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Mechanical Properties and Freezing and Thawing Behavior of 3D Printing Concrete containing Recycled Fine Aggregates from Construction and Demolition Waste

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Synopsis: This study focuses on evaluating the mechanical, microstructural, and durability properties of 3D printing mortar (3DPM), with a specific emphasis on the influence of incorporating Recycled Fine Aggregates (RFA). These RFA are produced from Construction and Demolition Waste (C&DW) in Belgium and are sieved to a maximum particle size of 2 mm [0.08 in].

Cast and printed samples of mortar containing 100% RFA, with a sand-to-cement ratio of approximately 1:1 and a water-to-cement ratio of 0.29, were subjected to mechanical tests, including flexural, compressive, and tensile strength, at 2, 7, 28, and 56 days. The possible anisotropic behavior of the printed material was also investigated. The results show that using RFA does not significantly affect the mechanical properties of the mortar, and some anisotropic behavior was observed based on the compression test results. The end-goal of the project is to print non-reinforced urban furniture; in order to assess its durability, only freezing and thawing (F-T) behavior was investigated. The F-T behavior were analyzed based on the quantity of spalling particles after 7, 14, 28, 56, and 91 F-T cycles. The results show that up to 91 F-T cycles, no significant surface damage occurred.

<u>Keywords</u>: Construction and Demolition Waste, 3D printing concrete, durability of concrete, mechanical properties, Recycling

Yeakleang Muy is a post-doctoral researcher at the University of Liège in Belgium, affiliated with the Urban and Environmental Engineering unit. Her initial post-doctoral project is the INTERREG CIRMAP project, focusing on integrating recycled aggregate into the formulation of 3D printing mortar for shape customizing of urban furniture. In 2022, she earned her Ph.D. in Civil Engineering from INSA Rennes in France, focusing on the valorization of marine dredge sediment for partial cement substitution.

INTRODUCTION

In recent years, the construction industry has witnessed the development of a new building technology driven by innovations in additive manufacturing technologies, commonly referred to as 3D printing (3DP). Three-dimensional printing is a groundbreaking technique that involves the sequential printing of layers, one on top of the other. This method was first introduced in 1986 for prototyping purposes [1]. Compared with the traditional cast-in-situ concrete construction method 3D printing concrete construction presents several advantages such as the freedom of design,

12 formwork-free fabrication, waste minimization and mass customization. [1-4].

13 3D printing concrete (3DPC) is also being explored in the framework of the transition of the construction industry 14 toward more sustainable and environmentally friendly building solutions [5, 6]. Indeed, the better material efficiency [4, 7] of this technology aligns with the global imperative to reduce the construction industry's carbon footprint and 15 16 minimize waste generation. Yet, this technology is still facing important challenges linked to the important amount of 17 cement and sand used in the process [8]. One promising avenue towards improving the environmental impact of 3DPC 18 while promoting a circular economy is the incorporation of recycled materials into 3DPC formulations. The integration 19 of Recycled Fine Aggregates (RFA) into concrete has been extensively studied due to its potential environmental 20 benefits; however, its impact on the mechanical properties of concrete, including 3D Printed Concrete (3DPC), remains 21 a topic of discussion. Several studies have highlighted the adverse effects of RFA on concrete's hardened properties, 22 attributing them to RFA's lower density and higher water absorption [9]. Similar findings have been observed in the 23 context of 3DPC, with Ding et al. reporting marginal decreases (up to 27%) in compressive strength but no clear influence on tensile and flexural strength with RFA substitution rates of up to 50% [10]. Moreover, Xiao et al. observed 24 25 significant losses in mechanical performance, particularly in compressive strength (up to 48%), when using 100% 26 recycled aggregates and sand [11]. This weakness can, in part, be addressed through fiber reinforcement, as 27 demonstrated in Xiao's research [12]. 28

29 RFA have, however, demonstrated potential in improving the buildability of 3DPC. Wu et al. and Xiao et al both 30 conducted rheological, workability and buildability tests, noting faster decrease in workability but higher yield stress 31 and faster shear modulus increase with increasing RFA substitution rate [12, 13]. These are both indicators of a better 32 buildability. This is, once again, attributed to the higher water absorption of recycled concrete aggregates but also to 33 their rougher surface compared to river sand. Similarly, Ding et al. reported accelerated early age strength development 34 with RFA incorporation of up to 50%, suggesting enhanced buildability [14]. However, these benefits may come at 35 the cost of a shortened printability window, posing challenges for large-scale applications [15]. Zou et al. proposed 36 mitigating this issue by adding sodium gluconate to printing mortar containing recycled sand, extending the printability 37 window while maintaining higher early-age strength compared to natural sand mortar limiting the strain in the bottom layer and therefore improving the overall buildability [15]. 38

39 Despite extensive research on the mechanical aspects, the impact of RFA incorporation on the durability performance 40 of 3DPC remains understudied [16]. This paper investigates the mechanical, microstructural, and durability properties 41 of 3D printed mortar(3DPM) in which the natural sand has been substituted RFA. Specifically, in this study, natural 42 quarry sand (crushed limestone) is being replaced by recycled fine concrete aggregates produced in the Tradecowall 43 St-Ghislain recycling plant in Belgium. The materials have been characterized and mortar mixtures have been designed

44 for both natural and recycled sands.

- 45 Comparative analyses are conducted, drawing distinctions between mortar samples containing RFA and those with
- 46 natural sand. The investigation evaluates mechanical performance through flexural, compressive, and tensile strength
- 47 tests. The changes in microstructure are assessed based on porosity, bulk density, and capillary absorption. The
- 48 durability of the material is investigated via Freezing and Thawing (F-T) cycles resistance which is a crucial parameter

49 for mortars and concrete-containing recycled materials. There is plenty of research which have been conducted on the F-T behavior of concrete [17-22] and mortar but for mortar, that is mostly for the masonry aspect. 50

51 The influence of the printing process itself is also studied and highlighted anisotropic behavior in printed mortar samples. Furthermore, the impact of curing methods on mortar properties is investigated because of the important 52

53 amount of cement involved in 3DPC compositions.

54

MATERIALS AND METHODS

55 Materials

56 In this research, the fine aggregates obtained from the recycling of all-mixture concrete, denoted as recycled sand (RS),

originate from the Tradecowall recycling center in St-Ghislain, Belgium. To ensure a ratio of at least 10 between the 57

58 diameter of the printing nozzle and the largest grain size [19], the maximum grain size is restricted to 2 mm [0.08 in]. Recycled sand possesses a density of 2.39 tons/m³ [149.21 lb/ft³] and exhibits a water absorption rate of 5.31%. 59

60 The virgin aggregates employed as reference material, designated as natural sand (NS) within this research, were

sourced from the Saint Bonnet quarry in France. The particle size distribution of the natural sand closely resembles 61

62 that of the recycled sand, as illustrated in (Fig.1). This natural sand has a density of 2.62 tons/m³ [163.56 lb/ft³] and a

water absorption capacity of 0.65%. 63

64 The portland cement employed is categorized as type CEMI 52.5N, procured from VICAT's manufacturing facility in

65 Créchy, France. It functions as a hydraulic binder and possesses a density of 3.16 tons/m³ [197.27 lb/ft³].

The plasticizer Polycarboxylate (PCE) and the viscosity modifying admixture (VMA) used in this research were 66

supplied by the company Chryso. These components play a crucial role in enhancing the workability of mortar, which 67

in turn improves printability factors such as pumpability and extrudability, ultimately resulting in enhanced buildability 68

69 (retaining capacity).

70 Mixture proportion and sample implementation

71 The mixtures proportions of the mortar used in the study are presented in Table 1. Two mortar formulations were

72 employed for the tests: one featuring natural sand (NSM) and the other incorporating recycled sand (RSM). In the case

73 of RSM, all the natural sand was substituted with recycled sand by mass. Additionally, the mixing water was adjusted

74 to accommodate the higher water absorption of recycled sand, ensuring that the effective W_{eff}/C remained constant. In addition, both mortar NSM and RSM have similar workabilities based on the measurement of flow diameter following

75

76 NBN EN 1015-3, which is equal to 125 mm [4.92 in] and 128 mm [5.04 in] respectively.

77 Cast samples

78 For laboratory fabricated cast samples, the mixing process begins with adding sand and water to the container, allowing

79 them to pre-saturate for a duration of 300 seconds (no saturation step is required for the mixture with natural sand).

80 Subsequently, cement, viscosity modifying agent, and superplasticizer are incorporated, and the mixture is blended for

30 seconds at low speed, followed by an additional 30 seconds at high speed. Following a brief 30 seconds pause, any 81

82 mortar adhering to the container's walls is repositioned to the center during the mixing process. The sequence concludes 83 by subjecting the mixture to high-speed mixing for 150 seconds. The implementation of these samples adhered to the

84 guidelines specified in NBN EN 196-1 [20].

85 **Printed** samples

86 For printed samples, the cartesian printer shown in the Fig. 2(a) was used. The mixing procedure begins with mixing

87 the first third of the materials, including sand, cement, and water, for a period of 2 min. This is followed by the inclusion

88 of the second portion, in which the admixtures are added to the water, which is similarly mixed for 2 min. Lastly, the

- 89 third portion is introduced and blended for an additional 11 min. Only the RSM formulation was utilized for the printing
- of elements. Specifically, "S"-shaped elements were continuously printed up to 6 layers as seen in Fig. 2(b). 90

91 Additionally, another "S"-shaped element, with the string distance adjusted to create a slab, was printed for subsequent 92 freezing and thawing testing. After printing, these elements were covered with a plastic film and left for 48 h prior to 93 controlled curing in a humid chamber (maintained at a relative humidity of $95 \pm 5\%$ and a temperature of $20 \pm 2^{\circ}C$ [68 94 \pm 33.8°F]). The nomenclature RSM C is employed for cast samples, while RSM P is used for printed samples.

- 95

EXPERIMENTAL METHODS

96 Flow table test

97 The flow table test was conducted to access the workability of fresh mortar. This test was performed following the 98 standard NBN EN 1015-3 and the flow diameter of both NSM and RSM mortar was measured.

99 Flexural and compressive strength test

100 Flexural and compressive strength evaluations were executed following the protocols outlined in the established standard NBN EN 196-1 [20]. Test prisms with dimensions of $40 \times 40 \times 160$ mm³ [1.57 × 1.57 × 6.30 in³] were 101 102 prepared and tested at intervals 2, 7, 28, 56 and 91 days.

103 For cast samples, they were manufactured and subsequently encased under plastic film for a 24 h period before demolding. Two groups of samples were created to assess the impact of curing conditions on the mortar. One group 104 was cured underwater in a room with a temperature of $20 \pm 3^{\circ}$ C [68 $\pm 5.4^{\circ}$ F] and a relative humidity exceeding 95% 105 until the specified time. The second group underwent dry curing in a room with a temperature of $20 \pm 3^{\circ}$ C [$68 \pm 5.4^{\circ}$ F] 106 107 and a relative humidity of $60 \pm 5\%$.

108 On the other hand, printed samples were extracted from the printed "S" element at each designated time and underwent

109 testing from two distinct loading directions: parallel (oz) and perpendicular to the printing direction (ox), as shown in

110 Fig. 3(b). The flexural test applied the load at a rate of 3 kN/min, while the compression test employed a loading rate

of 144 kN/min. The flexural strength (R_i) and compressive strength (R_c) were then calculated based on the recorded 111

failure load F_f and F, respectively, as shown in Eq. 1 and Eq. 2. 112

$$R_f = \frac{1.5 \times F_f \times l}{b^3}$$
 Eq. 1

$$R_c = \frac{F_c}{1600}$$
 Eq. 2

113 Where b is the side of the square section of the prism (mm), F_f is the maximum load until failure, applied to the middle

114 of the prism (N), l is the distance between the support (mm), F_c is the maximum load until failure, applied to the surface

of 40×40 mm² [1.57 × 1.57 in²] of two half prisms resulted from flexural test (N), and 1600 is the area of the platents 115

or auxiliary plates $40 \times 40 \text{ mm}^2 [1.57 \times 1.57 \text{ in}^2] (\text{mm}^2)$. 116

117 **Tensile strength test**

118 Tensile strength was evaluated following the guidelines outlined in the established standard NBN B15-211 [22]. 119 Cylindrical specimens, measuring 50 mm [1.97 in] in diameter and 50 mm [1.97 in] in height, were prepared and tested

120 at intervals of 7, 28, 56 and 91 days for cast samples and exclusively at 91 days for printed samples. The cast samples

were drilled from cubic specimens of dimensions of $150 \times 150 \times 150$ cm³ [59.06 × 59.06 × 59.06 in³]. On the other 121

hand, the printed samples were drilled perpendicularly to the printed layers from the printed "S" shape element. The 122

outline of the layers was then traced approximately based on the visible interface on the printed element, as depicted 123

124 in Fig. 4(a). The experimental procedure was executed using INSTRON instrument as shown in Fig. 4(b) with a pulling

125 rate set at 0.10 ± 0.05 MPa/s [14.50 \pm 7.25 psi/s]. The tensile strength (R_t) was also calculated based on its failure load

126 F_t, as shown in Eq. 3.

$$R_t = \frac{F_t}{S}$$
 Eq. 3

127 Where F_t is the maximum load using until the rupture of mortar (N), and S is the section of the fracture surface (mm²).

128 **Porosity and bulk density measurements**

Porosity ε and bulk density ρ_d of the mortar were evaluated in accordance with standard NF P18-459 [11]. Cube-shaped 129 130 samples, measuring 40 x 40 x 40 mm³ $[1.57 \times 1.57 \times 1.57 \text{ in}^3]$, were employed for these measurements and for both 131 cases of cast and printed samples. The assessment was conducted at specific time intervals: 2, 7, 28, 56, and 91 days. 132 The procedure involved placing the samples under vacuum conditions for a duration of 4 ± 0.5 h, followed by saturation under vacuum for an additional 44 ± 1 h. After saturation, the test samples were gently dried by removing excess 133 134 surface water with a damp cloth and then weighed (m_1) . Subsequently, hydrostatic weighing was performed (m_2) and finally, all the samples were placed in an oven and dried at 60° C [140°F] until a constant mass was attained (m_3). The 135 136 formulas to calculate the mortar's porosity and bulk density are provided in Eq. 4, and Eq. 5 respectively.

$$\varepsilon = \frac{m_1 - m_3}{m_1 - m_2} \times 100$$
 Eq. 4

$$\rho_d = \frac{m_3}{m_1 - m_2} \times \rho_w$$
 Eq. 5

137 Where ρ_w is the water density (kg/m³)

138 Capillary absorption measurement

139 The capillary water absorption assessment was executed utilizing the suction method adapted from the established 140 standard NBN EN 13057 [24]. Prism samples with dimensions of $40 \times 40 \times 160$ mm³ [$1.57 \times 1.57 \times 6.30$ in³] were employed for this examination, whether they originated from cast or printed samples. Following a 28-day curing 141 142 period, all specimens underwent a 1 cm [0.39 in] cut on the testing surface to ensure consistent surface conditions. They were, then, dried in an oven set at $40 \pm 2^{\circ}$ C [104 $\pm 3.6^{\circ}$ F] for 7 days, and the weight stability was ensure by 143 144 verifying a mass loss of less than 0.2% after two successive weighings with a 2-hour interval. According to the 145 specifications of the standard NBN EN 13057 [24], the tested specimens were submerged in a tank containing 146 demineralized water, with the tested surface immersed to a depth of $2 \pm 1 \text{ mm} [0.08 \pm 0.04 \text{ in}]$, facilitated by a small 147 pins holder. The tank was sealed with a lid. The amount of water absorbed by the samples was determined through 148 successive weighing at various time intervals: 12 min, 30 min, 1 h, 2 h, 4 h, 5 h, and 24 h. The water uptake per unit 149 area, i, was calculated for each time increment from the absorbed weight of water divided by surface area of the of the 150 specimen in contact with the water. However, it is important to note that this method does not ensure unidirectional 151 absorption. To verify this condition, two sets of samples were investigated: one set remained untreated while the other 152 wascoated with epoxy resin. The absorption was also evaluated in two directions: (oz) and (ox) for both cast and 153 printed specimens. In this case, for cast specimens, (oz) direction refers to the bottom and (ox) direction refers to the 154 side surface of specimens.

155 Freezing and thawing test

The freezing and thawing test adhered to the standard RNR 50-1 [25], which is based on CEN/TS 12390-9 [26]. For this experiment, cylindrical specimens were prepared, featuring a diameter of 113 mm [4.45 in] and a height of 50 ± 2 mm [1.97 in ± 0.08 in]. In the case of cast samples, the specimens with dimensions of 150 x 150 x 150 mm³ [5.91 ×

 $5.91 \times 5.91 \text{ in}^3$ were initially cured for 28 days before drilling to obtain two cylindrical specimens from each cube.

160 For the printed samples, the printed slab also underwent 28 days of curing before drilling to obtain the desired

165 91 freezing and thawing cycles.

166 The evaluation of freezing and thawing resistance was carried out under three distinct surface conditions, with four 167 samples for each condition:

- 168 No solution (NS) - Only the surface was saturated.
- Water solution (WS) The surface was covered with 3 mm [1.19 in] of demineralized water. 169 •
- 170 • Saline solution (SS) - The surface was covered with 3 mm [1.19 in] of saline solution (3% NaCl).

171 These surface conditions were chosen to simulate the different levels of severity in the environmental conditions which 172 showing the most severe conditions exposed to the freezing and thawing phenomenon.

173

RESULTS AND DISCUSSION

174 Effect of sand replacement and printing method on mechanical and microstructural properties

175 The results regarding the flexural and compressive strengths of cast mortar samples containing recycled sand (RSM_C)

176 and natural sand (NSM_C) are visually represented in Fig. 6. In Fig. 6(a), it can be observed that the flexural strength

of RSM C varied from 7.6 to 10.5 MPa [1102.29 to 1522.896 psi], while NSM C exhibited a range of 9.3 to 12.9 177

178 MPa [1348.85 to 1870.987 psi]. In general, the flexural strength of RSM C reached between 63.1% and 79.8% of that 179 of NSM C, except for the 7-day result. Importantly, statistical analysis using Student's t-distribution did not reveal any

180 significant differences in these results.

181 Conversely, the findings related to compressive strength indicated a slight decrease in the strength of RSM C 182 compared to that of NSM C when the water content was kept constant, as depicted in Fig. 6(b). As expected, the 183 compressive strength showed an overall increase with time. The strength values ranged from 52.5 to 85.1 MPa [7614.48

to 12342.71 psi] for RSM C and from 61.3 to 89.9 MPa [8890.81 to 13038.89 psi] for NSM C, respectively. Notably, 184

the compressive strength of RSM_C reached approximately 85.7% to 101% of that of NSM_C, with the 28-day result 185

reaching 101%. 186

187 Fig. 7 illustrates the findings regarding the capillary absorption of cast specimens RSM C and NSM C. These results

confirm that the change in sand type does not have a significant impact on the capillary system. Specifically, at 24 h 188

of absorption, all mortar samples absorbed a mass of water ranging from 0.65 to 0.75 kg/m² [0.13 to 0.15 lb/ft²], as 189

- shown in Fig. 7(a). The coefficient of absorption for the mortar was analyzed through linear regression, yielding values 190
- ranging from 0.168 to 0.191 kg/m².h^{0.5} [0.03 to 0.04 lb/ft².h^{0.5}], with R^2 values falling between 0.845 and 0.854, as 191 illustrated in Fig. 7(b). Notably, the RSM C specimens appear to exhibit greater homogeneity than the NSM C
- 192
- 193 specimens, as their coefficient of absorption values are closely clustered in all absorption directions.

194 Effect of curing method on development of mechanical and microstructural properties

195 Fig. 8 presents the flexural and compressive strength of RSM C under two different curing conditions such as water 196 curing and ambient (at 60% RH) curing.

197 In general, it appears that the curing method does not have a significant impact on flexural strength. However, there is

198 a noticeable drop in flexural strength at 2 days, which can be attributed to the loss of water necessary for the hydration

process. After 7 days of curing, the positive influence of water curing on hydration reactions becomes evident, as 199

200 depicted in Fig. 8(a). In contrast, when it comes to compressive strength, ambient curing results in a significant decrease in strength, even at later stages, as shown in Fig. 8(b). This decline is likely attributed to the early loss of water, which promotes the development of shrinkage and micro-cracks. This distinction is particularly prominent in the case of the studied cementitious ink due to the substantial amount of cement in its formulation.

When comparing the compressive strength of mortar RSM_C cured in ambient conditions to the water-cured mortar described in the previous section, the compressive strength of the mortar cured in an ambient environment reached only 60.3% of those cured in water at 28 days, with almost no significant evolution observed beyond 7 days of curing due to a poor hydration reaction.

209 Anisotropy of compressive strength

210 Fig. 9 presents the results of compressive strength tests conducted on printed mortars in two loading directions, namely

211 RSM_P (oz) and RSM_P (ox), in comparison to cast mortars RSM_C. In general, the compressive strengths of the

212 printed samples are lower than those of the cast samples, primarily due to differences in the processing methods. For

213 instance, at the 28-day mark, the compressive strength of the printed mortar differs from that of the cast mortar by

approximately 35% for the RSM_P (oz) orientation and approximately 16% for the RSM_P (ox) orientation.

215 Moreover, regarding the anisotropy of compressive strength, the results also indicate that printed mortars exhibit

greater strength in the (ox) orientation. This observation aligns with prior research by various authors [21], [27], [28],

as cited in the review by Rehman and Kim [129]. This phenomenon can be attributed to a less dense zone outside the

218 compression region of RSM_P (oz) samples compared to that of RSM_P (ox) samples. This less dense zone is created

219 during the printing because the layer being printed is "pushed" in the previous layer to widen them to obtain layers

that are at least 6 cm [2.36 in] wide while printing with a 2 cm [0.79 in] nozzle.

221 Bond strength properties of 3D printing mortar

The Table 2 presents the tensile strength values and the corresponding failure patterns for the printed specimens RSM_P. The average tensile strength falls within the range of 2.03 to 2.69 MPa [294.43 to 390.15 psi]. Notably, the observed failures did not occur between layers; instead, they seemed to manifest randomly, as depicted in both Table 2 and Fig. 10. This observation suggests the potential homogeneity of the mixture, possibly attributable to the layer compression applied during the printing process. Additionally, it's worth noting that the mixture composition did not include a setting time accelerator, which might have contributed to these outcomes.

228 These findings align with the results obtained from the compression tests. If the material had interfaces or weak planes

between the layers, one would expect a lower compressive strength in the RSM_P (ox) direction compared to the other direction, influenced by the Poisson's ratio. However, this was not observed, further supporting the conclusion of

231 material homogeneity.

232 Effect of printing method on microstructural properties of mortar

The Fig. 11(a) and Fig. 11(b) highlight the porosity and bulk density of cast and printed mortars at various time periods. In general, the porosity of the mortar ranged between 17.71% and 21.07%, with a decrease in porosity observed over time. For RSM_C mortar, the porosity decreased by 0.39% from 21.07% after 2 days of curing to 20.68% after 56 days of curing. In the case of NSM_C mortar, the porosity decreased by 1.66% from 19.37% after 2 days of curing to 17.71% after 56 days of curing.

However, it's worth noting that the bulk density of NSM_C mortar is significantly higher than that of both the recycled sand mortar, RSM_C, and RSM_P. As expected, the porosity of NSM_C is also lower than that of RSM_C. The printing process tends to create a denser microstructure in the mortar compared to conventional casting. The results of

capillary absorption, as shown in Fig.12, also confirm this observation, with RSM_P mortar exhibiting the lowest

242 water absorption.

243 Effect of tested surface size and coating preparation on the capillary absorption of mortar

Fig. 13 illustrates the impact of epoxy resin covering on the capillary absorption properties of cast mortar. The results make it evident that the smaller the absorption surface, the more significant the effect of the coating. For instance, the non-coated sample RSM_C, exhibits much higher water absorption in the (oy) direction, where the contact surface is 1600 mm² [2.48 in²] than in the other two directions. However, after coating, the water absorption becomes similar in all directions. In the case of samples with a contact surface of 6400 mm² [9.92 in²], a slight difference is observed between coated and non-coated samples in all absorption directions. These findings emphasize the importance of

250 selecting the appropriate surface size for testing the absorption of printed specimens.

251 Freezing and thawing behavior of mixture proportions

Fig. 14 shows the outcomes of the freezing and thawing test following exposure to 91 freezing and thawing cycles under three distinct surface conditions. Overall, the results indicate that the specimens' surfaces experienced minimal damage after undergoing 91 freezing and thawing cycles, whether the mortar was cast or printed. The cumulative spalling particles after 91 freezing and thawing cycles were found to be less than 1 g/m² [0.0002 lb/ft²]. This resilience can be attributed to the favorable matrix of the mortar, which inherently contains micro-pores that are less susceptible to frost-related damage. A similar outcome was observed by Algourdin et al. [30], where no significant surface damage was detected in recycled sand-based mortar, even after subjecting it to 96 freezing and thawing cycles.

Furthermore, the results reveal that different saturation surface conditions had varying effects on the freezing and thawing test. Using a salt solution was found to create the most severe environment for the freezing and thawing test, followed by using a water solution on the surface. The least severe condition was observed when no solution was applied to the surface, and only saturation was performed, as depicted in Fig. 14(a).

263

287

CONCLUSION

This comprehensive research study has shed light on several crucial aspects pertaining to 3D-printed mortar, particularly in the context of incorporating Recycled Fine Aggregates (RFA) in substitution to natural sand :

- The research showed that replacing natural sand with RFA did not significantly impact the mechanical properties of the mortar. Both mechanical performances and microstructural characteristics exhibited similar trends for mortars containing RFA and those with natural sand. This suggests that RFA can be a viable and sustainable alternative in 3D-printed mortar without compromising mechanical performances.
- An interesting finding was the anisotropic behavior observed in 3D-printed mortar samples. The compressive strength of printed mortar varied depending on the loading direction, with the perpendicular direction (RSM_P (ox)) exhibiting higher strength. Direct tensile tests showed that failures occurred randomly within the material rather than at the interfaces between printed layers. These observations challenge conventional assumptions about the existence of a weaker interface between layers of 3D-printed materials and highlights the need for further research.
- The study assessed the freezing and thawing resistance of the mortar, usually a limiting factor for concretes and mortars produced with recycled aggregates. Remarkably, both cast and printed mortars demonstrated excellent freezing and thawing resistance, with minimal surface damage even after 91 freeze-thaw cycles. This resilience is attributed to the favorable matrix of the mortar, which contains micro-pores that are less susceptible to frost-related damage.
- The curing method employed showed significant influence on the mechanical properties of the mortar.
 Ambient curing led to important decrease in mechanical performances underscoring the importance of appropriate curing conditions for 3D-printed mortar due to the important amount of cement involved.

These results show that RFA can be used in 3D-printed mortar without compromising mechanical or durability properties. The study also highlights the complexity of 3D printing processes, including anisotropic behavior and the need for careful consideration of curing methods.

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290		REFERENCES
291 292	[1]	M. D. Cohen, Y. Zhou, and W. L. Dolch, "Non-Air-Entrained High-Strength ConcreteIs it Frost Resistant?," <i>MJ</i> , vol. 89, no. 4, pp. 406–415, Jul. 1992, doi: 10.14359/2582.
293 294	[2]	G. Fagerlund, "FROST RESISTANCE OF HIGH PERFORMANCE CONCRETE- SOME THEORETICAL CONSIDERATIONS," 1994.
295 296	[3]	R. Zaharieva, F. Buyle-Bodin, and E. Wirquin, "Frost resistance of recycled aggregate concrete," <i>Cement and Concrete Research</i> , vol. 34, no. 10, pp. 1927–1932, Oct. 2004, doi: 10.1016/j.cemconres.2004.02.025.
297 298	[4] N	Ngo, T. D., Kashani, A., Imbalzano, G., Nguyen, K. T., & Hui, D. (2018). Additive manufacturing (3D printing): A review of materials, methods, applications and challenges. Composites Part B: Engineering, 143, 172-196.
299 300 301	[5]	Zhao, Z., Ji, C., Xiao, J., Yao, L., Lin, C., Ding, T., & Ye, T. (2023). A critical review on reducing the environmental impact of 3D printing concrete: Material preparation, construction process and structure level. Construction and Building Materials, 409, 133887.
302 303	[6]	Han, Y., Yang, Z., Ding, T., & Xiao, J. (2021). Environmental and economic assessment on 3D printed buildings with recycled concrete. Journal of Cleaner Production, 278, 123884.
304 305	[7]	Damineli, B. L., Kemeid, F. M., Aguiar, P. S., & John, V. M. (2010). Measuring the eco-efficiency of cement use. Cement and Concrete Composites, 32(8), 555-562.
306 307	[8]	Zhang, C., Nerella, V. N., Krishna, A., Wang, S., Zhang, Y., Mechtcherine, V., & Banthia, N. (2021). Mix design concepts for 3D printable concrete: A review. <i>Cement and Concrete Composites</i> , <i>122</i> , 104155.
308 309	[9]	Dapena, E., Alaejos, P., Lobet, A., & Pérez, D. (2011). Effect of recycled sand content on characteristics of mortars and concretes. <i>Journal of Materials in Civil Engineering</i> , 23(4), 414-422.
310 311	[10]	Ding, T., Xiao, J., Zou, S., & Wang, Y. (2020). Hardened properties of layered 3D printed concrete with recycled sand. <i>Cement and Concrete Composites</i> , <i>113</i> , 103724.
312 313	[11]	Xiao, J., Lv, Z., Duan, Z., & Hou, S. (2022). Study on preparation and mechanical properties of 3D printed concrete with different aggregate combinations. <i>Journal of Building Engineering</i> , <i>51</i> , 104282.
314 315	[12]	Xiao, J., Zou, S., Ding, T., Duan, Z., & Liu, Q. (2021). Fiber-reinforced mortar with 100% recycled fine aggregates: A cleaner perspective on 3D printing. <i>Journal of Cleaner Production</i> , <i>319</i> , 128720.
316 317	[13]	Wu, Y., Liu, C., Liu, H., Zhang, Z., He, C., Liu, S., & Bai, G. (2021). Study on the rheology and buildability of 3D printed concrete with recycled coarse aggregates. <i>Journal of Building Engineering</i> , <i>42</i> , 103030.
318 319	[14]	Ding, T., Xiao, J., Qin, F., & Duan, Z. (2020). Mechanical behavior of 3D printed mortar with recycled sand at early ages. <i>Construction and Building Materials</i> , 248, 118654.

- 320 [15] Zou, S., Xiao, J., Ding, T., Duan, Z., & Zhang, Q. (2021). Printability and advantages of 3D printing mortar with 321 100% recycled sand. Construction and Building Materials, 273, 121699.
- [16] Martínez-García, R., de Rojas, M. S., Jagadesh, P., Lopez-Gayarre, F., Morán-del-Pozo, J. M., & Juan-Valdes, 322 A. (2022). Effect of pores on the mechanical and durability properties on high strength recycled fine aggregate 323 mortar. Case Studies in Construction Materials, 16, e01050. 324
- [17] M. Pigeon, J. Marchand, and R. Pleau, "Frost resistant concrete," Construction and Building Materials, vol. 10, 325 326 no. 5, pp. 339-348, Jul. 1996, doi: 10.1016/0950-0618(95)00067-4.

- 327
- [18] M. Pigeon, R. Gagné, P.-C. Aïtcin, and N. Banthia, "Freezing and thawing tests of high-strength concretes,"
 Cement and Concrete Research, vol. 21, no. 5, pp. 844–852, Sep. 1991, doi: 10.1016/0008-8846(91)90179-L.
- [19] K. El Cheikh, S. Rémond, N. Khalil, and G. Aouad, "Numerical and experimental studies of aggregate blocking
 in mortar extrusion," *Construction and Building Materials*, vol. 145, pp. 452–463, Aug. 2017, doi:
 10.1016/j.conbuildmat.2017.04.032.
- [20] NBN EN 196-1, "Méthode d'essais des ciments Partie1: Détermination des résistances." 2016. Accessed: May
 19, 2023. [Online]. Available: https://www.nbn.be/data/r/platform/frontend/detail?p40_id=174469&p40_language_code=nl&p40_detail_id=78
- 336 149&session=0
- [21] R. J. M. Wolfs, F. P. Bos, and T. A. M. Salet, "Hardened properties of 3D printed concrete: The influence of
 process parameters on interlayer adhesion," *Cement and Concrete Research*, vol. 119, pp. 132–140, May 2019,
 doi: 10.1016/j.cemconres.2019.02.017.
- [22] NBN B 15 211, "Concrete testing Direct tensile strength." 1974. Accessed: May 21, 2023. [Online]. Available:
 https://shop.standards.ie/en-ie/standards/nbn-b-15-211-1974-740821_saig_nbn_nbn_1798581/
- 342 [23] NF P18-459, "Essai pour béton durci Essai de porosité et de masse volumique," p. 9p, Mar. 2010.
- [24] NBN EN 13057, "Products and systems for the protection and repair of concrete structures test methods determination of resistance of capillary absorption." 2002. Accessed: May 22, 2023. [Online]. Available:
 https://shop.standards.ie/en-ie/standards/nbn-en-13057-2002-763729_saig_nbn_nbn_1844397/
- [25] RNR 50-1 (3.0), "Note réglementaire pour fiches techniques, notes justicatives et études préliminaires du béton routier | COPRO." Accessed: Jan. 27, 2023. [Online]. Available: https://www.copro.eu/en/document/rnr-50-1-30note-reglementaire-pour-fiches-techniques-notes-justicatives-et-etudes
- [26] CEN/TS 12390-9, "Testing hardened concrete Part 9: Freeze-thaw resistance with de-icing salts Scaling," p.
 10p, 2016.
- [27] G. Ma, Z. Li, L. Wang, F. Wang, and J. Sanjayan, "Mechanical anisotropy of aligned fiber reinforced composite
 for extrusion-based 3D printing," *Construction and Building Materials*, vol. 202, pp. 770–783, Mar. 2019, doi:
 10.1016/j.conbuildmat.2019.01.008.
- [28] V. Mechtcherine *et al.*, "Extrusion-based additive manufacturing with cement-based materials Production steps,
 processes, and their underlying physics: A review," *Cement and Concrete Research*, vol. 132, p. 106037, Jun.
 2020, doi: 10.1016/j.cemconres.2020.106037.
- [29] A. U. Rehman and J.-H. Kim, "3D Concrete Printing: A Systematic Review of Rheology, Mix Designs,
 Mechanical, Microstructural, and Durability Characteristics," *Materials*, vol. 14, no. 14, p. 3800, Jul. 2021, doi: 10.3390/ma14143800.
- 360 [30] N. Algourdin, Q. N. A. Nguyen, Z. Mesticou, and A. Si Larbi, "Durability of recycled fine mortars under freezecycles," Construction Building Materials, 2021, 361 thaw and vol. 291, May doi: 10.1016/j.conbuildmat.2021.123330. 362
- 363 364

Table 1—Mixtures proportions of mortars (kg) [1 kg = 2.20 lbs]

	Cement	Sand	Water	Plasticizer	VCA	W _{eff} /C
RSM/NSM	905,00	995,60	313,52	22,63	1,81	0,29

Table 2—Tensile strength and failure pattern of printed specimens RSM_P

	Tensile strength	Failure pattern*	Average tensile strength
	MPa [psi]	mm [in]	MPa [psi]
7 days	2.02 [292.98]	28 to 34 [1.10 to 1.34]	
	2.14 [310.38]	13 to 25 [0.51 to 0.98]	$2.03 \pm 0.11 \; [294.43 \pm 15.95]$
	1.92 [278.47]	18 to 30 [0.71 to 1.18]	
28 days	2.71 [393.05]	12 to 16 [0.47 to 0.63]	
	2.52 [365.50]	18 to 25 [0.71 to 0.98]	$2.51 \pm 0.20 \; [364.04 \pm 29.00]$
	2.32 [336.49]	18 to 27 [0.71 to 1.06]	
56 days	2.43 [352.44]	17 to 28 [0.67 to 1.10]	
	3.03 [439.46]	20 to 33 [0.79 to 1.30]	$2.69 \pm 0.31 \; [390.15 \pm 44.96]$
	2.60 [377.10]	23 to 33 [0.91 to 1.30]	
91 days	2.28 [330.69]	26 to 32 [1.02 to 1.26]	
	2.11 [306.03]	17 to 31 [0.67 to 1.22]	$2.25 \pm 0.13 \; [326.33 \pm 18.85]$
	2.37 [343.74]	19 to 27 [0.75 to 1.06]	

*The failure pattern was measured from the top of the specimens







(a)

Fig. 2—Cartesian printer of IMT Nord Europe (a) and the printed element in « S » shape (b)



Fig. 3—Schematic depicting the printed sample and the illustration of the sampling (a), along with the various orientations of load application on the samples [9] (b)



(a)

(b)

379 Fig. 4—Cored of printed samples (a) and the INSTRON instrument using for tensile strength test (b)



Fig. 6—Flexural (a) and compressive strength (b) of cast mortar containing recycled sand RSM_C and natural sand
 NSM_C underwent water curing



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 $[1 \text{ kg/m}^2 = 0.2048 \text{ lb/ft}^2]$

Fig. 7—Amount of water absorbed per unit area (a) and the water absorption coefficient (b) for cast mortar samples
 containing recycled sand RSM_C and natural sand NSM_C in two orientations, namely (oz) and (ox), after 24 h of
 absorption.



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[1 MPa = 145.04 psi]

Fig. 8—Flexural (a) and compressive strength (b) of cast mortar with recycled sand under water curing and ambient curing





400 Fig. 9—Compressive strength of printed mortar specimens which the layer parallel to loading direction RSM_P (oz),
 401 the layer perpendicular to loading direction RSM_P (ox), and cast mortar RSM_C



Fig. 10-Failure pattern of printed specimens after tensile test

[1 cm = 0.3937 in]





Fig. 11—Porosity (a) and bulk density (b) of cast and printed specimens underwent water curing





411Fig. 12—Amount of water absorbed per unit area of cast mortar RSM_C and NSM_C and printed mortar RSM_P412along the (oz) orientation





(a)



(b)

$[1 \text{ kg/m}^2 = 0.2048 \text{ lb/ft}^2]$

Fig. 14—Cumulative spalling particles of cast and printed specimens submitted to different surface conditions during
 test (a), and surface conditions of RSM_P specimens after 91 freezing and thawing cycles (b) (1st row are the
 specimens with water solution, 2nd row are the specimens with salt solution, and 3rd row are the specimens with no
 solution)