier Transform Infrared Spectroscopy 637

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  $\text{cm}^{-1}$  are indicated. At wavelength 1105  $\text{cm}^{-1}$ , 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  $\text{cm}^{-1}$ ,  
666 with a reduction in peak intensity for AF2 in comparison to AN. At wavelength 1735  $\text{cm}^{-1}$ , 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  $\text{cm}^{-1}$ , the 'A' treatment category  
670 samples show a small peak, with variation in the AF2 spectra between 1735 and 2900  $\text{cm}^{-1}$

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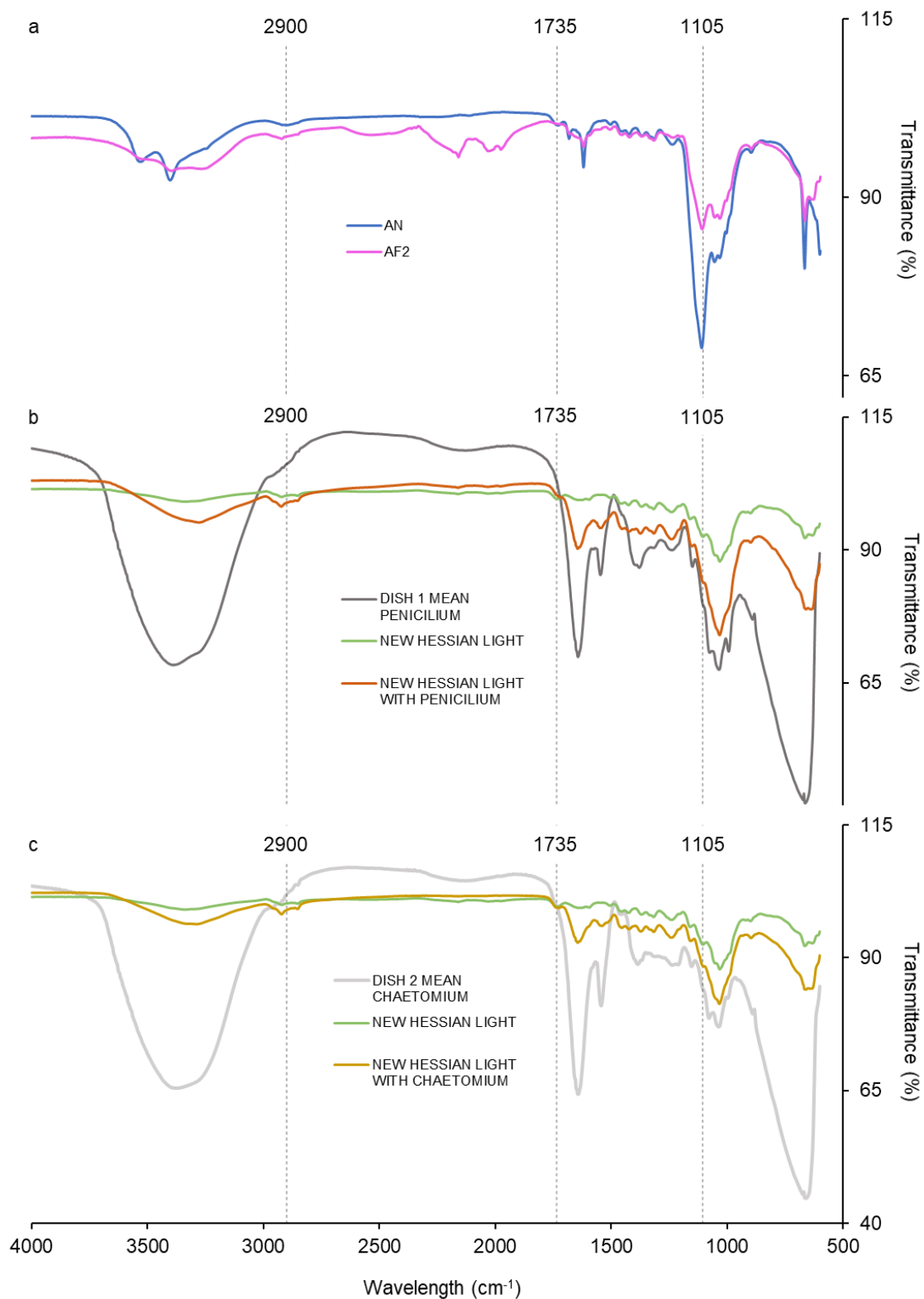
and from 2900 to 4000  $\text{cm}^{-1}$  in comparison to AN. Small peaks are in evidence with the new hessian and new hessian brushed with mould; no peaks are evident with pure mould samples.



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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 wavelengths 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  $1105\text{ cm}^{-1}$  (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  $1105\text{ cm}^{-1}$  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  $1105\text{ cm}^{-1}$  (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  $1735\text{ cm}^{-1}$ , 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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922 manuscript are available from the Dataset for the results of fibrous plaster tests, University of  
923 Bath Research Data Archive, <https://doi.org/10.15125/BATH-01213>.

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