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A study on the optimization of superplasticizer and water-to-binder ratio for enhancing 3D printable concrete with marble powder waste

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ABSTRACT

This study focuses on developing a 3D-printable concrete mix by optimising the superplasticizer dose and water-to-binder ratio while substituting 10% of marble powder waste as a cement replacement. The step-by-step methodology for achieving the optimal 3D printable material mix is outlined, encompassing the mix proportions, material fineness, and the procedures involved. Experimental investigations were carried out using a 3D Printer, including tests for flow, extrudability, and buildability to refine the printable mix and compressive strength tests were carried out. The optimal mix was achieved by adjusting the water-to-binder ratio, and superplasticizer dosage. The results show that a printable mix with 0.35% SP dosage, 0.35 w/b ratio, and 10% marble powder replacement exhibits superior printing quality.

Keywords: 3D printable mix, buildability, extrudability, flow value, methodology.

INTRODUCTION

3D concrete printing (3DCP), an emerging additive manufacturing technique, has attracted considerable interest in revolutionizing the construction industry over the past decade. The construction sector has encountered substantial challenges in recent years, grappling with labor shortages, resource constraints, and safety concerns that have impeded its growth amid rapid urbanization and infrastructure expansion globally. To address these pressing issues and drive productivity, the industry can embrace automation and digitalization of its processes. Through 3DCP, the quality and speed of construction can be enhanced while eliminating the need for formwork and reducing labor costs. Additionally, the layer-by-layer deposition approach of 3DCP saves time and material resources, enable customization of complex design configurations, reduces material wastage and minimizing safety hazards [1–4]. These technological advancements can help the construction industry overcome the constraints it currently faces and support its growth in the face of growing demand for infrastructure and urban development [6, 7].

In 3DCP, various measures such as extrudability, workability, open time and buildability were the four crucial factors that were suggested by the researchers to evaluate the newly developed characteristics of 3D printable concrete [8, 9]. The ratio and type of the material used are the influencing factors for 3DCP in both its fresh and hardened properties [10]. The admixtures like chemical and mineral additives plays a crucial role in 3D printing to achieve the printable concrete which is essential for successful printing. These additives are utilized to enhance various characteristics of the printable concrete, including buildability, shape retention, pumpability, thixotropy, inter-bond layer adhesion, strength development, and shrinkage control [11]. Admixtures such as superplasticizers and retarders dosage helps in maintaining the rheological properties of concrete, increasing its open time, and enhancing the buildability of the mix while printing [6, 12]. The amount of superplasticizer is important in influencing the behaviour of fresh cementitious paste [13] Additionally, the addition of superplasticizer in 3DCP mixtures is essential for successful extrusion, as it can lower the yield stress and plastic viscosity of the concrete, contributing to its printability. The dosage of superplasticizer should be carefully controlled within a specific range to achieve the desired rheological properties for 3D printing concrete effectively [14]. The slump flow test and flow table test are the most commonly adopted basic methods to assess the flowability and printability of 3DCP [11, 15].

3DCP offers many advantages, such as the ability to create complex designs, reduce material waste, and streamline construction processes. However, a key challenge is that the printable concrete mixes typically contain 1.5 to 2 times more Portland cement compared to conventional concrete. The cement factory is responsible for 8–9% of the world's CO_2 emissions. So, the researchers often rely on various mineral additives to reduce the use of cement content. Numerous studies have shown the advantages of using GGBS, flyash, silica fume and other supplementary cementitious material on the rheological characteristics and hardened performances of 3D printing materials [16–20]. Fly ash improves the pumpability of 3D printing by lowering the fresh cement composite's yield stress and plastic viscosity [21], when there is a greater replacement percentage buildability suffers from delayed initial setting and early hydration. On the other hand, silica fume could be used to enhance the buildability of the cement-FA-silica fume blended ternary binder system for 3DCP [22]. GGBS helps to improve the buildability of the mix, which can be crucial for the stability, strength, and durability of the final structure due to angular particle interlocking [23].

Nevertheless, the literature lacks investigation on using marble powder waste (MPW) in 3DCP. In marble industry, 50% of waste is produced during cutting and polishing of the marble blocks which are disposed outside, polluting and harming the natural landscape. An estimated 0.28 million tons of CO_2 emissions were reduced annually as a result of the replacement of 10% MPW to cement [24]. Waste products from the marble industry must be used as raw materials in promoting waste valorization, and offering economic advantages in construction practices. MPW can be used as cement replacement or as filler material which it enhances the flow consistency, decreasing the voids and increasing packing density in the material mix [25, 26]. The marble powder of 10–15% replacement is found to be an optimum percentage by many of the researchers [27–32]. When comparing 3DCP to traditional constructions, one issue is the weakness caused by the interlayer zones, which are usually more porous [33, 34]. In such cases, this can be overcome by using finer materials like marble powder, can fill in the small voids or pores in the mix, potentially improving density and strength while reducing porosity and also helps in improving the workability of the mix.

This study aims to enhance the development of sustainable 3D printing concrete compositions by systematically evaluating the content of waste marble powder, water-to-binder ratio, and superplasticizer. The results are intended to support innovation and sustainability in the building sector by providing guidance for concrete mix design techniques for 3D printing applications.

MATERIALS AND EXPERIMENTAL APPROACH

Materials

Cement

Ordinary Portland cement (OPC) of 53 grade, as shown in Figure 1 (a) conforming to IS: 269-2015 [35], was used for the experiments. This cement has a specific gravity of 3.15. The chemical composition of the cement is provided in Table 1. The cement exhibits an initial setting time of 105 minutes and a final setting time of 255 minutes. Additionally, the cement has a fineness of 3.5% and a consistency of 31%.

Marble powder waste

The marble powder waste obtained from marble processing industries in the Figure 1 (b) was used as a partial replacement for cement in the mix, which has a specific gravity of 2.83. The chemical composition of the marble powder waste (MPW) is determined using X-ray fluorescence (XRF) analysis, presented in Table 1.

Table 1. Chemical composition of cement and marble powder

Chemical composition	Percentage (%)	
	Cement	MPW
CaO	66.47	56.86
MgO	0.93	30.76
SiO_2	18.66	4.19
Al_2O_3	4.35	1.35
Fe_2O_3	4.89	3.86
SO_3	2.93	0.04
K_2O	0.58	0.01

M-Sand

In a consequence to the scarcity of river sand, contemporary construction practices increasingly employ manufactured sand. Accordingly, this study adopts manufactured sand (M-sand) in the Figure 1c as fine aggregate with the specific gravity of 2.4. The fineness of the aggregate in the mix is an important factor in the better extrusion and printing of 3DCP. Herein, sand particles passing through a sieve size of 1.18 mm are utilized.

Superplasticizer

The Polycarboxylic ether polymer-based Superplasticizer (SP) is used to maintain the workability retention in the mix, which helps for better extrusion and print quality while printing.

Methodology

Mixing procedure

In the mixing process, dry materials are initially added to the mixer and rotated for a duration of 3 minutes to achieve a homogeneous mixture. Subsequently, 70% of the required water content which is blended with the optimal dosage of superplasticizer is added and the mixture is rotated for the next 6 minutes. Finally, the remaining 30% of the liquid component is incorporated into the mixture and rotated for 6–8 minutes until attaining the better consistency.

3D printing trials

This study incorporated the replacement of cement with 10% of marble powder waste as supplementary cementitious material, maintained binder to sand ratio of 1:1.2. The trials were performed by changing the superplasticizer dosage from 0.2 to 0.35 with offset of 0.05, water to binder ratio 0.3 and 0.35 as detailed in Table 2. Mixes M1 to M6 were formulated by adjusting the dosage of superplasticizer (SP) and water-to-binder (w/b) ratio, focusing to achieve the better extrudability, buildability and printability. Based on the experimental trials through flow table test, extrudability test and buildability test, methodology for mix optimisation has been framed for good printing and it is as shown in the Figure 2.

(a) (b) (c)

Figure 1. Raw materials used in 3DCP mix (a) cement; (b) marble powder; (c) M-sand

Table 2. Mix proportion of experiments

Mix	Cementitious materials: Fine aggregate	MPW replacement %	w/b	SP
M1	1:1.2	10	0.30	0.20
M2	1:1.2	10	0.30	0.25
M3	1:1.2	10	0.30	0.30
M4	1:1.2	10	0.30	0.35
M5	1:1.2	10	0.30	0.40
M6	1:1.2	10	0.35	0.35

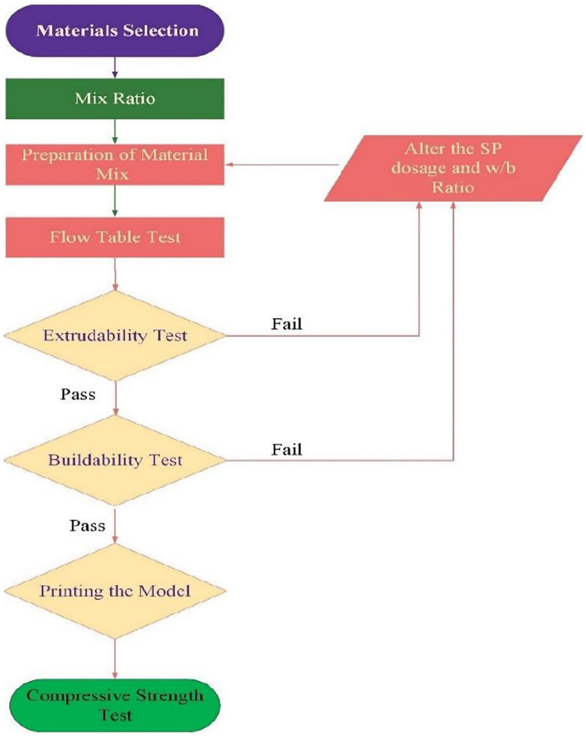

Figure 2. Methodology for optimising the mix

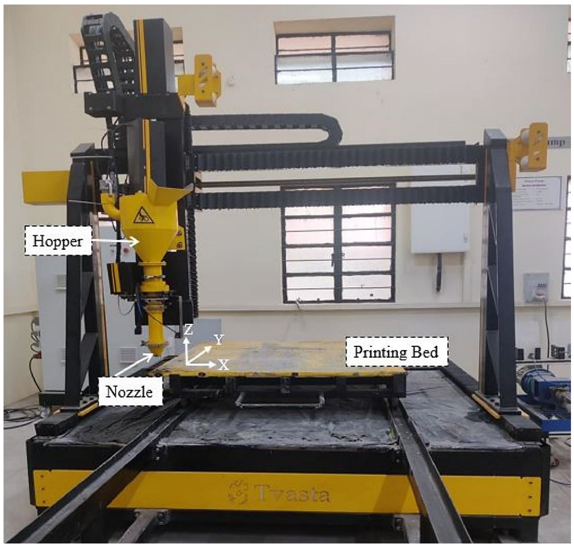

Figure 3. 3D printer

3D printer and stages of printing

Printing has been carried out using Tvasta Nirmaan Research & Development concrete 3D printer as shown in Figure 3, a gantry-based 3D construction printer with built volume 1100 × 1100 × 600 mm. In this experimental study for printing, a 30 mm circular nozzle was used with the printing speed of 50 mm/s in three directional axes of X, Y and Z. Hopper in the printer is screw based extrusion with the capacity of 10 litres, and manual loading of material input method in hopper is followed. Tvasta Digital Modelling software is used for printing the sample. The stages of printing are shown in Figure 4, Modelling and printing of the sample is carried out as illustrated in the Figure 5.

EXPERIMENTAL METHODS

As there is no codal provision for the tests in 3D printing concrete. Based on the literature, flow table, extrudability, buildability, and compressive strength tests were conducted and concluded accordingly.

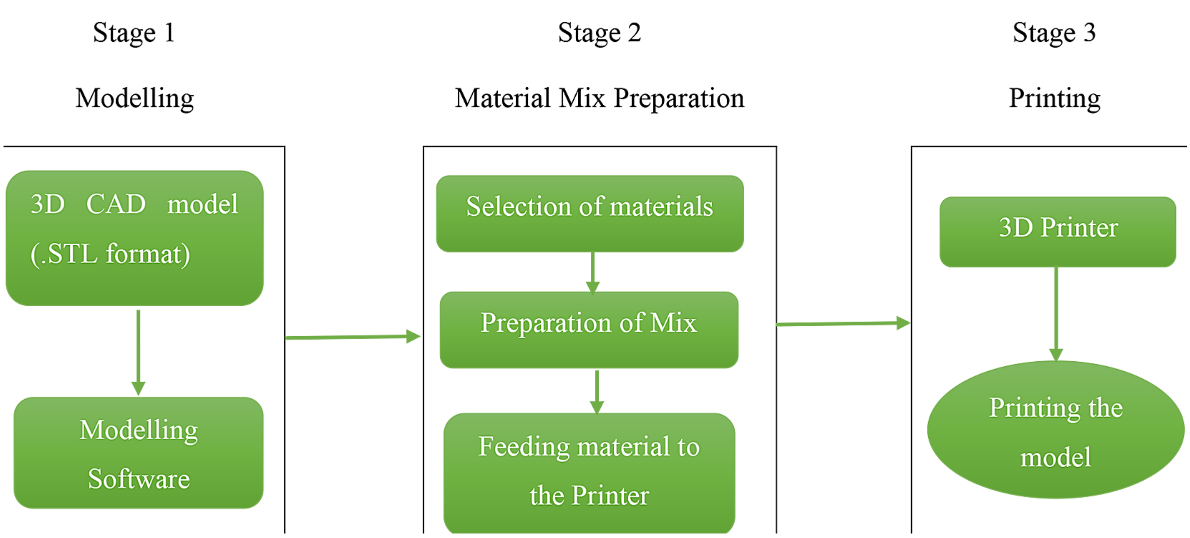

Figure 4. Stages of 3D printing

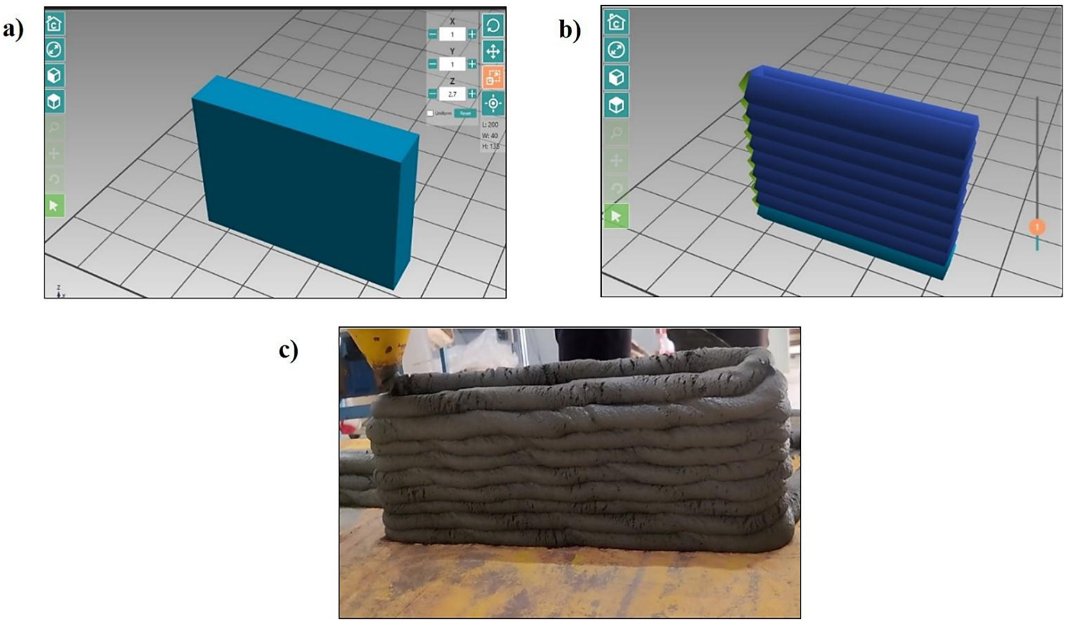

Figure 5. Modelling and printing a) 3D modelling (.stl file) b) 3D slicing the model c) 3D concrete printing

Flow table test

The flowability of the material mix was assessed in a flow table following the ASTM C1437-20 standard [36]. The procedure involved filling the prepared material mix into a flow table mold, tamping it 20 times with a tamping rod. Then filling the mold with material in layer and removing the mold from the flow table. Dropping the table 25 times and measuring the spread diameter of the material to calculate the flow.

Extrudability test

The extrudability is tested by the continuous extrusion of the fresh material mix from the nozzle of the printer without any obstacles like blockage in the nozzle, layer tearing, layer breakage, cracks, discontinuities, segregation, or bleeding [1, 37]. This extrusion depends upon the water to binder (w/b) ratio, SP dosage, printer parameters such as size of the nozzle, etc., [9]. This criterion of ensuring the better extrusion is crucial as it directly impacts the buildability and structural integrity of the printed material. After the material is extruded out of the nozzle the extrudability is assessed by printing the specific model. Better extrusion and printing are achieved by changing the water to cement ratio, SP dosage and some parameters such as extrusion nozzle size, printing speed [38]. In overall, the flow value of the mix and particle size of the material and its proportion also plays major role in achieving the printable mix [9, 15].

Buildability test

The buildability of 3D Printable Concrete is evaluated based on the higher number of layers that can be deposited without collapsing and the layers on top of the other need to be able to withstand the weight of the subsequent layers, which is a critical factor in the fabrication of concrete structures through 3D printing [1, 23, 24]. This property is closely related to the vertical deposition of filament layers and is influenced by the flowability of the fresh material, as well as the bonding between extruded filaments. The buildability of the developed 3DCP mixes is examined by printing the rectangle shape structure.

Compressive strength test

One of the typical criteria for evaluating the quality of concrete is its compressive strength test. It has been performed on both cast and printed specimens. The mixtures that met the criterion for flow table, extrudability, and buildability tests are the printable mix. The printable mix, samples were printed and tested. To determine the compressive strength, the compression testing machine was used with the rate of loading 0.6 kN/s as per IS

516-1959 [39]. A rectangular prism specimen with dimensions of 500 × 120 × 60 mm was printed as in Figure 6a, from which 50 × 50 × 50 mm cubes samples were cut for testing. These cubes were oriented along the X, Y, and Z loading directions as depicted in Figure 6b and subjected to curing period of 7 and 28 days for testing. The test results were then compared to the mold-cast specimen.

RESULTS AND DISCUSSION

Flow table test

The results from the flow table tests, as depicted in Figures 7 and 8, provide valuable insight into the flow characteristics of the various mixtures. The mix M1 and M2 had flow values

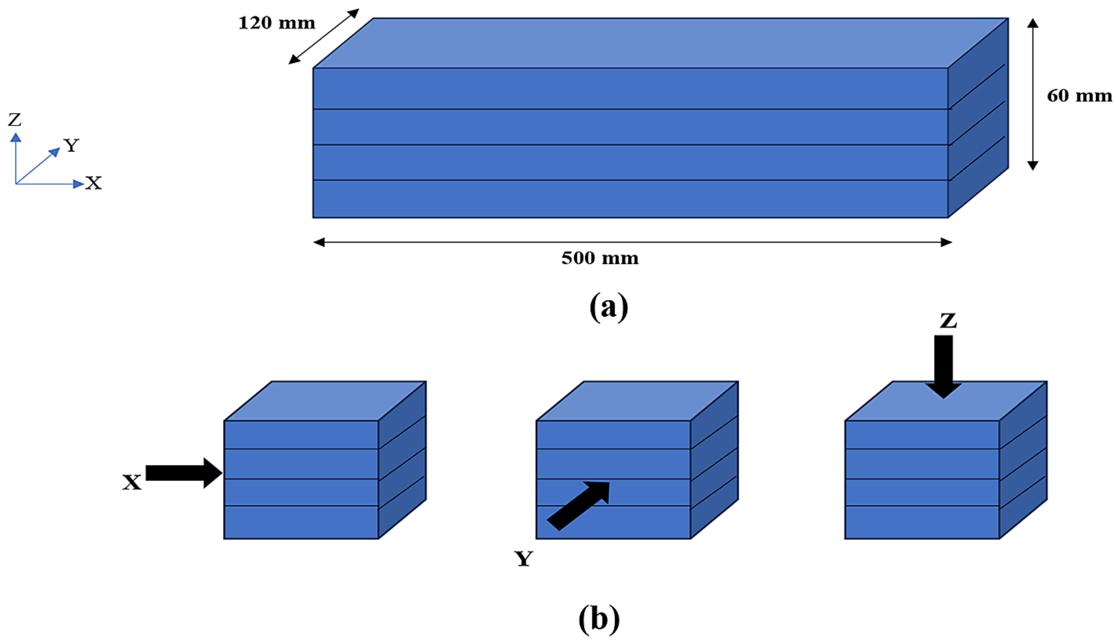

Figure 6. (a) Printed specimen (b) X, Y, and Z loading direction in the test specimens of 50 mm cube

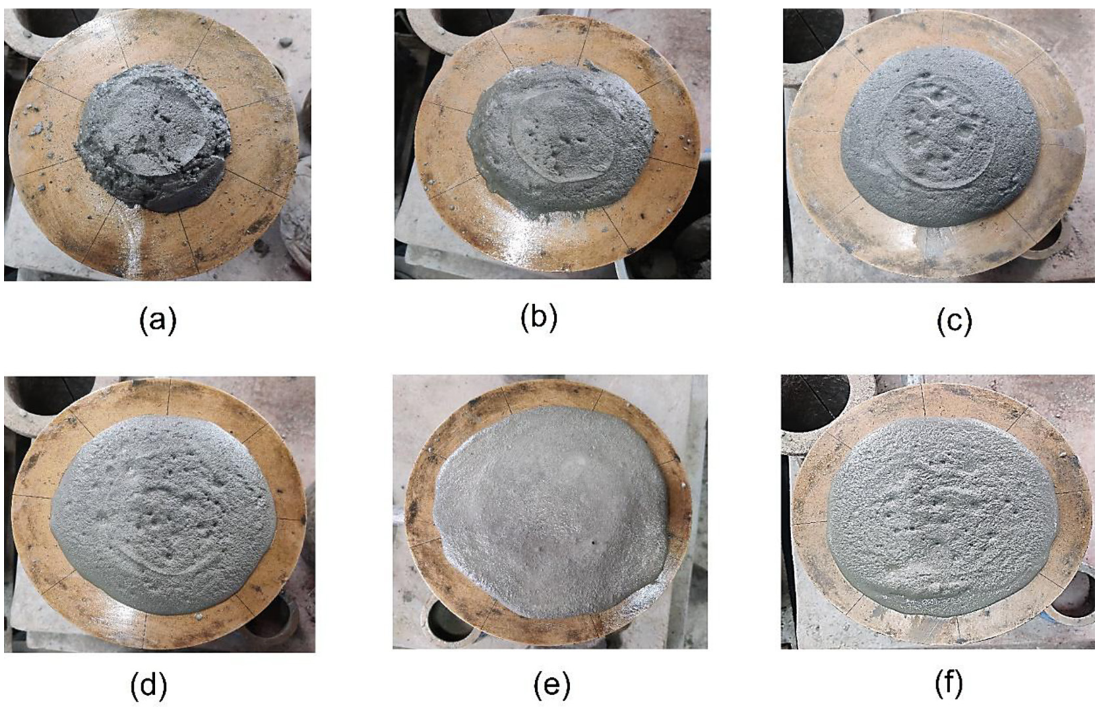

Figure 7. Flow table test (a) M1 (b) M2 (c) M3 (d) M4 (e) M5 (f) M6

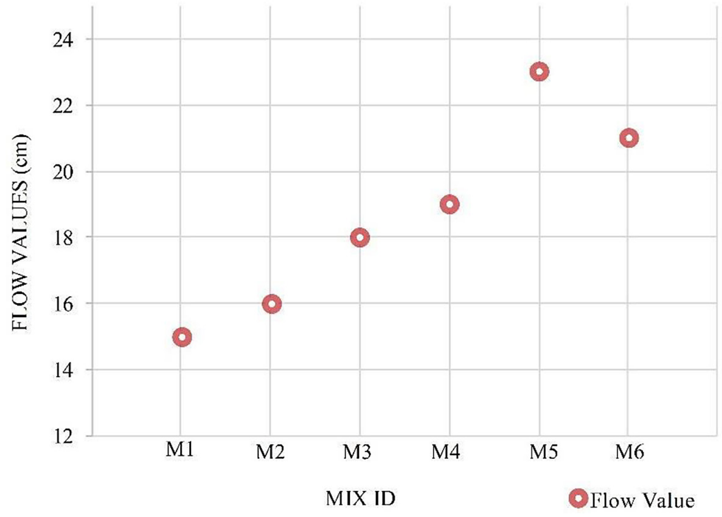

Figure 8. Flow value of mixes

of 15 and 16 cm, respectively, as seen in Figure 7a, 7b which falls outside the acceptable range commonly cited in the literature for printable mixes, which is typically between 19 and 21 cm [8, 30]. This may not possess the ideal workability required for efficient printability, potentially affecting its application in additive manufacturing processes. Furthermore, the addition of a superplasticizer (SP) appears to have a significant impact on the flow behavior of the mixtures. As indicated by prior research [7, 10], incorporating a superplasticizer enhances the flowability by improving the dispersion of the mix components, resulting in a wider flow spread. The flow values of the mixtures M3 to M6, increases to a range of 18 to 23 cm as shown in Figure 7c to 7f on increasing the SP dosage from 0.3% to 0.4% with an interval of 0.05%. This range suggests that the incorporation

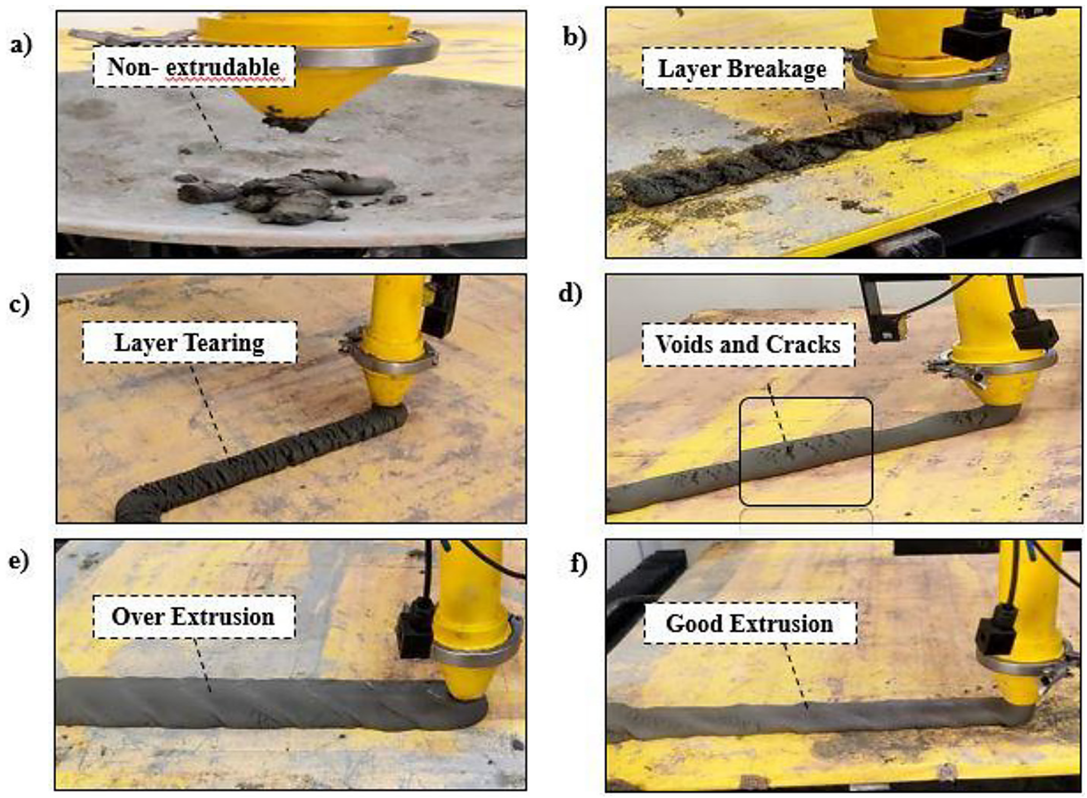

Figure 9. Extrudability test (a) M1 (b) M2 (c) M3 (d) M4 (e) M5 (f) M6

of SP within this dosage window can effectively bring the flowability of the mixtures within the target range for printable materials.

Extrudability test

A 500 mm length layer was simulated and extruded from the nozzle for concrete mixtures M1 to M6, as shown in Figure 9, to evaluate their extrudability and assess the presence of any extrusion defects. Mixture M1, with SP dosage of 0.2% and w/b ratio of 0.3 as depicted in Figure 9a, exhibited considerable stiffness and dryness, rendering it non-extrudable from the nozzle. Mixture M2, which incorporated a SP dosage of 0.25% and a water-to-binder (w/b) ratio of 0.3, showed layer breakage during extrusion, as illustrated in Figure 9b. This suggests that the mix is unsuitable for extrusion-based applications, likely due to insufficient fluidity and workability. Mixture M3, with an increased SP dosage of 0.3% and a w/b ratio of 0.3, demonstrated layer tearing during extrusion, as shown in Figure 9c. The tearing may result from a lack of consistency in the mix. Mixture M4, which contained 0.35% SP dosage and a w/b ratio of 0.3, was tested for extrudability, and the results showed the formation of voids and cracks in the printed layer after a certain distance, as seen in Figure 9d. These defects suggest that the mixture exhibited enhanced flowability, but inadequate bonding, causing the formation of voids and cracks during the deposition process [6]. Upon increasing the SP dosage to 0.4% with a w/b ratio of 0.3, as in Mixture M5, over-extrusion occurred, resulting in excessive flowability. This over-extrusion, depicted in Figure 9e, suggests that the mix was too fluid to maintain its structural form and poor shape retention during printing. The Mixture M6, with an SP dosage of 0.35% and a w/b ratio of 0.35, exhibited the better extrusion quality, as shown in Figure 9f. The printed layer was free from layer cracks and voids, indicating that this mix provided an optimal balance between flowability and consistency. The improved extrudability and shape retention of Mixture M6 suggest that a careful adjustment of SP dosage and w/b ratio can enhance the quality of the printed layers, leading to better shape extrusion.

Buildability test

Mixture M5 exhibited significant challenges in buildability, as evidenced by layer deformation, which can be attributed to the higher SP dosage used in the mix [10], as shown in Figure 10c. This mixture encountered issues of over-extrusion, resulting in excessive flowability that hindered the material's ability to maintain its shape during the printing process. As a consequence, the printed layers failed to adequately resist the pressure exerted by subsequent layers, leading to height deformation in the subsequent layers. This instability suggests that while the increased SP dosage may have improved the flowability of the mix, it also reduced its ability to retain structural integrity under the applied load, resulting in compromised

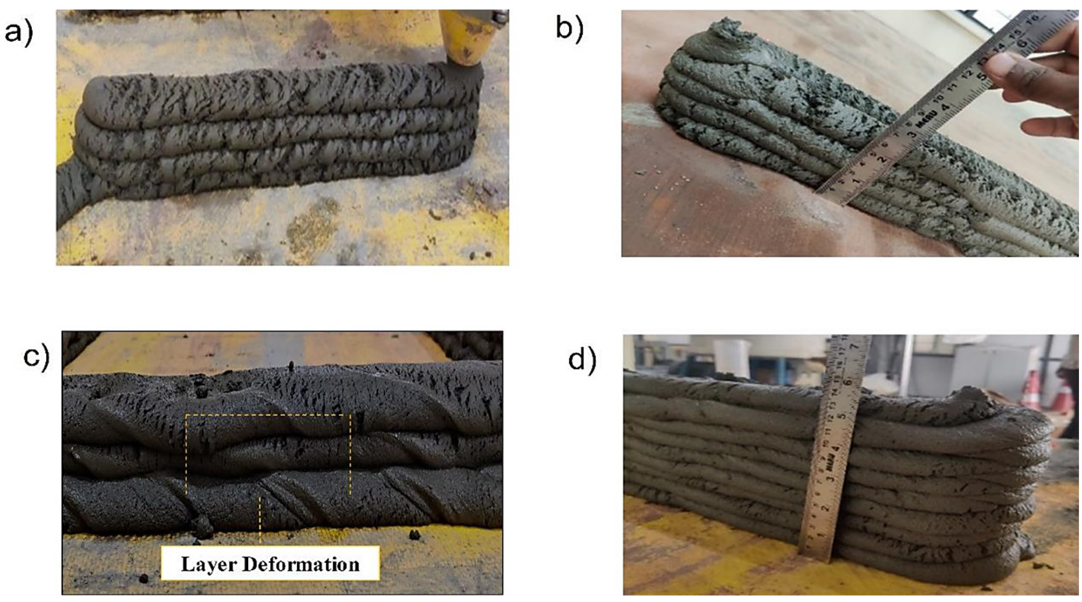

Figure 10. Buildability test, (a) M3, (b) M4, (c) M5, (d) M6

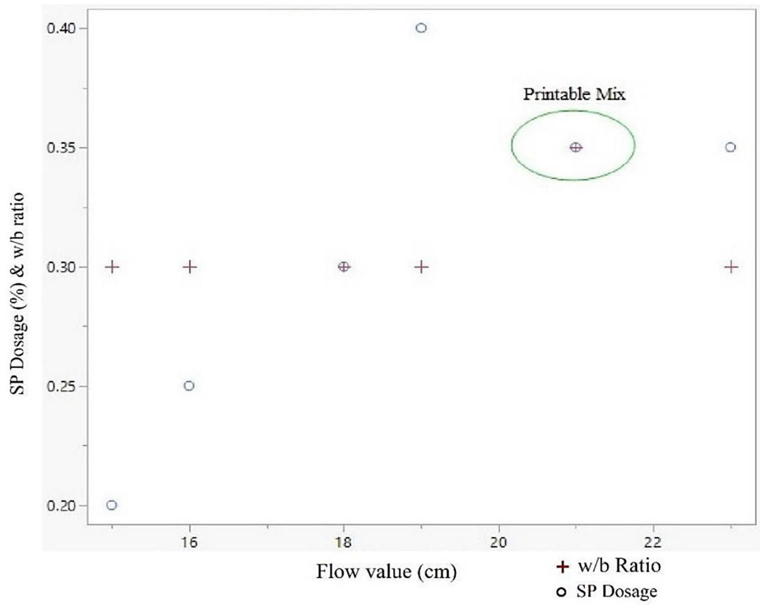

Figure 11. Printable mix with flow value, w/b ratio and SP dosage(%)

Table 3. Mixes and results of various SP dosage and w/b ratio

Mixes	Flow value (cm)	SP dosage (%)	w/b	Extrudability	Observation	Buildability
M1	15	0.20	0.30	Fail	Non extrudable	-
M2	16	0.25	0.30	Fail	Layer breakage	-
M3	18	0.30	0.30	Pass	Layer tearing	Pass
M4	19	0.35	0.30	Pass	Voids and cracks	Pass
M5	23	0.40	0.30	Pass	Over Extrusion	Fail
M6	21	0.35	0.35	Pass	Good Extrusion	Pass

shape retention. Despite the improved flowability and workability in mixtures M3 and M4, both mixtures still displayed layer printing imperfections, including tearing, void formation, and cracking, as illustrated in Figures 10a and 10b. The mixture M6 was tested by printing a rectangular shape with dimensions of 60 mm in width and 135 mm in height, consisting of 9 layers, each with a 15 mm layer height. Two adjacent filaments were printed, as shown in Figure 10d. The printed specimen was measured to verify the deformation and layer size, with results indicating that M6 demonstrated improved shape retention and structural stability compared to mix M3 and M4. These measurements provided an effective means of evaluating the impact of mix adjustments on the overall buildability of 3DCP.

Printable mix

From mix M1 to M6, varying experiments were carried out and observations are shown in Table 3. The printable material mix is attained by optimising SP dosage from 0.2% to 0.4%, w/b ratio from 0.3 to 0.35 and with the flow value ranges between 19 to 21 cm. Mix M6 obtained the desired printable mix with flow value 21 cm, SP dosage of 0.35% and w/b ratio of 0.35 as Figure 11 illustrates.

Compressive strength test

The compressive strength results for both cast and printed specimens are presented in Figure 12. Mix M6 was subjected to extrudability and buildability tests, both of which it passed successfully, indicating its suitability for use in 3DCP process. Following these preliminary assessments, compressive strength tests were conducted on the specimens to evaluate their mechanical performance.

The compressive strength of the printed specimens, measured along the X, Y, and Z directions, showed notable reductions when compared to the

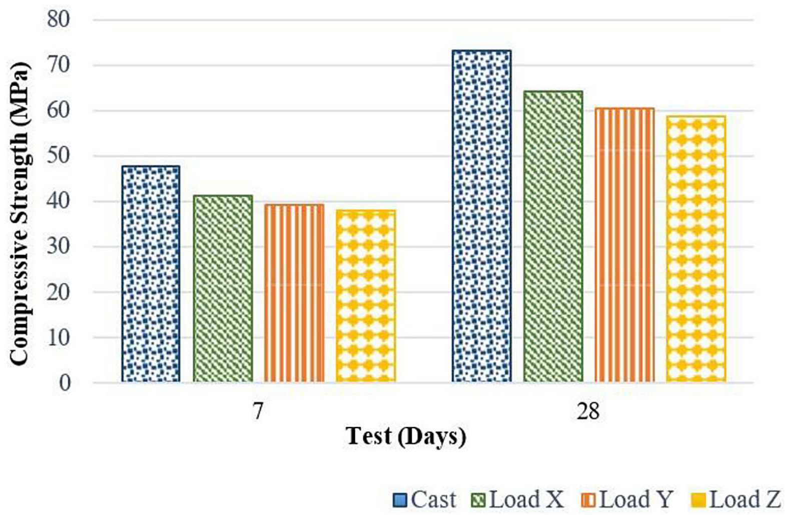

Figure 12. Compressive strength of the cast and printed specimens (Loaded along X, Y, and Z directions) at 7 and 28 days

cast specimens. Specifically, the strength in the loading along X-direction was reduced by 14.2%, in the Y-direction by 21.2%, and in the Z-direction by 24%, relative to the cast specimens. These reductions highlight the impact of the 3D printing process on the material's overall structural integrity. The strength loss in the printed specimens was observed to be more pronounced along the Y and Z directions as compared to the X-direction, and also relative to the cast specimens. This directional variance in strength loss can likely be attributed to the layer-by-layer deposition process inherent in additive manufacturing [40, 41].

CONCLUSIONS

In this study, the utilization of marble powder waste as supplementary cementitious material is an effective way to utilize the enormous volumes dumped in landfills and open places worldwide. The experimental results demonstrate the flowability, extrudability, and buildability with the incorporation of MPW, varying the SP dosage and w/b ratio in the development of mix proportion and methodology for 3D printable mix indicating the following key findings:

- The mix M6 met the requirements with 10% replacement of cement to marble powder, superplasticizer dosage of 0.35% and a water-to-binder ratio of 0.35%, with the flow value of 21 cm providing a superior printable mix that passes the extrusion test and buildability test which is suitable for 3DCP.
- Altering the dosage of SP from 0.2 to 0.4 in the mixture M1 to M6 enhances the workability of the material, facilitating improved extrusion for better print quality.
- A superplasticizer dosage of 0.4% in mix M4 leads to over extrusion, causing buildability issues due to excessive flow of 23 cm. Conversely, a superplasticizer dosage of 0.2% results in a very stiff mix that is non-extrudable and fails as a printable mixture.
- The increase in flowability with SP addition indicates its potential role in optimizing the workability of cementitious mixtures, a critical factor for ensuring successful extrusion in 3D printing applications. The findings suggest that the mix M5 is suitable for printing, the strategic inclusion of a superplasticizer can enhance its performance to meet the necessary criteria.
- Experimental results of the compressive strength test of printed specimen were 41.15, 64.1 MPa in loading along X direction, 39.2 and 60.4 MPa in loading along Y direction, 38 MPa, 58.6 MPa in loading along Z direction and cast specimens results 47.65, 73.2 MPa respectively at 7 and 28 days. In particular, the compressive strength of the printed specimens along the Y and Z directions was lower than that of the cast specimens as the alignment of the printed layers in these directions. Additionally, the anisotropic behaviour of 3D printing, where mechanical properties are direction-dependent due to the orientation of the layers, may contribute to the observed differences in strength along various axes.

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