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A review of microstructural characterization and liquid transport in porous materials through image processing techniques

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1 **A review of microstructural characterization and liquid**
2 **transport in porous materials through image processing techniques**

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48 **ABSTRACT**

49 The mechanism of fluid and heat transmission within materials with complex porous
50 structures has not yet been fully explored and understood using basic analytical techniques.
51 Therefore, the lack of advanced equipment and techniques has left an important knowledge
52 gap in explaining the complex mechanisms of fluid motion and heat transfer in complex
53 porous structures. This review provides an overview of how image analysis and processing
54 techniques allow insight into the complex and heterogeneous porous structure of materials
55 and explains the mechanism of heat and mass transfer in these complex porous materials
56 in 3D and 4D observation in different directions. Accordingly, it provides interesting
57 results related to the evaluation of microporous properties of complex porous materials
58 including porosity, distribution and size of pores, distribution and orientation of fibers,
59 tortuosity and mechanism of cracking and destruction of the porous materials under
60 mechanical tests. It also explains the mechanism of liquid transport in porous materials
61 through 3D/4D observation thanks to image processing techniques. Therefore, this review
62 has completed some limited knowledge in microstructural analysis and helped to
63 understand the physical phenomena of liquid transfer in complex porous materials that
64 were not fully exploited by experimental or simulation work. The paper also provides
65 useful data for physical model simulation of imbibition and drying porous materials.

66 **Keywords:** microstructure; image processing technique; tomography; porous materials;
67 liquid transport in porous media.

68

69 **1. Introduction**

70 The microstructure is one of the most important properties because it directly affects their
71 physico-chemical, mechanical, and hygrothermal properties as well as the heat and mass
72 transfer mechanism within the porous materials. The microstructure including porosity,
73 pore size and shape, pore surface area, and pore distribution of the materials are usually
74 studied by experimental analysis techniques such as Brunauer-Emmett-Teller (BET),
75 mercury intrusion porosimetry (MIP), and nuclear magnetic resonance (NMR) (Lai et al.,
76 2018) and classic image processing methods (Arashpour et al., 2021). However, these
77 methods have many limitations because they cannot fully describe the porous structure
78 properties of materials such as the inability to measure closed pores or the connection
79 between pores as well as some limitations observing the distribution of pore and
80 reinforcement phases inside the materials. In fact, the development of image techniques
81 such as tomography or synchrotron enables the observation of the three-dimensional (3D)
82 structure of materials at a high resolution. Accordingly, a series of two-dimensional (2D)
83 images obtained from tomography/synchrotron techniques was processed through image
84 processing techniques to analyze and extract the porous characterization that was not
85 possible with previous experimental techniques. In the literature, tomography has
86 combined different image processing techniques to analyze a series of images obtained
87 from this device regarding the microporous structure of different materials. Accordingly,
88 the 3D porous structure of poplar wood was observed at a very high resolution of about
89 400 nanometers thanks to nanosource X-ray tomography (Perré et al., 2022). Accordingly,

90 the morphology and function of pits on the cell wall of wood were observed in detail to
91 explain the physical transport phenomenon inside this wood and confirmed the proposed
92 hypotheses. Furthermore, the porous structure of biobased composites reinforced with
93 natural fibers has also been studied using tomography combined with Avizo and ImageJ
94 software for image processing (Nguyen et al., 2022a). Accordingly, the porous properties
95 including porosity, distribution, and size of pores and fibers inside the composites have
96 been processed and reported. Similarly, the microporous characterization of various natural
97 fiber-reinforced concrete materials has also been studied and reported in the literature (du
98 Plessis and Boshoff, 2019; Pham et al., 2020b; Ríos et al., 2019). The morphological
99 characteristics of aggregate concrete recycled were also investigated using digital image
100 processing technique (Ma et al., 2021). Accordingly, the 3D observation of the pore and
101 fibers has been processed and represented through different colors inside the materials.
102 Moreover, the porous properties of different sandstone and carbonate samples (Safari et al.,
103 2021) and some porous materials for geoscience applications were also investigated and
104 reported in the literature (Luo et al., 2019; Roostaei et al., 2020; Song et al., 2019a; Song
105 et al., 2019b) using X-ray microtomography (μ CT) and scanning electron microscopy
106 (SEM) techniques in combination with image processing techniques. In addition, combined
107 tomography with image processing was also applied to detect microcracks and crack
108 development mechanism inside materials through 3D observations (Flansbjer et al., 2018;
109 Rucka et al., 2021). These results have allowed to explain the failure mechanism of
110 materials when subjected to external forces through methods of testing mechanical

111 properties. The wood and bio-sourced materials are become more interesting today,
112 especially, in the field of civil construction because of the increasing demand for
113 sustainable buildings including hygrothermal comfort and efficiency and reduction in CO₂
114 emission to the environment. Many European countries have introduced regulations on
115 increasing the content of green materials such as wood and bio-sourced materials in new
116 constructions to reduce the environmental impact reported in many regulations. As a result,
117 wood or wood-based materials such as cross-laminated timber (CLT) have been
118 particularly interested in using in high quantities. In fact, at the construction site, the contact
119 of wood materials with liquid water is inevitable. Wood is a highly hygroscopic material,
120 the contact of wood with water causes serious problems such as swelling, structural damage,
121 and creating a humid environment conducive to the growth of fungus and threatening the
122 works safety. Therefore, to minimize and avoid problems arising and protect wood-based
123 materials at construction sites, the interactions with water need to be understood first. In
124 addition, emerging problems need to be carefully evaluated for precaution. Warnings must
125 be given as regulations to guide construction sites to eliminate potential problems as much
126 as possible. In fact, the interactions between water vapor and wood or wood-derived
127 materials have been reviewed and reported for a long time (Engelund et al., 2013; Nguyen
128 et al., 2021a; Thybring et al., 2022). The behavior, interactions, hygrothermal and
129 durability properties of wood and bio-sourced materials with water vapor have been
130 reported (Nguyen et al., 2018a; Nguyen et al., 2017; Promis et al., 2018; Promis et al., 2019;
131 Rahim et al., 2017). However, so far, there is still a lack of information, actual data, or

132 predictive models between these materials and water (liquid or vapor phase) to guide the
133 construction sites. Furthermore, the mechanism of liquid water penetration into wood and
134 evaporation of water from wood and bio-sourced materials under different conditions
135 (pressure or surface heating) has less been established and specifically reported.

136 This review provides detailed approaches in image processing techniques to characterize
137 the microporous structure of porous materials including wood, concrete, and composite
138 materials reinforced with natural fibers. Accordingly, a series of image processing methods
139 have been synthesized and provided to fully analyze the porous properties of these
140 materials from the shape and pores/fibers distribution to the total porosity calculation. In
141 addition, the pores connection, the tortuosity, and the interface between the phases inside
142 the materials are also analyzed to explain the microstructure influence on physical,
143 physicochemical, mechanical, and other properties of the porous materials. In addition,
144 tomography techniques combined with image processing methods are also applied to
145 explain the mechanism of crack formation and internal destruction of the materials under
146 the influence of external force. Finally, the movement of liquids including water and oil
147 inside porous materials through imbibition and drying is also observed in 3D through these
148 techniques to give new knowledge and analytical methods related to the field of physics
149 transfer within bio-based porous materials.

150 **2. Pore identification and characterization**

151 **2.1. A pore type classification**

152 In the literature, pores could be divided into different types due to their diameter:
153 micropores smaller than 1.25 nm, mesopores from 1.25 nm to 25 nm, macropores
154 macropores from 25 nm to 1 μm , and other large pores from 5 μm to 50 μm (Moravcová
155 et al., 2016). They can also be divided into gel pores (1-5 nm), capillary pores (5-4000 nm),
156 entrained air pores (1-2000 μm), and entrapped air pores (1-10 mm) according to their
157 characteristics (Moravcová et al., 2016).

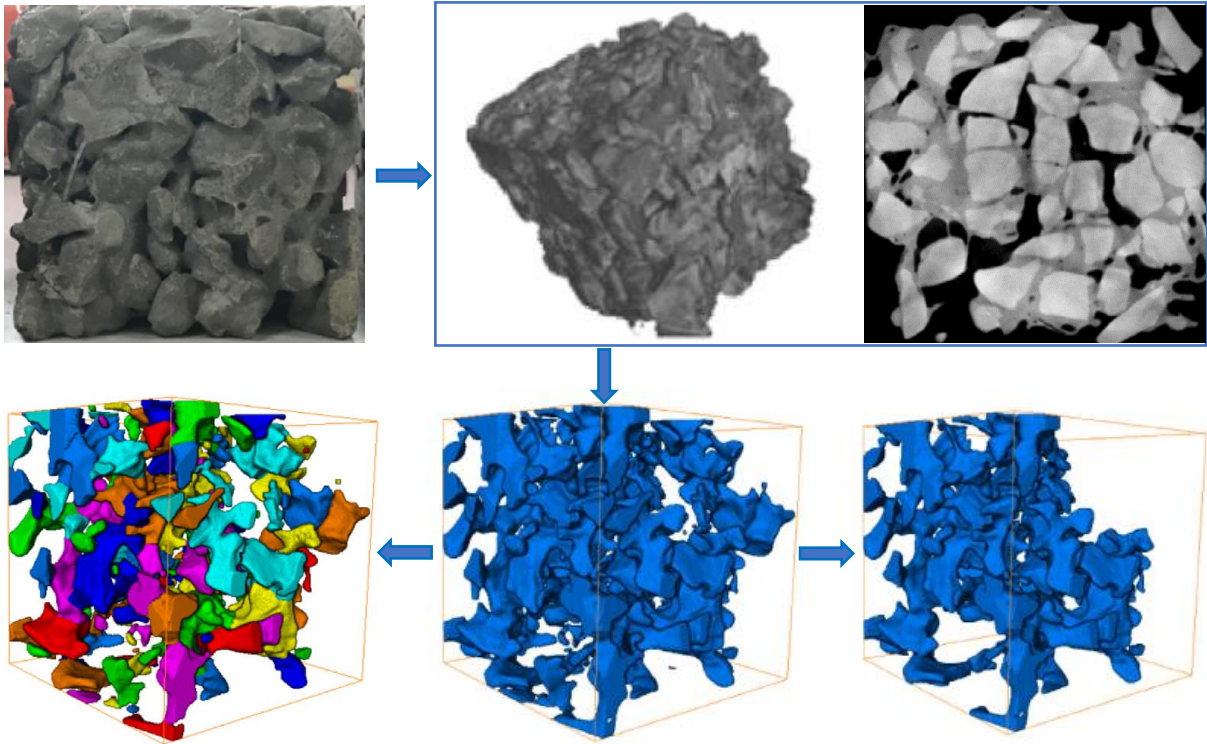
158 **2.2. Pore geometry and characterization**

159 Direct and indirect measurement methods have been used to acquire information on pores
160 (Wu et al., 2019). Direct methods include the mercury injection capillary pressure method
161 (MIP, AutoPore IV mercury porosimeter, in the range of 5 nm to 10^6 nm) (Niu et al., 2020),
162 using air-pore analyzers like RapidAir 457 (Carlson et al., 2006), gas adsorption method
163 using Autosorb-1-c (Zhang et al., 2017), etc. The latter normally indicates image-based
164 techniques that can detect not only air pores but also fibers in the meantime. Image-based
165 techniques, both 2D and 3D, will be mainly discussed in this section. Problem of this
166 method is high pressure which can lead to damage the porous structure. Thanks to rapid
167 advances in imaging technology, such as scanning electron microscope (SEM, 2D), X-ray
168 computed tomography (X-CT), Nuclear Magnetic Resonance (NMR), X-ray microscope,
169 the structure of concrete can be observed at different scales. Lim et al. (Lim et al., 2018)
170 used SEM combined focused ion beam method to obtain 3D images of concrete, the
171 material was iteratively milled each 10 nm and then scanned by SEM. The resolution
172 reached up to 10 nm, therefore, not only the porosity, and pore diameter but also the

173 capillary pores and the gel pores could be detected. Micro-computed tomography (μ -CT)
174 is one of the most popular methods to visualize concrete, and the specimen can be
175 reconstructed by associate software such as Avizo (Nguyen et al., 2021a), Volume
176 Graphics VGSTUDIO MAX (Borges de Oliveira et al., 2016; du Plessis and Boshoff,
177 2019), TXM Reconstructor (Bacaicoa et al., 2017), Amira ZIB Edition (Trofimov et al.,
178 2018), etc. Once the 2D/3D image of the specimen is obtained, various image processing
179 methods such as Avizo, ImageJ, coding software such as MATLAB, Python, C++, a
180 combination of these, etc. can be applied to obtain more information (Nguyen et al., 2021a).
181 The image processing procedure is similar to that in other fields of life and medical science.
182 Normally, these commercial software programs work well, are more friendly, and are
183 easier for operators to use, but their algorithm is unknown, which can lead to confusion,
184 especially in rigid research areas. For ImageJ, a great deal of research and testing must be
185 done to select an appropriate package, users need to code themselves in the non-standard
186 case, and a reputable operation needs to be performed when too many datasets exist.
187 Nevertheless, ImageJ is a convenient open-source software and has a huge package.
188 Practically, in-house codes, written by MATLAB, Python, C++, or other software, are
189 more useful and can satisfy those special cases and each procedure is clear. Those programs
190 are written in a laboratory and are used for future researchers, therefore, continuous work
191 in a laboratory shows their own advantages. To conclude, users should choose the
192 appropriate methods according to their own situation. Bordelon et al. (Bordelon et al., 2020)

193 detailed the image processing method for pores and fibers detection, which may be useful
194 for beginners who want to understand or use the image processing method.

195 Pore size distribution, porosity, and the fractal dimension of the pore surface are three
196 crucial parameters to express pore characteristics. The pore size distribution has been
197 studied in both 2D and 3D, and pixel (or voxel) number counting is the most convenient
198 size measurement method. 2D pore size distribution could be obtained by the average of
199 10 Feret diameters and 3D pore size distribution by the average equivalent diameter of
200 pores as regarded as spheres (Wen et al., 2020; Yuan et al., 2018). Considering the real
201 feature of pores in the concrete, which is no-spherical, the chord-length distribution was
202 also used to describe the pore size distribution (Pandit and Ranade, 2016). Lately, Chung
203 et al. (Chung et al., 2020) reported that the chord-length distribution was closer to the result
204 using the conventional method of RapidAir than the volume-based pore diameter
205 distribution. Wen et al. (Wen et al., 2020) used ImageJ to analyze 2D images (Figure 1 top-
206 right) and Avizo to analyze 3D images (Figure 1 top-center) which were obtained by μ -CT
207 and reconstructed by the associate software Avizo. The 2D pore size was calculated by the
208 average of 10 Feret diameters of all the 720 slices and the 3D equivalent diameter was
209 generated from pores volume considering the pores as a sphere (Figure 1 bottom). Wen et
210 al. (Wen et al., 2020) reported that the pore size ranged from 0 to 15 mm in 3D analysis,
211 while the pore size ranged from 0 to 45 mm in 2D analysis and that the pore size obeyed a
212 normal distribution with a large number of small pores, regardless of the size of the
213 concrete aggregates both in 2D and 3D analysis.

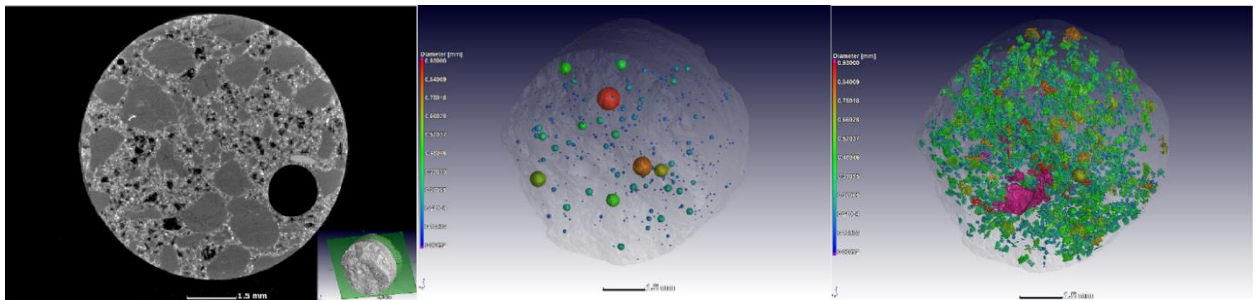


214

215 Figure 1 Specimen (top-left), 3D images reconstructed by Avizo (top-center) and 2D
 216 slice of specimen treated by ImageJ (top-right) based on the scanning data from μ -CT,
 217 pores of the specimen generated by the image processing function of Avizo (bottom-center),
 218 pores colored according to their sizes (bottom-left), and the connected pores (bottom-right)
 219 (Wen et al., 2020). The specimen size is $(100 \text{ mm})^3$.

220 Porosity can be obtained by setting the spherical and size parameters empirically, as shown
 221 in Figure 2: the left image displays a slice of the μ -CT image of concrete, then segmentation
 222 was performed by the threshold, and finally, the porosity was generated based on spherical
 223 (sphericity > 0.6 and diameter $> 60 \mu\text{m}$) or non-spherical pores (sphericity < 0.6) (du
 224 Plessis and Boshoff, 2019). The porosity generated from 2D and 3D images got the same
 225 value and no difference from the experimental value, which was obtained by Archimedes'

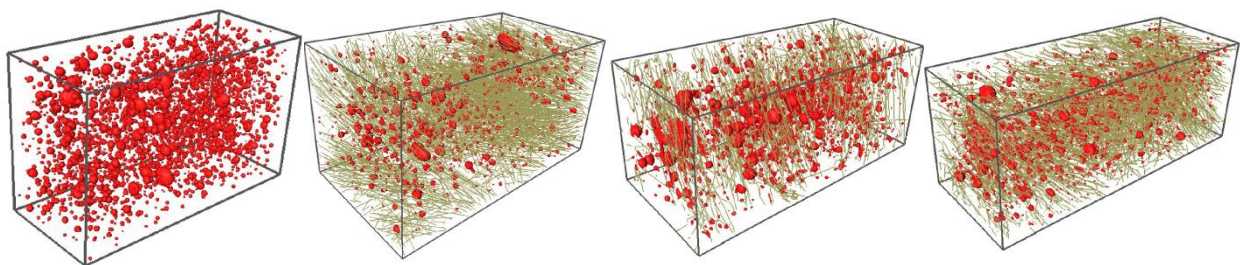
226 principle-based method performed by immersing the concrete in water (Wen et al., 2020).
227 Based on pores volume separated by pore throats, Yu et al. (Yu et al., 2019) found that
228 pores are more like an ellipse and express the shape of pores by the number from 1 to 6,
229 which was calculated using the major axis divided by the minor axis to express how close
230 the pores to a circle. Zhang et al. (Zhang et al., 2018) and Yu et al. (Yu et al., 2019) obtained
231 the porosity by the average pores area of about 20 slices of 2D CT images divided by the
232 slice area and no significant difference were found comparing to the porosity from 3D
233 images. The 3D connected porosity was logically lower than the actual porosity obtained
234 by the displacement of water test (Zhang et al., 2018). The porosity had a higher value at
235 the center of the concrete than at the top and bottom and the pore size increased along the
236 porosity (Zhang et al., 2018).



237
238 Figure 2 Porosity of concrete sample. CT slice image (left), the largest spherical
239 (center) and non-spherical pores (right). The colors in the middle and right images
240 represented the different diameters of pores, scale bar, 0.15 mm (du Plessis and Boshoff,
241 2019).

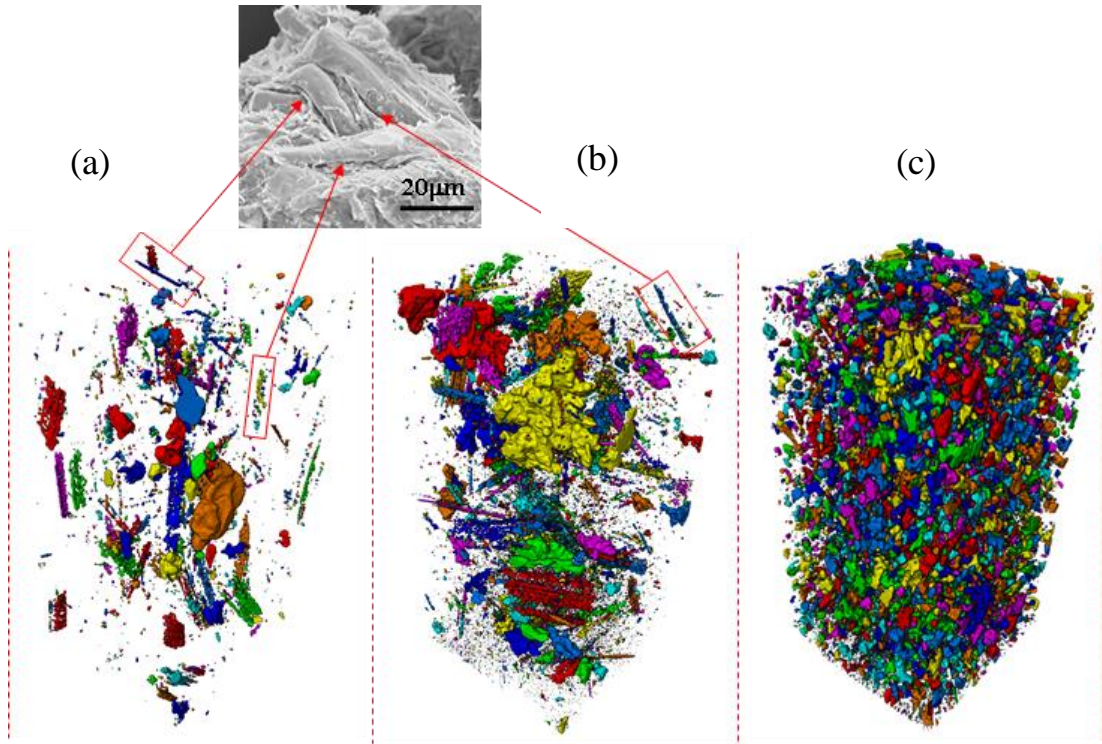
242 Ríos et al. (Ríos et al., 2019) reported a reduced porosity and an increased pore size due to
243 the addition of fibers and they could also be affected by the characteristics of fibers (Figure

244 3). Additionally, the average sphericity which was defined as $\pi^{1/3} \times (6V_{\text{pore}})^{2/3} / A_{\text{pore}}$,
 245 where V_{pore} and A_{pore} represents the pore volume and surface area respectively, reduced
 246 with the presence of fibers. The shape of pores showed a tendency from spherical to oval
 247 with fibers due to the distortion of the matrix (Ríos et al., 2019). Simões et al (Simões et
 248 al., 2017) specifically compared the concrete containing three different fibers of
 249 polypropylene, glass, and steel with dosages of 0.5%, 1%, 1.5%, and 2%, respectively. The
 250 porosity of the concrete containing the polypropylene fibers increased over the fibers
 251 content, while it showed a slight decrease in the case of glass. In the case of steel fibers,
 252 the porosity had a slightly decreased along the content up to 1.5%, however, it increased at
 253 the content of 2%. Thus, the porosity of fiber-reinforced concrete needs to be observed
 254 specifically and no general conclusion can be conducted. Additionally, the concrete
 255 manufacturing method also affects the porosity. In addition, the porous properties including
 256 pore size, porosity, and pore distribution of biodegradable composite from PLA/PEG
 257 reinforced by bamboo fibers and powders were investigated by using tomography and
 258 Avizo software for image processing, as presented in Figure 4.



259
 260 Figure 3 : Pores distribution in the concrete contains different kinds of fibers (Ríos et
 261 al., 2019). (a) no fibers, (b) micro-fibers, straight OL 13/.20, 13 mm in length and 0.20 mm

262 in diameter, (c) macro-fibers, hooked end Dramix RC80/30CP, 30 mm in length and 0.38
263 mm in diameter, and (d) mixture of 50% micro- and 50% macro-fibers. The red and gold
264 represent the pores and fibers, respectively.



265
266 Figure 4 : The pore size distribution in biobased composites with PLA/PEG matrix
267 reinforced with different fibers: (a) 30 wt% bamboo fibers, (b) 40 wt% bamboo fibers and
268 (c) 50 wt% bamboo powders (Nguyen et al., 2022b).

269 3. Influence of pore characteristics on properties of materials

270 3.1. Mechanical properties

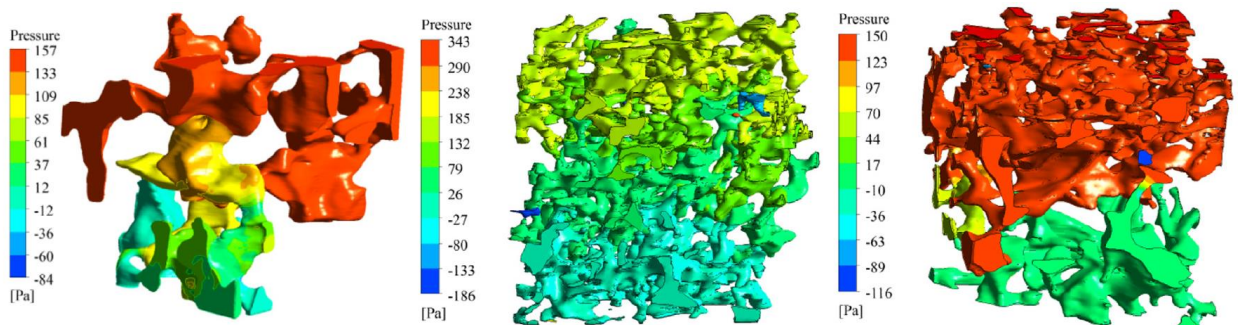
271 Porosity, pore size, pore size distribution, and the specific surface area are critical to the
272 durability and mechanical properties of concrete (Zhang et al., 2017). Compressive
273 strength is related to porosity and pore size and can be expressed as: $\sigma =$

274 $KC(1 - P)/\sqrt{r_{0.5}}$, where K is the constant of proportionality, C the cement content of
275 concrete, P the porosity, and $r_{0.5}$ the mean pore radius distribution with the unit of
276 nanometer (Das and Kondraivendhan, 2012). Not only compressive strength but also
277 tensile strength both decrease with increased porosity and pore size, yet compressive
278 strength is more dependent on porosity than tensile strength (Li et al., 2018). The increase
279 in porosity may result in a decrease in the ratio of compressive strength to indirect tensile
280 strength (Chen et al., 2013). Additionally, both compressive strength and freeze-thaw
281 durability increase if the pore size is uniform and the distance between pores is large (Liu
282 et al., 2020b). The concrete with a higher content of small pores ($<0.01 \text{ mm}^3$) had a higher
283 fatigue life, and a higher porosity may result in a lower fatigue life (Vicente et al., 2018).
284 For basalt fiber reinforced concrete, the strength and fractural energy increased along the
285 fractal dimension in the macropore region, while in the transition pore zone (diameter in
286 the range of 20 nm to 50 nm), shrinkage strain linearly related to the fractal dimension (Li
287 et al., 2020). The matrix strength of hybrid basalt-polypropylene fiber-reinforced concrete
288 affected the cumulation pore volume, porosity, and volume fraction of pores at different
289 scales (Niu et al., 2020).

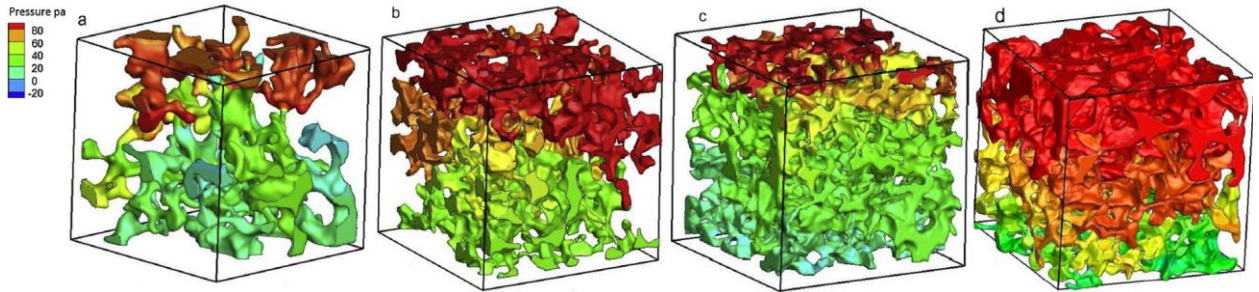
290 **3.2. Water permeability**

291 Yu et al. (Yu et al., 2019) reported that the coefficient of permeability was linearly related
292 to the ratio of total pore volume to total pore surface area. In 2D terms, the coefficient of
293 permeability increased linearly with the pore area. In terms of 3D, the permeability
294 coefficient increased along the pore volume, but its growth rate slowed down once the pore

295 volume was higher than 500 mm³. The internal pore structure could lead to different
296 permeability coefficients even with a similar porosity (Liu et al., 2020b). ANSYS FLUENT
297 for Computational Fluid Dynamics analysis (CFD) could simulate concrete pressure (Wen
298 et al., 2020). The maximum pressure was presented at the narrow pore near the entrance of
299 the seepage flow and the pressure decreased more slowly with small aggregates with a
300 small pore size (Figure 5). Zhang et al. (Zhang et al., 2018) used the triangular patches to
301 compose connected pores in 3D, imported them into ICEM CFD software to repair the
302 geometry, then imported them into another CFD software ANSYS FLUENT to simulate
303 the water seepage flow, and finally the pressure contours of seepage flow (Figure 6) and
304 the path lines (see detail in the reference) were obtained.



305
306 Figure 5 Pressure contours of seepage flow under pressure gradients of 3000 Pa/m in
307 concretes composed of different sizes of aggregate: 5-10 mm (left), 10-15 mm (center),
308 and 5-10 mm in the upper part and 10-15 mm in the lower part (right) (Wen et al., 2020).



309

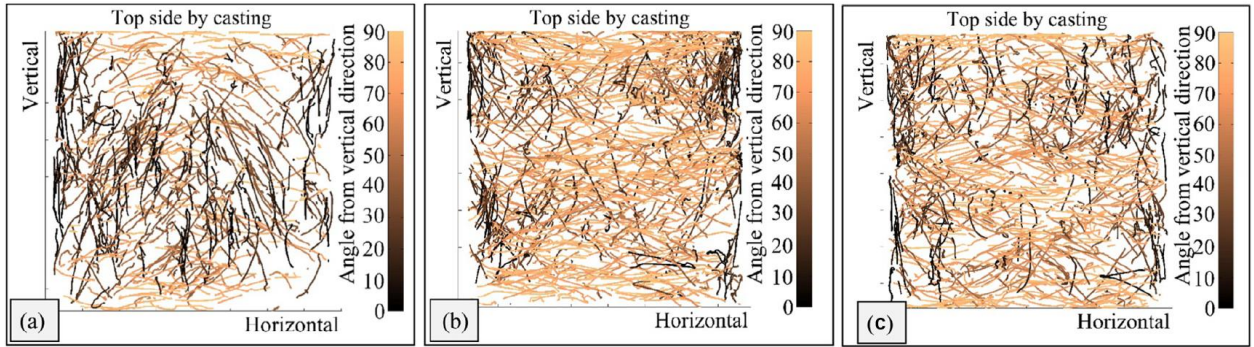
310 Figure 6 : Pressure contours of seepage flow under pressure inlet of 60 Pa in pervious
 311 concretes with different porosities (a) 13.2%, (b) 20.3%, (c) 24.8%, and (d) 28% (Zhang et
 312 al., 2018).

313 **4. Image processing techniques application**

314 **4.1. Analyses of fiber distribution in porous materials**

315 The image acquisition and processing method used in pores detection can also be used to
 316 detect the fiber distribution because the main knowledge is in common. In addition, the
 317 dynamic image analysis system (DIA) could be used to destructively detect the size and
 318 morphology of the particle or fiber (Bacaicoa et al., 2017), for instance, serials products of
 319 CAMSIZER could detect the particles in the range of 0.8 μm to 135 mm. Fibers are mainly
 320 presented as fold lines and curves in the concrete (Qin et al., 2019). Fiber distribution could
 321 be affected by many factors such as mixing time and fiber content. As shown in Figure 7,
 322 and the lowest and highest fiber densities were found in the top and bottom layers of the
 323 concrete, respectively, while a longer mixing time could improve the homogeneity of fiber
 324 distribution (Blaber et al., 2015). Fiber orientation depends on several factors, such as
 325 coarse aggregate (Qin et al., 2019), and printing process including fibers volume content,
 326 and nozzle size, but independent of print speed (Arunothayan et al., 2021). The orientation

327 of the fibers near the nozzle walls showed a higher possibility towards the printing
328 direction than in the center of the nozzle, especially in the case of large nozzles
329 (Arunothayan et al., 2021). Nguyen et al. (Nguyen et al., 2022b) reported about 50% of the
330 bamboo fibers/powders reinforced in the PLA/PEG biobased composites. The distribution
331 and orientation of bamboo fibers and powder were well observed in the composites, thanks
332 to image processing by using aviso software, as shown in Figure 8. For the spraying fiber-
333 reinforced concrete, the fibers were aligned perpendicular to the spray direction (Segura-
334 Castillo et al., 2018). Concerning hemp lime concrete, it has an oriented internal structure
335 that is determined by the direction of compacting force and the degree of orientation of
336 hemp particles depends on the production method. Some image analysis methodologies
337 have been also developed for the assessment of the frequency distributions of hemp shives
338 orientation in hemp lime concrete of three compaction levels: 30, 45 and 60% volumetric
339 decrease from the uncompact state. The results showed that the frequency distribution of
340 particle orientations in the perpendicular plane is strongly swayed indicating the
341 anisotropic material's structure. Based on a real-time observation technique, Marks et al.
342 (Marks et al., 2021) reported that a different aspect ratio of formwork could lead to
343 variations in concrete flow and end up with a different fiber orientation. In addition, the
344 orientation of fibers varied during the casting time. 3D fiber distribution obtained by the
345 different image acquisition and processing methods could be used to validate the models
346 and distribution functions (Alberti et al., 2018).



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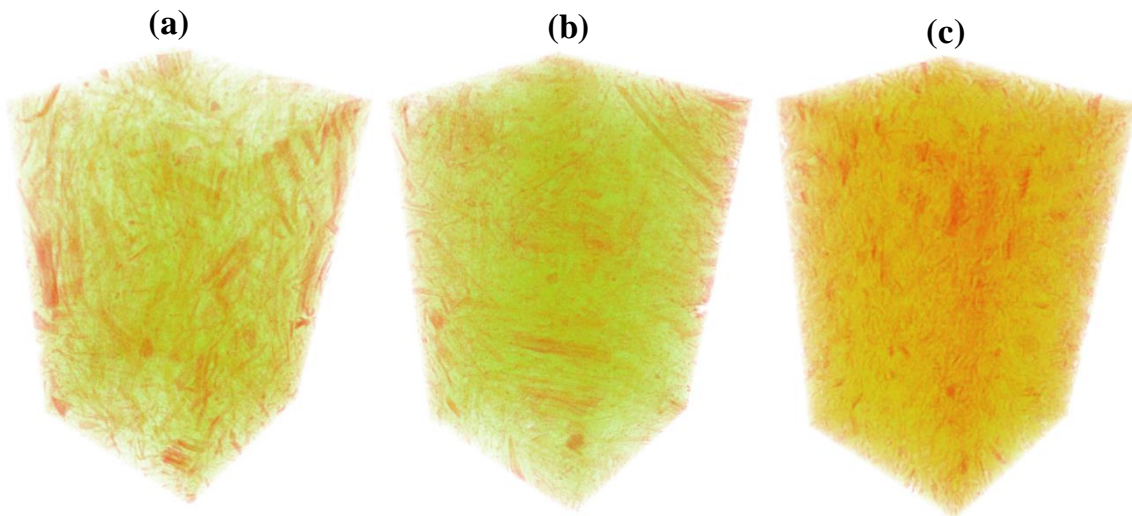
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Figure 7 The projection of fibers on the x-y plane in the concrete. (a) 0.3%(v/v) fiber content after 5 mins of mixing (b) 0.5%(v/v) fiber content after 5 mins of mixing (c) 0.5%(v/v) fiber content after 30 mins of mixing (Blaber et al., 2015).



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Figure 8 The distribution and orientation of bamboo fibers in PLA/GEG matrix composites: (a) 30 wt% fibers, (b) 40 wt% fibers and (c) 50 wt% powders. The resolution of scan was 5 μm , the images was analyzed by using Avizo software (Nguyen et al., 2022b)

366 **4.2. The correlation between fiber distribution and the mechanical properties**

367 Normally, the addition of fibers could improve the mechanical properties of concrete. The
368 addition of fibers led to an improvement of the bending tensile strength by 11.23% and of
369 the compressive strength by 11.75% for concrete (Radojičić et al., 2022). The addition of
370 2% v/w bamboo fiber could improve the mechanical strength and static modulus of
371 elasticity of concrete (Ferreira et al., 2017). The flexural strengths of concrete could be
372 improved by 13.40% by the addition of aligned carbon fibers (Hambach et al., 2016). The
373 mechanical properties of concrete are also related to the properties of fibers, such as the
374 type of fibers in relation to the splitting tensile strengths (Hong et al., 2020), compressive
375 strength (Simões et al., 2017), and cracking resistance (Lyu et al., 2021), the length of
376 fibers in relation to the flexural strengths (Pham et al., 2020a; Schleiting et al., 2020) and
377 tensile strain (Ahmed and Maalej, 2009), aspect-ratio of fibers in relation to the flexural
378 strength (Schleiting et al., 2020), the volume content of fibers in relation to the flexural
379 strengths, but not to the compressive strength (Kang et al., 2021). Besides, the compressive
380 and flexural strength of concrete can also be affected by fiber distribution, which is more
381 related to the manufacturing methods of concrete. Raju et al. (Raju et al., 2020) also
382 indicated that better fiber distribution and orientation could increase fiber content in the
383 direction of improved flexural strength. The manufacture modes, which affected the fiber
384 distribution, might affect the compressive strengths of concrete as well. The printed high-
385 performance fiber-reinforced concrete was reported had a higher compression strength than
386 the cast concrete, while no significant difference was found in the case of ultra-high-

387 performance steel reinforced concrete (Arunothayan et al., 2021). The printed concrete
388 reinforced with polyvinyl alcohol or polypropylene based fiber had a complex compressive
389 and flexural strength, and both compressive and flexural strength differed in the x and z
390 directions compared to casted specimens (Pham et al., 2021). In addition, for the sprayed
391 fiber-reinforced concrete, the load parallel to the spray direction was measured to be 3.5
392 times greater than the perpendicular load (Segura-Castillo et al., 2018).

393 It should be noted that machine learning can also be used in the field of fiber-reinforced
394 concrete. Manca et al. (Manca et al., 2018) used the machine learning algorithm to detect
395 fiber modes based on 2D images acquired by a normal digital camera. For the training,
396 4350 “snap” and “pull-out” fiber images were first labeled and then the Linear Support
397 Vector Machine model was applied to recognize those two categories with an accuracy of
398 91%. Finally, the position, failure modes, length, orientation, and amount of fibers were
399 detectable. Based on the above steps, Manca et al. (Manca et al., 2018) detected that the
400 macro synthetic fiber would snap if the fiber orientation was in the same direction as the
401 main stress. A model based on the Finite Element Method, including the input of the spatial
402 and angular orientation of fibers and the pore size distribution, was used to simulate the
403 elastic properties of polypropylene fiber-reinforced concrete and the numerical result was
404 consistent with the experiment result (Trofimov et al., 2018).

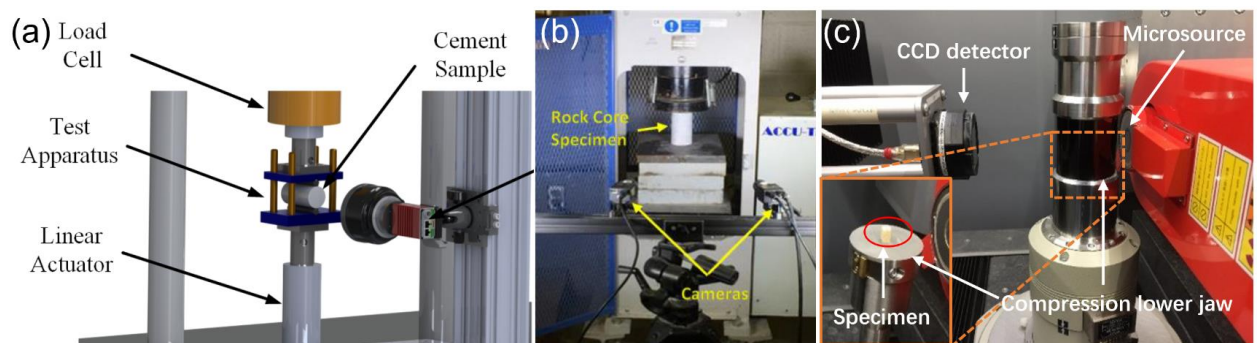
405 **4.3. DIC applied to the evaluation of surface deformation and cracks**

406 Digital Image Correlation (DIC) is a non-destructive method that is normally used to
407 measure the displacement and strains in full field. DIC can be used for both real-time

408 (Rucka et al., 2021) and delayed-time observation and it progressed and is applied in a
409 wide field. Articles searched on Web of Science with the keyword “Digital Image
410 Correlation” have increased from 1 in 1975 to 427 in 2010 and continue to increase to 1482
411 in 2020. DIC can be subdivided into 2D-DIC (based on two-dimensional images, Figure
412 9a), StereoDIC (or 3D-DIC, based on three-dimensional images from multiple camera
413 orientations, Figure 9b), and Digital Volume Correlation (DVC or VDIC, based on three-
414 dimensional tomographic images from CT or other machines, Figure 9c), therefore, some
415 pros and cons are common (Bouterf et al., 2020; Sutton and Hild, 2015). The detailed
416 history and technique of DIC can be found in (Pan, 2018). The observation of advanced
417 4D images, which consist of 3D space and time, has also developed, and attracted attention.
418 In addition, the characterization of concrete behavior under cyclic loading was studied
419 using 2D digital image correlation (Bello et al., 2023). The rapid development of
420 photographic technology, especially 3D techniques, such as X-ray imaging, Magnetic
421 Resonance Imaging (MRI), atomic force microscope (AFP), and scanning tunnel
422 microscope (STM) (Pan, 2018) and corresponding image processing and analysis method
423 are being developed and widely used in the field of mechanical analysis. Some commercial
424 software helps users perform DIC analysis, such as SeptD, VIC-2/3D, StrainMaster, GOM-
425 2/3D, and Image processing toolbox in MATLAB. In addition, some open-source software
426 such as Python-based TomoWarp2 (Tudisco et al., 2017), and MATLAB-based Ncorr
427 (Blaber et al., 2015) are widely used as well. In the field of concrete, searching “Digital
428 Image Correlation” and “Concrete” on the Web of Science, there are 1073 papers up to

429 29/02/2021, and 202 papers in 2020.

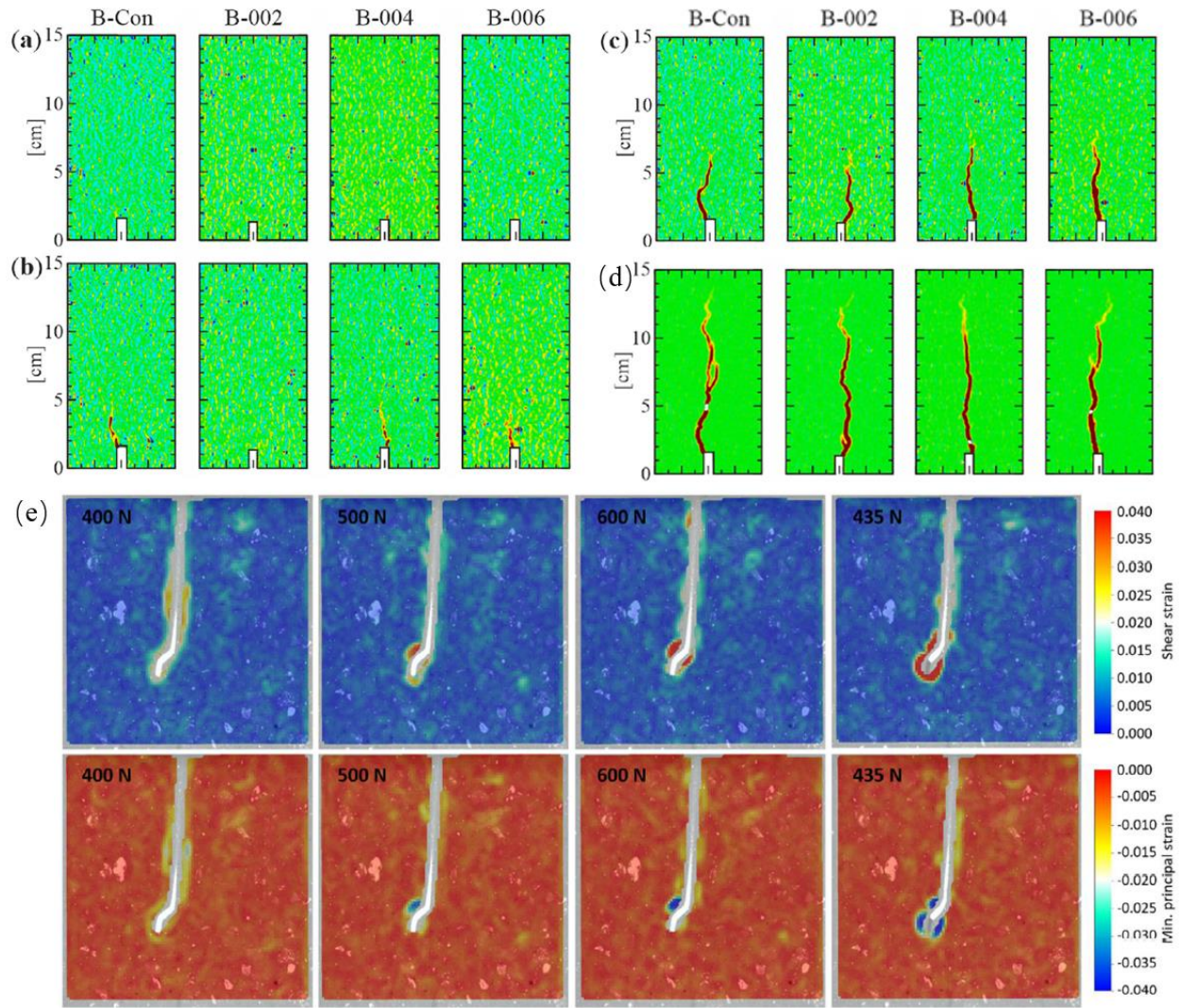
430 For example, Nguyen et al. (Nguyen et al., 2018b) obtained a series of images using the
431 LaVision StrainMaster optical system and recorded the load over time by Zwick device,
432 the DIC software of StrainMaster and VIC-2D was used to measure the full-field
433 displacement and calculated the tensile and fracture distribution. Based on the full-field
434 displacement obtained via DIC technology, Liu et al. (Liu et al., 2020a) divided the flexural
435 damage process into a microcrack initiation phase, a macrocrack evolution phase, and a
436 main crack propagation phase. DIC was also used to study the failure of the concrete with
437 different types of fiber (Melenka and Carey, 2015), detect the crack propagation in real-
438 time (Figure 10a-d) (Rucka et al., 2021), monitor the shear and principle strain of the fiber-
439 reinforced concrete (Figure 10e) (Flansbjer et al., 2018; Lakavath et al., 2019; Picazo et
440 al., 2018). A series of works and examples of DVC was presented in an update of reviews
441 (Bouterf et al., 2020; Buljac et al., 2018; Sutton and Hild, 2015). However, it remains
442 difficult to process such a large dataset in real time and keep the specimen static during the
443 load test.



444

445 Figure 9: 9 Setup of 2D-DIC (a) (Melenka and Carey, 2015), 3D-DIC (b)

446 (Abdulqader and Rizos, 2020), and DVC (c) (Nguyen et al., 2021a).



447

448 Figure 10: (a-d) 2D-DIC analysis. Cracking evolution of concrete with different content
 449 fibers at different time instances: (a) the load value equals to 90% of the maximum force
 450 (F_{max}) before reaching the peak value, (b) F_{max} , (c) 90% post- F_{max} , and (d) crack mouth
 451 opening displacement equals 0.3 mm. B-con, B-002, B-004, B-006 represent the macro
 452 polymer fiber content of 0, 2 kg/m³, 4 kg/m³ and 6 kg/m³, respectively (Rucka et al., 2021).
 453 (e) DVC analysis: shear and principle strain of the hooked fiber concrete (Flansbjer et al.,
 454 2018).

455 **4.4. Tortuosity calculation**

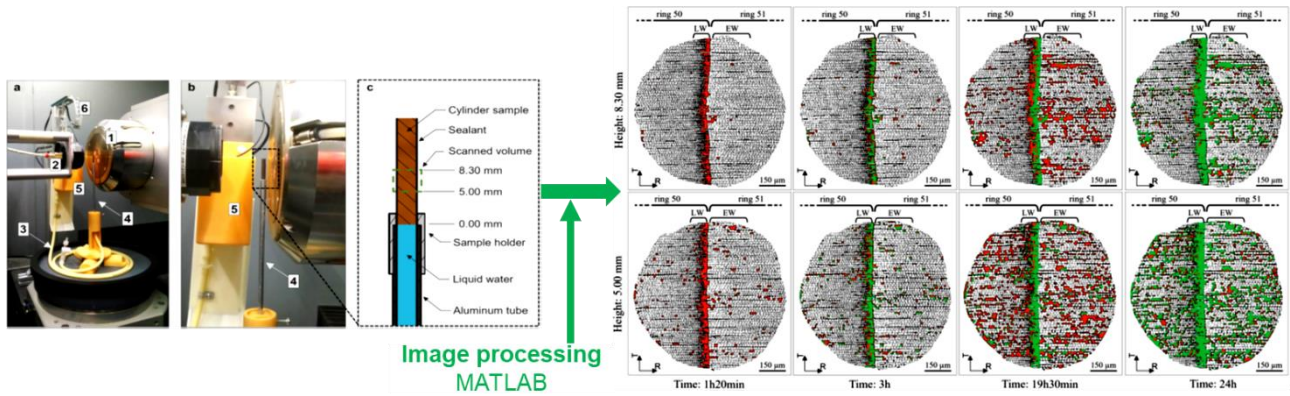
456 The tortuosity is defined as the ratio of the preferential length of fluid pathways to the
457 straight-line distance in the microstructure. Tortuosity is an important parameter that
458 significantly affects the transport behavior of fluid, molecular diffusion, heat transfer
459 within porous materials. Therefore, the mass and heat transfer inside the porous material
460 depends greatly on its tortuosity value. Accurate determination of this value allows
461 accurate assessment and prediction of multi-physics properties including acoustic
462 properties, effective diffusivity, thermal insulation, and permeability of porous materials.
463 To date, there are several approaches to determining the tortuosity of porous materials such
464 as experimental, numerical, and analytical methods (Laudone et al., 2015; Morin et al.,
465 2010; Wu et al., 2018). These methods have been applied to directly measure tortuosity
466 value or some models have been also developed to compute the tortuosity in two-
467 dimensional (2D) and even three-dimensional (3D) configurations. Therefore, a 3D pore
468 network modeling was used to calculate the tortuosity of porous media by using network
469 elements for connecting each pore to neighbors in a random direction. The tortuosity of
470 sandstone was computed by this model, and it showed a good agreement with the
471 experimental data (Nemati et al., 2020). However, these methods still have many
472 limitations in determining the tortuosity value of porous structures with irregular voids
473 sizes of the realistic porous materials. Furthermore, they could not fully describe the
474 relationship between the porous structure (void space distribution and pore connectivity)
475 and tortuosity (Zhao and Zhou, 2022).

476 The emergence of X-ray CT imaging techniques today combined with image processing
477 techniques has opened an interesting approach to porous structures materials. The
478 application of this technique was not small in adding useful approaches to the previously
479 developed techniques related to the calculation of microstructure parameters from 3D
480 images such as porosity, pore distribution, and pore sizes (Nguyen et al., 2021a; Nguyen
481 et al., 2022a). Specifically, this technique was also gradually being applied in exploring
482 the tortuosity of porous materials to explain its influence on microscopic permeability and
483 heat and mass transfer in different fields. Accordingly, the 3D pore models reconstructed
484 from 2D projections from tomography allowed to calculate pore size distribution and
485 permeability of sandstone (Zhao and Zhou, 2019). The tortuosity and pore connectivity of
486 porous alumina structures were identified according to its 3D images obtained from X-ray
487 CT (Shanti et al., 2014). The X-ray μ CT imaging techniques allowed extraction of the
488 porosity and tortuosity of different types of sandstone using a labeling segmentation
489 algorithm. It allowed to present the relation between tortuosity and pore characteristics
490 (pore size and pore shape). The finding helps to evaluate the pore-scale permeability and
491 diffusivity of geomaterials in the field of geothermal exploration (Zhao and Zhou, 2022).

492 **4.5. Liquid imbibition/drying mechanism in porous materials**

493 The advent of imaging technology and image processing tools has significantly supported
494 the morphological studies of the internal structure of materials, especially, multi-
495 component and porous structures materials. The interactions between water and wood and
496 wood-derived materials have also been specifically observed through 2D and 3D imaging

497 observations. Accordingly, water imbibition to wood in different directions was studied by
498 3D imaging technique using 2D image series obtained from high-resolution X-ray
499 tomography (Perré et al., 2022). Accordingly, the cylindrical wood sample is prepared with
500 a diameter of several centimeters. The sample was then continuously exposed to a water
501 source and placed inside the chamber of the tomography device. The penetration of water
502 into the sample was studied through continuous in-situ scanning on this device at different
503 time intervals. A series of 2D images obtained from this scan are processed through various
504 image processing techniques to analyze the process and movement of liquid water within
505 the porous structure of the wood. Accordingly, in the literature, the liquid water imbibition
506 to Norway spruce wood (softwood) was studied using X-ray tomography in longitudinal
507 direction (Martin et al., 2022). The sample preparation and experimental design were
508 described in Figure 11. A series of 2D projections were obtained from the scanning of the
509 device which were then processed with MATLAB to observe the mechanism of water
510 permeation along the pathways inside the microstructure of the wood in different directions
511 (radial tangential and longitudinal). The result has contributed to explain the mechanism
512 of liquid water transmission inside earlywood and latewood of Norway spruce wood
513 (softwood). Accordingly, the study concluded that several different fluid transfer
514 mechanisms were observed inside Norway spruce wood for earlywood and latewood
515 regions, respectively. However, this study did not consider the movement of the measuring
516 point linked to the swelling and shrinkage of the wood samples.

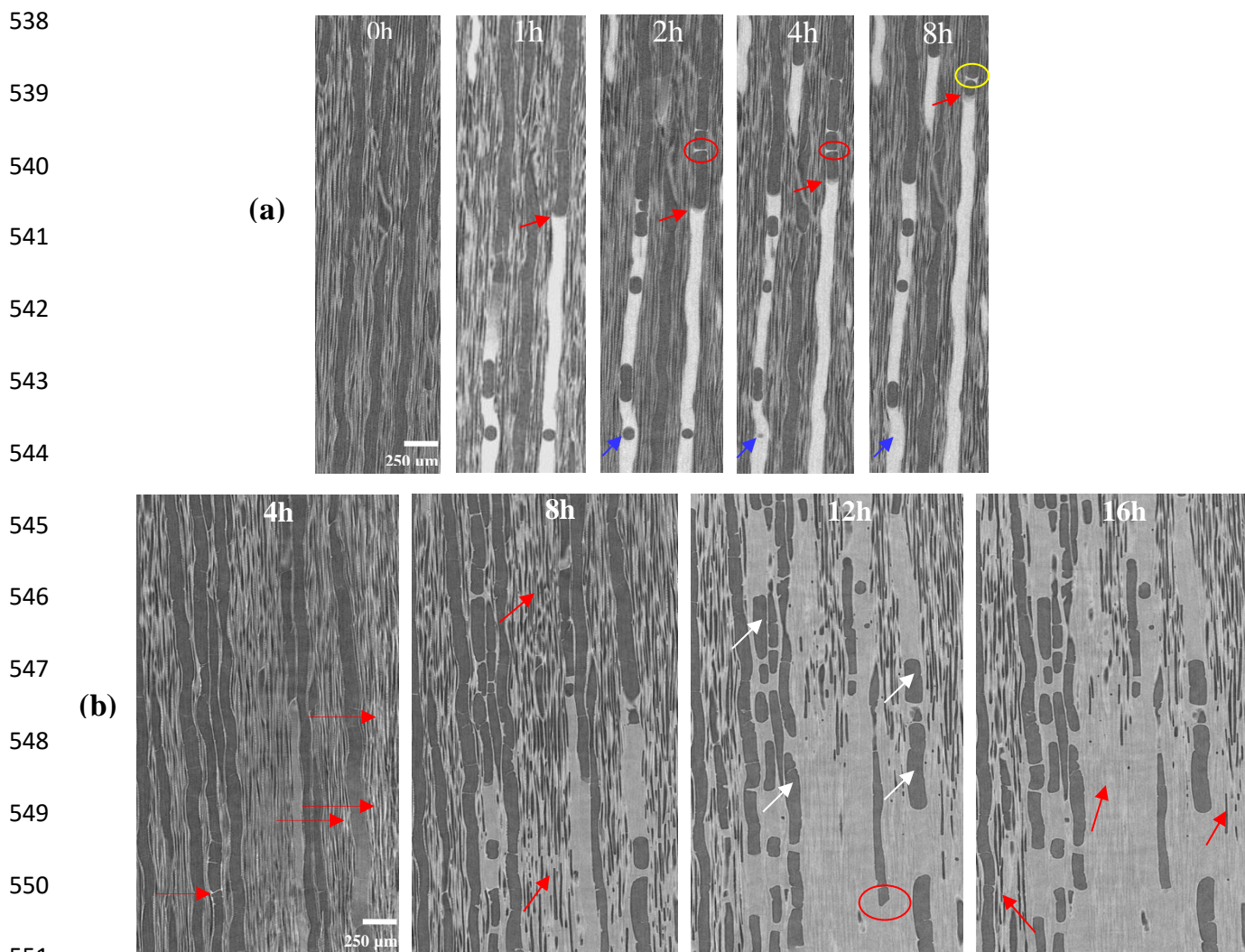


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518 Figure 11: Water imbibition in Norway spruce wood: (a) experimental setup in the
 519 tomography with nano X-ray source; (b) wood specimen placed between the nano X-ray
 520 source and CCD camera; (c) schema of the experimental setup and 3D scans volume and
 521 position; and (d) cross-section of the wood surface during imbibition at different time
 522 (Martin et al., 2022).

523 The mechanism of fluid transfer (water and oil) in hardwood and softwood was also
 524 performed using X-ray tomography combined with Avizo image analysis technique (Perré
 525 et al., 2022). The observed results added significantly to the comparison and explanation
 526 of the fluid transport mechanism of different liquids (polar and non-polar) within the
 527 complex porous structure of two different woods (poplar and spruce). Accordingly, spruce
 528 and poplar were prepared in cylinders with a diameter of about 3-4 cm. The samples were
 529 then exposed to the liquid and scanned at different times using a tomography device with
 530 micro-source and CCD camera at 3.6 μm resolution. The samples were reconstructed in
 531 3D through 2D projections through AVIZO image processing software. The images were
 532 processed using ImageJ. The results showed that there is a great difference in the

533 mechanism of water and oil penetration into the wood. Accordingly, the oil penetrates the
534 wood at a very fast rate and spreads through the gaps inside the wood under the action of
535 capillary force. Meanwhile, water penetrates the wood very slowly and penetrates the cell
536 walls of the wood before being present in the voids inside the wood, as presented in Figure
537 12.



552 Figure 12: The 3D slide observation of liquid imbibition in poplar using tomography
553 at a resolution of 3.6 μm, the images were processed using ImageJ: (a) oil imbibition

554 and (b) water imbibition (Perré et al., 2022).

555 Thus, studying the mechanism of liquid imbibition into the wood through imaging
556 techniques (2D or 3D) has contributed significantly to understanding the mechanism and
557 behavior of liquid transport into the internal microstructure of different wood. The time, it
558 takes to reach saturation and the structural change of the wood under the impact of water,
559 provides early warning of potential consequences occurring in actual buildings. However,
560 the limitation of the imaging technique does not allow to quantify the amount of water
561 absorbed into the wood accurately because the contrast of the water and the fibers of the
562 wood could not be clearly distinguished during the image processing. In addition, it is
563 necessary to consider in the analysis the coupling between the diffusion of the liquid and
564 the deformation of the pores. Furthermore, the limitation of the technique is not being able
565 to detect signs of water inside the wood makes it impossible to accurately quantify the
566 water content. Moreover, current experimental designs face many difficulties in integrating
567 measurement methods in image acquisition experiments such as in-situ experiments on
568 tomography equipment or synchrotrons.

569 The combination of image analysis techniques with the quantification method allowed to
570 measure the mass change of wood in the presence of oil and water, which allows the
571 calculation of the liquid penetration time to reach the saturation level inside the wood, as
572 shown in Figure 13. The results showed that the oil penetration was actually very fast and
573 quickly ended with its presence on the top of the sample. At this point, the penetration of

574 oil seemed to slow down and ended through the saturation curve (plateau) and the amount
575 of oil entering the wood was very small because the oil only penetrated the interconnected
576 spaces inside the sample, not the cell wall. Meanwhile, the kinetics of water imbibition into
577 wood was very slow because water penetrated the cell wall of the wood before being
578 present in the voids of the wood. Therefore, it took a very long time to achieve saturation
579 and the amount of water entering the wood was also considerable.

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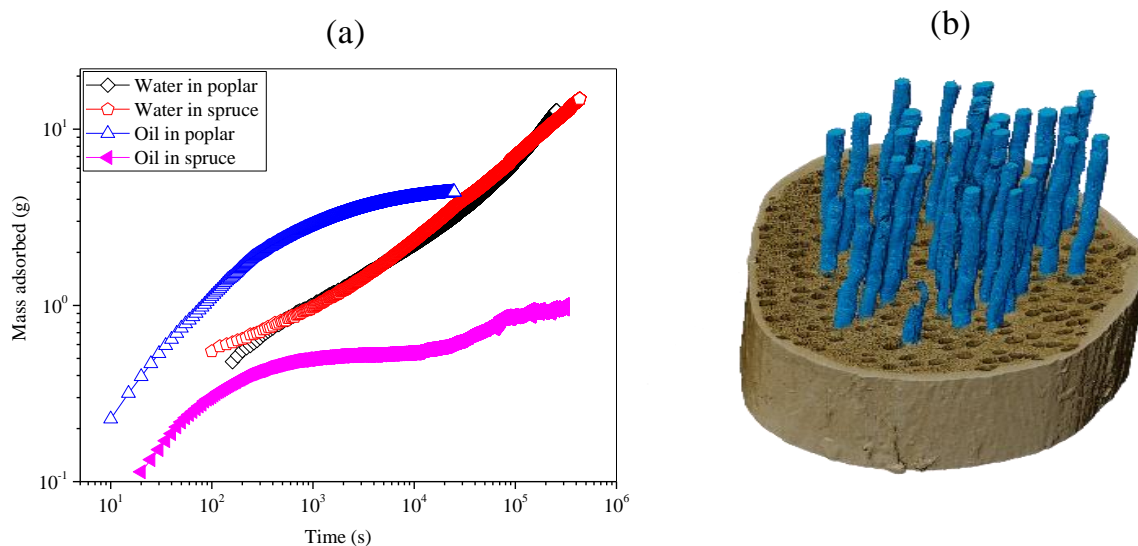
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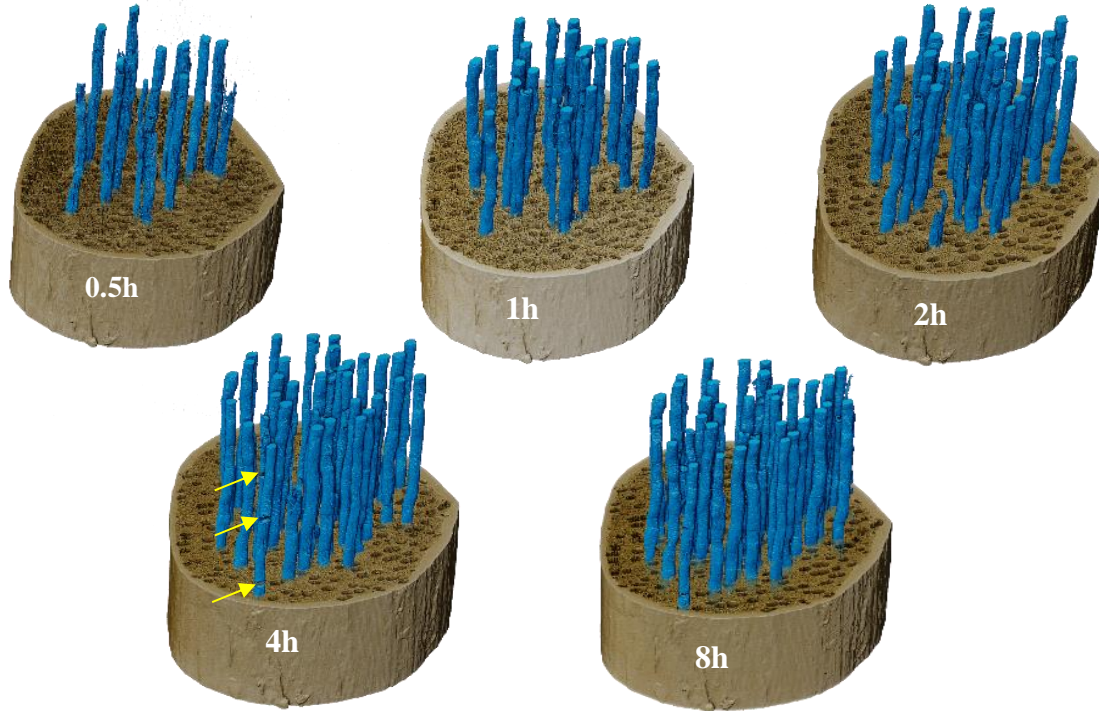


588 Figure 13: The dynamics of water and silicone oil imbibition in poplar and spruce: (a)
589 evolutions of mass absorbed of the liquid into the wood with time and (b) a miniature scale
590 in 3D volume observation of the internal structure of poplar after 2 h of silicone oil
591 imbibition test (Perré et al., 2022).

592 Furthermore, the amount of water and oil absorbed inside the poplar by experimental time
593 was also quantified by NMR method (Nguyen et al., 2020). Compared to X-ray
594 tomography, this method also allowed quantification of the free water content present in

595 the sample based on its T_2 relaxation. The water bounding to the cell wall of the wood was
596 calculated based on the swelling coefficient of the wood (Zhou et al., 2018). Thus, the
597 combination of quantitative methods and imaging techniques allows a profound
598 understanding of the nature and mechanism of water absorption into the wood. It also
599 allowed to explain the difference in the mechanism of water penetration of different wood
600 species (Nguyen et al., 2020; Zhou et al., 2018). Up to now, this is the technique that can
601 most accurately quantify the volume of liquid penetrating and moving inside porous
602 materials. This technique also allows the observation of 2D images at different positions
603 of the sample as well as different directions of the porous materials.

604 Finally, one of the interesting applications of current imaging techniques is to allow the
605 observation of fluid transmission in porous materials in 4D (3D observations with time).
606 Accordingly, the penetration and migration of the oil and water in spruce and poplar have
607 been studied in 4D observation, as shown in Figure 14 (Perré et al., 2022).



608

609 Figure 14: The 3D volumetric observation of the dynamics of silicone oil imbibition in
 610 poplar samples at different times (4D) (Perré et al., 2022).

611 According to these 4D observations, the oil moves at a very rapid rate from the beginning
 612 inside the pores and voids of the wood and rapidly reaches saturation afterward whereas,
 613 at the same time, the penetration and migration of water is very slow. Furthermore,
 614 different transmission mechanisms of liquids of different natures in porous materials were
 615 also discovered in this study. Another interesting physical transport phenomenon also
 616 detected in 4D observation was the existence and migration of air bubbles inside the pores.
 617 They tend to decrease in volume and then appear to be compressed to the smallest volume
 618 (see yellow arrows in Figure 14). Until now, several imaging techniques and image

619 processing software used to study the transfer of liquids (water and oil) inside porous
 620 materials such as wood and bio-based materials were summarized in Table 1 which helps
 621 the user to choose the most suitable one.

622 Table 1: Some imaging techniques used to analyze liquid migration in porous materials

Materials	Image technique	Dimension	Image processing methods	References
Poplar and spruce	Tomography	3D	Avizo, ImageJ	(Perre et al., 2022)
Norway spruce wood	Tomography	3D	Matlab	(Martin et al., 2022)
Norway spruce	Tomography	2D	Matlab	(Martin et al., 2022)
Douglas-fir	MRI	2D	ImageJ	(Nguyen et al., 2021b)
Poplar	MRI	2D	Image J	(Zhou et al., 2018)
Hemp concrete	MRI	2D	-	(Fu et al., 2021)
Coal	Tomography	3D	Avizo	(Lu et al., 2022)

Hornbeam	Synchrotron	3D	-	(Zhou et al., 2019)
Hornbeam	MRI	2D	-	(Zhou et al., 2019)

623

624 Finally, based on observations and data obtained from experiments, the 1D and 2D
625 modeling were developed to simulate oil and water imbibition in the vessels and fibers of
626 the poplar and spruce. Four simulations were applied, with assumptions inferred from the
627 3D microstructural observations (Perré et al., 2022). Accordingly, the 3D experimental
628 observations were used to develop a simple imbibition model that satisfactorily simulates
629 macroscopic imbibition kinetics. The 1D modeling approach is dedicated to oil imbibition
630 in poplar and spruce while a 2D modeling approach is developed for water imbibition in
631 poplar and spruce.

632 **5. Conclusions**

633 This paper has provided knowledge related to the image techniques used today to study the
634 microstructure of porous materials including wood and bio-based materials from natural
635 fibers by 3D approach. A variety of imaging techniques and image processing methods
636 were analyzed, focusing on the microstructure including porosity, distribution and size of
637 pores and fibers in the porous materials. In addition, these techniques were also used to
638 explain the mechanism of penetration and movement of liquids (oil and water) into porous
639 materials, which are intended to confirm the hypotheses and explain the results obtained

640 by the simulation as well as the experimental methods in the previous works. This paper
641 also provides 4D observation of fluid movement inside porous materials, thereby allowing
642 to explain the mechanisms of transport of polar and non-polar fluids inside bio-based
643 materials. However, the main drawback of the image processing technique is the
644 quantification of components within bio-based materials as well as the mass or volume of
645 water changes in porous materials. This was due to the similar or close densities or signals
646 of the components, which made it impossible to accurately distinguish each specific
647 component, thereby causing incorrect quantification. Therefore, further studies should
648 develop advanced techniques, possibly supported by artificial intelligence (IA), to enable
649 more efficient quantification of this image processing technique. Furthermore, the
650 resolutions of the analytical methods should be increased to obtain high image quality and
651 to identify the smallest possible pore size to study more accurately of the porous properties
652 of materials. Finally, the image analytical methods should be integrated with loading,
653 temperature and humidity controllers to study their influence on the comprehensive
654 properties of porous materials.

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