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Innovative Pavement Materials: Utilizing Corn Stover and Fly Ash in Geopolymers

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Abstract: The development of each nation is evaluated by its infrastructure, and each nation is competing with the others in infrastructure advancement, especially in the construction of roadways, since they play a vital role in the economic and social development of the nation. The conventional materials used for road construction are concrete and asphalt, which pose significant environmental challenges. This research gives insight into the potential of fly ash (FA) and corn stover (CS) in synthesizing geopolymer, as an alternative material for the construction of roads. This study examines the impact of three FA and CS mixture percentages and the particle size of CS on the compressive strength and porosity of geopolymer. The results indicate that incorporating larger amounts of CS in fly ash-based geopolymer may decrease the compressive strength of the geopolymer. Smaller CS particle sizes also tend to lead to lower compressive strength. Porosity of the geopolymer tended to increase with the incorporation of higher percentages of CS, particularly for smaller corn stover sizes. As a fine aggregate replacement for geopolymer, CS incorporation has the potential to reduce mined aggregate obtained from a process that harms the environment.

Keywords: biomass; waste; agriculture; aggregate; sodium hydroxide; sodium silicate; sustainability

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1. Introduction

The growing use of asphalt and concrete in the construction of pavement can affect the world's environment adversely. Both asphalt and concrete are produced using unsustainable methods; asphalt depends on crude oil production, and concrete production requires mined rock, sand, and cement feedstocks [1]. Crude oil transportation has caused oil spills that harm ocean ecosystems [2]. While efforts to reuse existing materials have emerged, the overall production of these materials still consumes substantial energy and produces large amounts of carbon dioxide [3].

The conversion of petroleum into asphalt releases significant quantities of volatile organic compounds (VOCs), adding pollutants to the air [4]. Similarly, the cement production process for concrete involves high temperatures and generates substantial VOC emissions [5,6]. The environmental impact of building roads and parking lots is heightened by the application and curing of asphalt, which releases more toxic elements into the atmosphere [5].

Mining fine aggregates used in concrete is harmful to the environment [7]. The disturbance of soil and vegetation during extraction can increase the risk of erosion and runoff, transporting sediments and pollutants into nearby water bodies and leading to deteriorating water quality. Sediments and contaminants from riverbed extraction sites also impair aquatic ecosystems [8].

To mitigate the negative impact on the environment, addressing these concerns demands an integrative strategy that considers sustainable construction processes and alternative materials. An alternative to using fossil fuel or mining-based binders for road

materials is using geopolymers. Geopolymers are inorganic polymers made by activating aluminosilicate minerals, which are typically industrial byproducts or wastes [9,10]. Comparable strength characteristics are provided by geopolymers, and they can have a significantly lower environmental impact. Geopolymers can use the waste material fly ash (FA), a leftover from coal combustion, as a silica source [10,11].

Other non-mined possible additives to geopolymeric material include powdered banana peel, which has been reported to improve strength [12]. Adding discarded cork to geopolymers has been investigated and found to not interfere with the geopolymerization process, while reducing density of composites [13]. A mixture of coffee grounds, granulated blast furnace slag, and FA was reported to meet the structural strength requirement for road embankment subgrade materials [14]. FA and rice husk ash can also be used in geopolymer to give reasonably high compressive strengths [15]. Mechanically ground glass powder has been used in geopolymers and is reported to give reasonable strength and durability [16].

FA mixes' compactness has been reported to increase with the addition of raw biomass, especially fibrous materials like wood residues [10]. Corn stover (CS), the leaves and stalks of corn (maize), is an abundant agricultural waste left over after corn is harvested. CS shows great promise as aggregate for use in geopolymer in the United States because of its low cost, concentrated distribution in the Midwestern U.S., and its availability of 31 billion kg per year without depleting the soil [17]. Little research has been reported on the combination of CS and FA in geopolymer.

Adding raw biomass to geopolymer is challenging and there are limitations to the amount able to be added, as certain raw biomass, such as powdered banana peel, can result in long setting times due to additional potassium in the biomass. The goal of this research was to study the impact of adding CS and FA to standard geopolymer materials to better understand their potential uses in the construction industry. The effect of changes in the percentage of CS and the size of the particles on the compressive strength and porosity were examined. The work investigated possible advantages, such as enhanced workability and sustainability, related to CS addition to fly ash-based geopolymer samples. The innovative nature of this study is that it uses raw corn stover, a relatively unprocessed food production waste product that is abundant.

2. Materials and Methods

2.1. Materials

The Class F fly ash (FA) used in this experiment was donated by ECO Materials Technology, South Jordan, Utah, USA. FA was generated from combusting coal in a high-temperature furnace. It consisted of calcium oxide (CaO), iron oxide (Fe_2O_3), silicon dioxide (SiO_2), and aluminum oxide (Al_2O_3). Table 1 shows the composition.

Table 1. Chemical composition of fly ash used [18].

Chemical Analysis	Results %	ASTM Limit Class F/C	AASHTO Limit Class F/C
Silicon Dioxide	51.30	-	-
Aluminum Oxide	27.18	-	-
Iron Oxide	12.21	-	-
Sum of Primary Oxides	90.69	70.0/50.0 min	70.0/50.0 min
Sulfur Trioxide	0.89	5.0 max	5.0 max
Calcium Oxide	2.72	-	-
Magnesium Oxide	0.83	-	-
Sodium Oxide	0.23	-	-
Potassium Oxide	2.41	-	-
Sodium Oxide Equivalent	1.82	-	-
Moisture	0.73	3.0 max	3.0 max
Loss on Ignition	3.22	6.0 max	5.0 max
Carbon	2.10	-	-
Available Alkalis	0.59	Not required	1.5 max

The corn stover (CS) used in this experiment was donated from Idaho National Lab, Idaho Falls, Idaho, USA. Table 2 shows its composition. The remainder of the material would be non-extractable inorganics.

Table 2. Composition of CS used.

Component of Corn Stover	% Composition
Structural Ash	3.46
Extractable Inorganics	2.23
Structural Protein	1.58
Extractable Protein	0.96
Water-extracted Glucan	0.53
Water-extracted Xylan	0.26
Water extractives Others	2.36
EtOH Extractives	2.62
Lignin	16.52
Glucan	37.52
Xylan	21.77
Galactan	1.66
Arabinan	3.37
Acetate	2.45
Total	97.28

Sodium hydroxide (NaOH) solution (12 M) was purchased from Fisher Scientific (Hampton, NH, USA). Sodium silicate (Na_2SiO_3) solution was purchased from Sigma-Aldrich (St. Louis, MO, USA). The 5.08 cm (2 inch) poly-cube 3-gang cubic mold used in the experiment was made of polyethylene (HDPE) and purchased from American Cube Mold (Brunswick, OH, USA).

2.2. Methods

2.2.1. Geopolymer, Fly Ash, and Corn Stover Ratios

The ratios of the FA and CS compositions that were chosen for sample preparation are displayed in Table 3. Each experimental condition was tested in triplicate.

Table 3. The ratios and particle size used in geopolymers with CS and FA.

Experimental Condition	Mass Ratio of FA to CS	Particle Size of CS (μm)
1	100:0	-
2	95:5	180
3	90:10	180
4	95:5	710
5	90:10	710

2.2.2. Geopolymer Sample Preparation

FA, CS, NaOH, and Na_2SiO_3 were combined to form the geopolymer samples. The mass ratio of combined FA and CS to alkaline activators was 1.86, while the mass ratio of Na_2SiO_3 to NaOH was 0.33. In separate beakers, the alkaline activators and the masses of FA and CS were measured. After pouring the weighed samples of CS and FA, one at a time, into the mixing bowl, the mixer was set to stir at low speed. Three minutes later, the weighed NaOH samples were added gradually and mixed for the next 3 min. After that, the mixer was switched off, and samples that had adhered to the mixing bowl's side were physically scraped using a silicon spatula. After 1 min, the mixer was started, and 3 more minutes of mixing was performed, with the weighed Na_2SiO_3 added gradually. The samples that were stuck to the sidewalls were then scraped again after the mixer was switched off, and the process was repeated. After 1 min, the mixer was left running constantly for the following 4 min. The mixing process took about 15 min in total.

Molds were prepared and thoroughly oil-lubricated. Using the tamper that came with the mold, each portion of the mold was filled with the mixture and tamped. Lids were placed over the upper part of the molds and secured with rubber bands. After that, the molds were put inside the oven, which had been preheated to 70 °C. After the samples had been cured for 18 h in the oven, the oven was shut off and the molds allowed to stay inside it for a minimum of 24 h. The samples were taken out of the molds after a day and kept inside zip-lock bags for the following 5 days before compression testing.

2.2.3. Geopolymer Sample Compression Testing

The compression tests were carried out on the seventh day following the samples' preparation. A hydraulic compression test machine (Test Mark Industries, East Palestine, OH, USA) was used to perform the compression test. The loading channel applied was 658.4 kPa/s (300 lbs./s). If a crack or fracture in the sample caused the stress on the cylinder to fall below 85% of the load, this loading was considered to have crushed the sample.

2.2.4. Geopolymer Sample Porosity Testing

A water porosity method was used to find the porosity of samples [19]. Briefly, 5–10 g of the fragments from compression testing of the samples were immersed in DI water for 24 h, after which they were removed and vacuum filtered. The samples' wet weights were measured and noted. The samples were then put in the oven for 72 h at 105 °C, and every 24 h, the dry weights of the samples were recorded.

3. Results and Discussion

3.1. Compression Tests of Synthesized Geopolymer with Corn Stover (CS) at 0%, 5%, and 10%

Nine samples in total—three sets of three samples each—were prepared for each mixture. The ratios of FA to CS lower than the specified ratios (higher ratios of CS) had far lower strength and were hence excluded from this study.

The compressive strength data for the 710 µm samples are shown in Figure 1 (Tables S1–S3). The average strength for 100% FA was 9510.4 kPa (1379.37 psi). When the CS particle size was 710 µm, the average strengths of 95% FA with 5% CS and 90% FA with 10% CS ratio were 7939.7 kPa (1151.55 psi) and 564.4 kPa (817.2 psi), respectively. No significant difference can be seen between the compressive strength of the 100% FA samples and the samples where 5% of the FA is replaced with CS, suggesting that adding this quantity would be acceptable in geopolymer materials. The reduction in the number of coal-fired power plants in the United States means that less coal fly ash may be available in the future [20]. If CS can replace part of the FA, such biomass-integrated geopolymer could potentially replace other more environmentally detrimental structural materials. Other biomass-based materials that have been studied in geopolymer include ash from rice husk, which when prepared under certain conditions can show good compression strength since it is high in silica [15]. Powdered banana peel can also provide good compression strength since it can act as an additional alkali source, but only a 10% addition is feasible [12].

Figure 2 displays the compressive strength for the 180 µm samples, as are in Tables S1, S4 and S5. The CS-containing average strengths were 4702.9 kPa (682.1 psi) for 90% FA with 10% CS and 4496.8 kPa (652.2 psi) for 95% FA with 5% CS, with a particle size of 180 µm. The compressive strength of the lower percentage composition of CS was found to be the highest, as shown in Figure 2. However, experimental variability means that no significant difference in compressive strength can be found between the 0% and 5% CS samples.

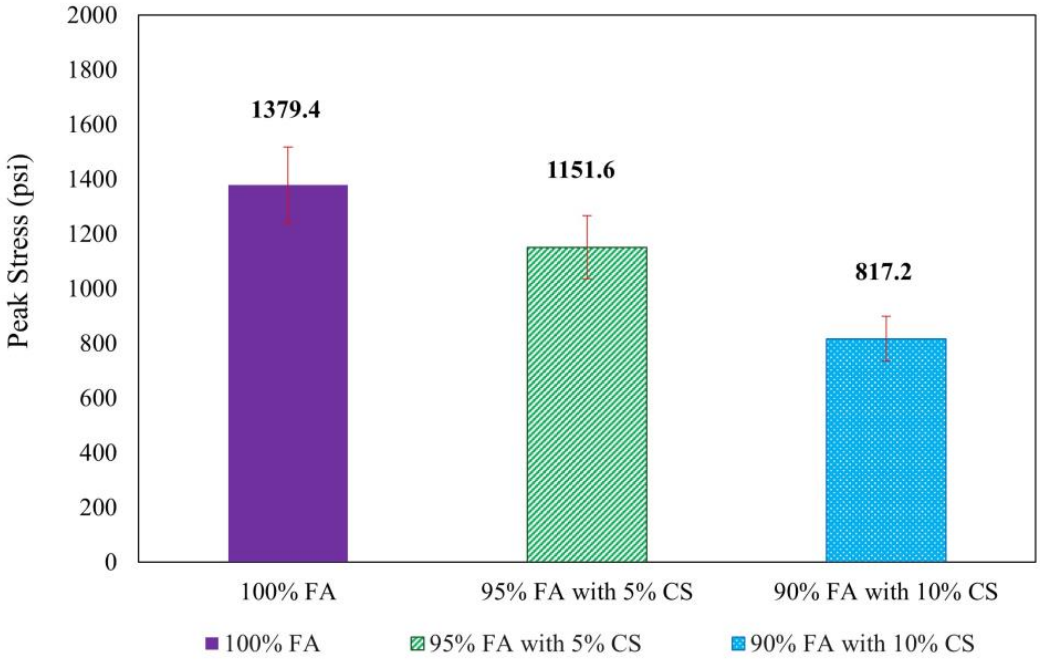

Figure 1. Compressive strength of synthesized geopolymer with and without CS (710 µm particles) at 0%, 5%, and 10%, with standard error bars.

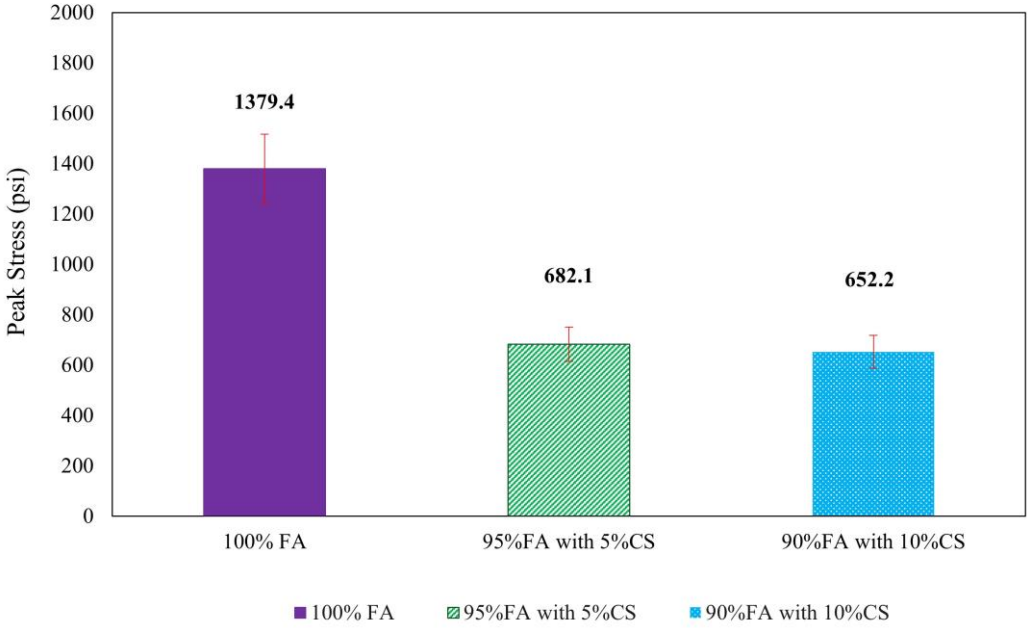

Figure 2. Compressive strength of synthesized geopolymer with and without CS (180 µm particles) at 0%, 5%, and 10%, with standard error bars.

Comparing Figure 1 (710 µm particle size) with Figure 2 (180 µm particle size), it is obvious that the larger particle size gave higher compressive strength. The larger particle size CS may be acting as an aggregate, impeding crack line formation in the samples and increasing the compressive strength. The smaller particle size CS may, even if well bonded with the other components, may not present such a barrier to crack formation [21,22].

3.2. Porosity Testing of Synthesized Geopolymer with CS at 0%, 5%, and 10%

Figure 3 shows the porosity of 710 µm samples after drying for 24, 48, and 72 h, as are in Tables S6–S8. For 100% FA samples, porosity tests showed readings of 39.26% (24 h), 40.78% (48 h), and 40.98% (72 h). Regardless of drying time, similar results were obtained

from porosity tests on 5% CS and 95% FA samples and on 10% CS and 90% FA samples; all these samples with CS had an average porosity of ~46%. No significant difference can be seen in porosity with CS addition. The slightly higher average difference compared to samples with no CS may be due to imperfect bonding of the CS with the other geopolymer components. The CS again may be acting more like an aggregate. Higher porosity tends to lead to lower compressive strength, corroborating the data for these CS containing samples shown in Figure 1 [23].

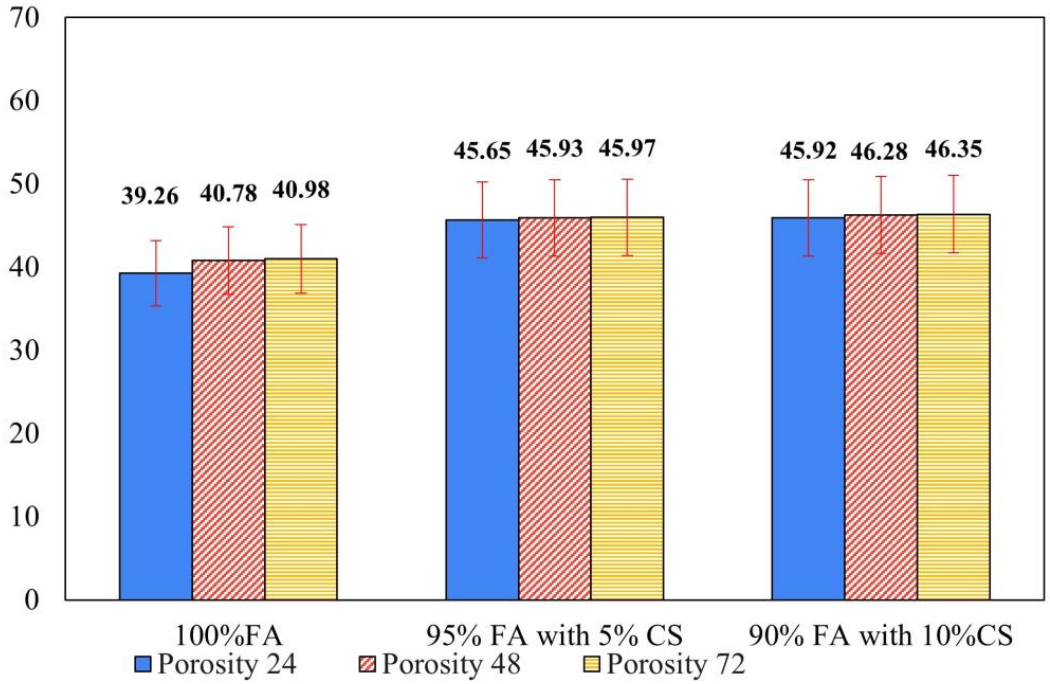

Figure 3. Porosity of synthesized geopolymer with and without CS (710 μm particles) at 0%, 5%, and 10% after drying for 24 h, 48 h, and 72 h.

Figure 4 shows the porosity data for samples with and without 180 μm particle size CS addition, as in Tables S6, S9 and S10. Although not proven significant, the averages seen suggest that porosity is increased with CS addition and the greater the amount of CS added, the higher the porosity. This finding would corroborate the data in Figure 2, since higher porosity tends to decrease compressive strength [23]. However, variation in the data means that increased porosity with CS addition cannot be definitely concluded.

Comparing Figures 3 and 4, a higher average porosity can be seen for the samples with smaller particle size CS added. This difference may be attributed to the higher surface area for the smaller particle size CS added to the geopolymer.

Liu et al. (2022) investigated the use of crushed coconut shell as an aggregate in geopolymer [24]. They reported the thickness of the interface transition zone between the concrete cement material matrix and the crushed coconut shell to be greater than that for typical aggregate, due to the shell aggregate absorbing moisture [24]. The corn stover may have also been absorbing moisture, with increased interface transition zone resulting in higher porosity. This would explain the smaller CS particles, with higher surface area, giving higher average porosity. Larger interface transition zones would likely decrease compressive strength, as was found with CS addition. Liu et al. (2022) found lower compressive strength with crushed coconut shell addition, compared to the control [24].

Adding CS and FA to the geopolymer could have real applications in not only pavement and building construction, but also in soil stabilization [25]. Geopolymer has been found to have a positive environmental impact compared to conventional cement [25].

Future work, attempting to use larger-particle-size CS in geopolymer, is needed to find if it can serve as a more sustainable aggregate and possibly increase geopolymer porosity to reduce runoff that generates flooding [26].

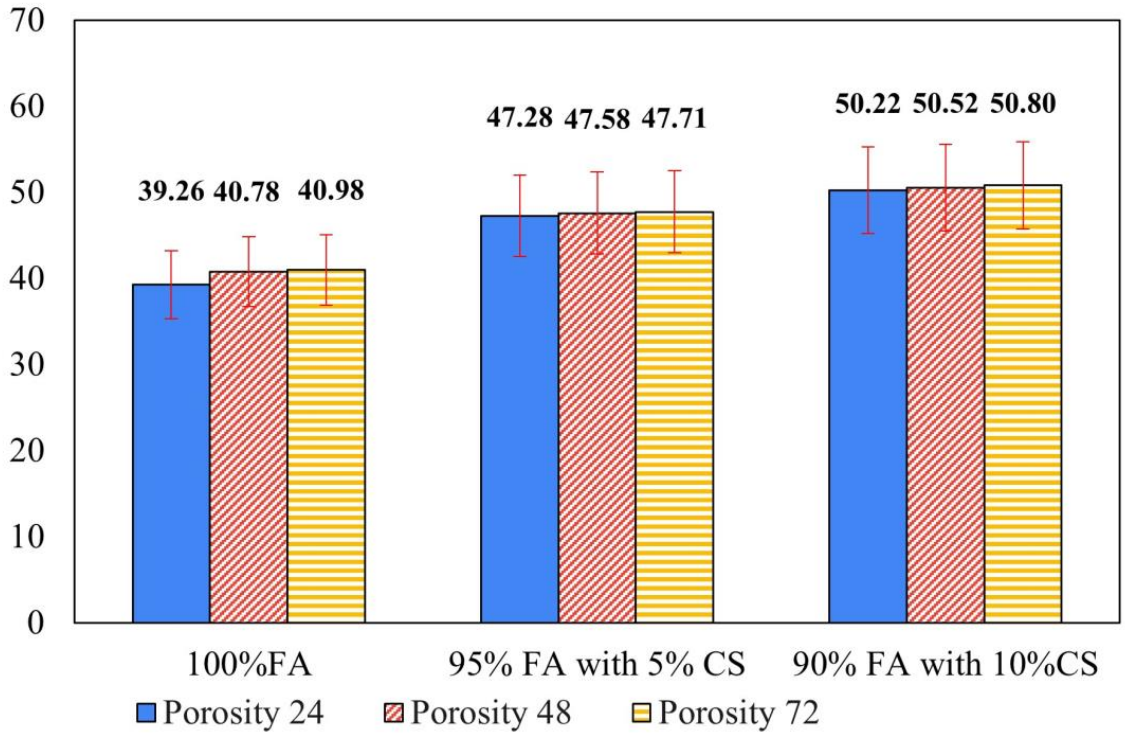

Figure 4. Porosity of synthesized geopolymer with and without CS (180 µm particles) at 0%, 5%, and 10% after drying for 24 h, 48 h, and 72 h.

4. Conclusions

Agricultural wastes have the potential to offer viable solutions for sustainable building materials because they would substitute for mined resources and may reduce carbon emissions. Sustainability in building materials is becoming critically important as environmental concerns and the depletion of natural resources increase. Using waste biomass from food production, such as CS, could have a significant impact in reducing carbon-intensive mining. Geopolymers can be made from coal industrial wastes like FA. In this present study, compressive strength decreases when more than 5% of CS replaces FA in the geopolymer. This implies that, to preserve load-bearing capacity, there may be a limit to the percentage of CS incorporation possible. The CS of 710 µm particle size contributed to higher compressive strengths than the 180 µm smaller CS particles. Average porosity tended to increase with CS addition.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/environments11090192/s1, Table S1: 100% FA, 0% CS Compressive Strength; Table S2: 95% FA, 5% CS (710 µm) Compressive Strength; Table S3: 90% FA, 10% CS (710 µm) Compressive Strength; Table S4: 95% FA, 5% CS (180 µm) Compressive Strength; Table S5: 90% FA, 10% CS (180 µm) Compressive Strength; Table S6: 100% FA, 0% CS Porosity % measured at 24 h, 48 h, and 72 h; Table S7: 95% FA, 5% CS (180 µm) Porosity % measured at 24 h, 48 h, and 72 h; Table S8: 90% FA, 10% CS (710 µm) Porosity % measured at 24 h, 48 h, and 72 h; Table S9: 95% FA, 5% CS (180 µm) Porosity % measured at 24 h, 48 h, and 72 h; Table S10: 90% FA, 10% CS (180 µm) Porosity % measured at 24 h, 48 h, and 72 h.

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Validation, J.G.L., A.P., V.S., M.T. and S.A.; Visualization, J.G.L., A.P. and V.S.; Writing—original draft, A.P.; Writing—review and editing, J.G.L., A.P. and V.S. All authors have read and agreed to the published version of the manuscript.

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