

Article

Utilisation of Oil-Contaminated Sand in 3D-Printed Concrete: Rheological, Mechanical, and Microstructural Assessment

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Abstract

The growing demand for construction materials has intensified concerns regarding natural sand depletion and the accumulation of industrial waste. Among these wastes, oil-contaminated sand (OCS) generated from petroleum-related activities presents environmental challenges while also offering potential for beneficial reuse. Previous studies have reported that low OCS contents can enhance workability and improve mechanical properties, highlighting its potential as a sustainable construction material. This study investigates the feasibility of incorporating OCS as a partial replacement for natural sand in 3D concrete printing (3DCP). Three mixes containing 0%, 0.5%, and 1% OCS were evaluated in terms of flowability, setting behaviour, printability, rheological response, hydration behaviour, mechanical performance, anisotropic response, and microstructural characteristics. The results showed that OCS incorporation had only a limited influence on flowability, while both the initial and final setting times exhibited noticeable delays. The maximum printable layers reached from 19 for the control mixture to 22 and 26 layers for the 0.5% and 1% OCS mixes, respectively, accompanied by reduced settlement and structural deformation. Rheological analysis revealed higher static yield stress and structuration rates for the OCS-modified mixes, contributing to enhanced buildability and geometric stability. Hydration calorimetry showed comparable heat-flow behaviour between the control and OCS-modified mixes, suggesting only a limited influence of OCS on cement hydration kinetics. The incorporation of OCS improved the compressive strength of the printed mixes and reduced compressive anisotropy, while SEM observations revealed a denser and more homogeneous microstructure, particularly for the 0.5% OCS mix. Thus, the findings indicate that low-level incorporation of oil-contaminated sand is a viable strategy for improving the printability and performance of 3D-printed concrete, promoting the sustainable reuse of contaminated industrial waste.



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1. Introduction

Three-dimensional concrete printing (3DPC) is a rapidly developing additive manufacturing technology capable of fabricating structural components through layer-by-layer extrusion without the need for conventional formwork [1]. Compared with traditional

construction methods, 3DPC and prefabricated structures offer several advantages, including reduced material consumption, lower labour dependency, reduced construction waste, improved construction efficiency, and enhanced geometric flexibility [2–6]. Due to these advantages, 3DPC has attracted increasing attention as a sustainable construction technology with the potential to transform modern construction practices. Nevertheless, the global construction industry continues to consume vast quantities of natural resources, with concrete remaining the most widely used construction material worldwide. Global concrete production has increased nearly fourfold over the past three decades, reaching approximately 26 gigatonnes annually in 2020 [7,8]. The continuously increasing demand for construction materials has intensified concerns regarding natural resource depletion, greenhouse gas emissions, and construction-related waste generation. Therefore, the utilisation of industrial residues and waste-derived materials in cementitious systems has gained significant interest as a strategy to improve resource efficiency and reduce environmental impacts associated with conventional construction materials [9]. As a result, the incorporation of sustainable alternative materials into 3DPC systems has become an important research area for improving the environmental sustainability of digital construction technologies.

Successful extrusion-based 3DPC requires strict control of both fresh-state and hardened-state properties, including flowability, extrudability, buildability, shape retention, and interlayer bonding performance [10]. Unlike conventional cast concrete, 3D-printable cementitious mixes must possess highly controlled rheological behaviour to ensure continuous extrusion, adequate layer stability, and sufficient structural integrity during the printing process [11–13]. Among the various constituents of 3DPC, fine aggregate plays a critical role in controlling particle packing density, rheological behaviour, extrusion stability, and interlayer bonding characteristics [14]. Consequently, considerable research has focused on the utilisation of alternative fine aggregate materials in 3DPC to improve sustainability while maintaining acceptable printing performance. Zhou et al. [15] observed that recycled glass powder improved flowability owing to its smooth particle morphology, while Mim et al. [16] reported that incorporating copper heap leach residue as a fine aggregate replacement improved particle packing density and enhanced compressive strength at appropriate replacement levels. Similarly, researchers found that waste marble powder enhanced buildability and shape retention through filler effects [17], whereas studies on recycled sand demonstrated that aggregate grading significantly influenced extrusion quality and interlayer bonding performance [18,19]. These findings indicate that the characteristics of alternative fine aggregates can substantially affect the fresh and hardened properties of 3DPC. Therefore, a comprehensive evaluation of newly proposed alternative fine aggregate materials is essential before their application in 3DPC systems.

Among various industrial waste materials, oil-contaminated sand has attracted increasing attention due to the substantial quantities generated during oil extraction, transportation, and refining activities [20,21]. Improper disposal of oil-contaminated sand can lead to severe environmental problems, including soil and groundwater contamination [22]. Therefore, reutilising oil-contaminated sand in cementitious composites presents a potential solution for both waste management and natural resource conservation [20]. Abousnina et al. [23] observed that low levels of oil-contaminated sand enhanced the fresh-state behaviour of conventional concrete, resulting in improved workability and cohesiveness due to the lubricating effect of residual hydrocarbons. This lubricating effect may also offer potential advantages for 3DPC, where controlled material flow during pumping and continuous extrusion are essential for successful layer-by-layer deposition. Furthermore, the modified particle interactions caused by the oil film may influence the rheological behaviour, structural build-up, and interlayer bonding of printable mixtures, which collectively govern buildability, shape retention, and the mechanical integrity of printed

structures. Furthermore, Almutairi [24] investigated the use of Kuwaiti oil-contaminated sand as a fine aggregate substitute and found that oil contamination had no significant adverse effect on the compressive strength of concrete, although the water absorption of the mixes increased to approximately three times that of the control. However, excessive incorporation of oil-contaminated sand may adversely affect cement hydration, interfacial bonding, and mechanical performance because the residual oil can interfere with bond development between the cement paste and aggregate particles [25,26]. Since residual oil can significantly alter the rheological behaviour and surface interaction of cementitious mixes, the incorporation of oil-contaminated sand may also influence the printability, extrusion stability, layer deformation, and interlayer bonding characteristics of 3DPC.

Despite several studies investigating the application of oil-contaminated sand in conventional concrete, limited research has explored its utilisation as an alternative fine aggregate material in 3DPC mixes. Unlike conventionally cast concrete, the successful performance of 3DPC depends not only on hardened mechanical properties but also on fresh-state characteristics such as rheological behaviour, printability, buildability, and interlayer bonding. Consequently, the influence of OCS on these critical performance requirements remains insufficiently understood. Previous work by Abousnina and Allister [27] demonstrated that low levels of oil contamination can improve the mechanical performance of conventional concrete, with 0.5% OCS increasing compressive strength by 7.49% and 1.0% OCS increasing compressive strength by 24.4% compared with uncontaminated concrete. Based on these promising findings, OCS contamination levels of 0.5% and 1.0% were selected in the present study to investigate whether similar benefits could be achieved in 3DPC.

Accordingly, this study investigates the feasibility of incorporating oil-contaminated sand as a partial replacement of natural sand in 3DPC mixes. Replacement levels of 0.5% and 1% were considered to evaluate their influence on flowability, setting behaviour, buildability, shape retention ability, extrudability, rheological response, hydration behaviour, mechanical performance, and microstructural characteristics. Unlike previous studies that focused primarily on conventional cast concrete, this study provides an integrated assessment of the influence of oil-contaminated sand on the fresh-state, rheological, hydration, mechanical, and microstructural properties of 3DPC mixes. The findings are expected to contribute to the sustainable utilisation of contaminated industrial waste while expanding the current understanding of alternative fine aggregate materials for 3DPC applications.

2. Materials and Experimental Methods

2.1. Raw Materials

Ordinary Portland Cement (OPC) supplied by Cockburn Cement Pty Ltd., Perth, Western Australia, Australia, fly ash (FA) supplied by Cement Australia Pty Ltd., Darra, Queensland, Australia, and silica fume (SF) supplied by Xypex Australia Pty Ltd., Perth, Western Australia, Australia were used as the cementitious materials for preparing the 3DPC mixes. Fly ash and silica fume were incorporated as supplementary cementitious materials to improve the particle packing density, rheological behaviour, and mechanical performance while reducing the cement consumption. Table 1 summarises the chemical compositions of the OPC, FA, and SF adopted in the present work.

Natural sand (NS) was used as the fine aggregate supplied by Holcim Australia Pty Ltd., Perth, Western Australia, Australia in the control mix, while oil-contaminated sand (OCS) was prepared and utilised in the modified mixes to evaluate the feasibility of incorporating contaminated aggregates into 3DPC. The selected motorcycle fork oil (Fork W 2.5 MOTUL, Aubervilliers, France) possessed a density of 0.827 kg/L and a viscosity of 6.74 mm²/s at 40 °C, which are comparable to those of light crude oil (density of 0.825 kg/L

and viscosity of 5.96 mm²/s) [28]. Therefore, the motorcycle oil was adopted as a crude oil simulant to produce controlled contamination under laboratory conditions. The OCS was prepared by thoroughly blending the required quantity of motorcycle oil with dry natural sand to achieve uniform coating of the sand particles. Three aggregate conditions were considered: uncontaminated natural sand (0% oil contamination), sand contaminated with 0.5% oil by weight of sand, and sand contaminated with 1.0% oil by weight of sand. The selected contamination levels were based on previous studies reporting that low contamination levels may improve workability without significantly compromising mechanical performance [23].

Table 1. Chemical compositions of cementitious materials [16].

Compositions	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	P ₂ O ₅	Na ₂ O	LOI
Cement (OPC)	20.70	5.70	2.90	63.1	1.3	3.3	0.40	-	0.30	1.81
Fly ash (FA)	55.90	26.94	6.59	4.25	1.51	0.20	0.76	0.50	0.31	1.51
Silica fume (SF)	94.58	0.50	0.06	1.54	0.41	0.14	0.64	0.11	0.23	1.79

Particle size distribution (PSD) curves of the sand used in this study and a sample of sand collected from the site where oil contamination happened in Libya are shown in Figure 1, which shows close particle distributions.

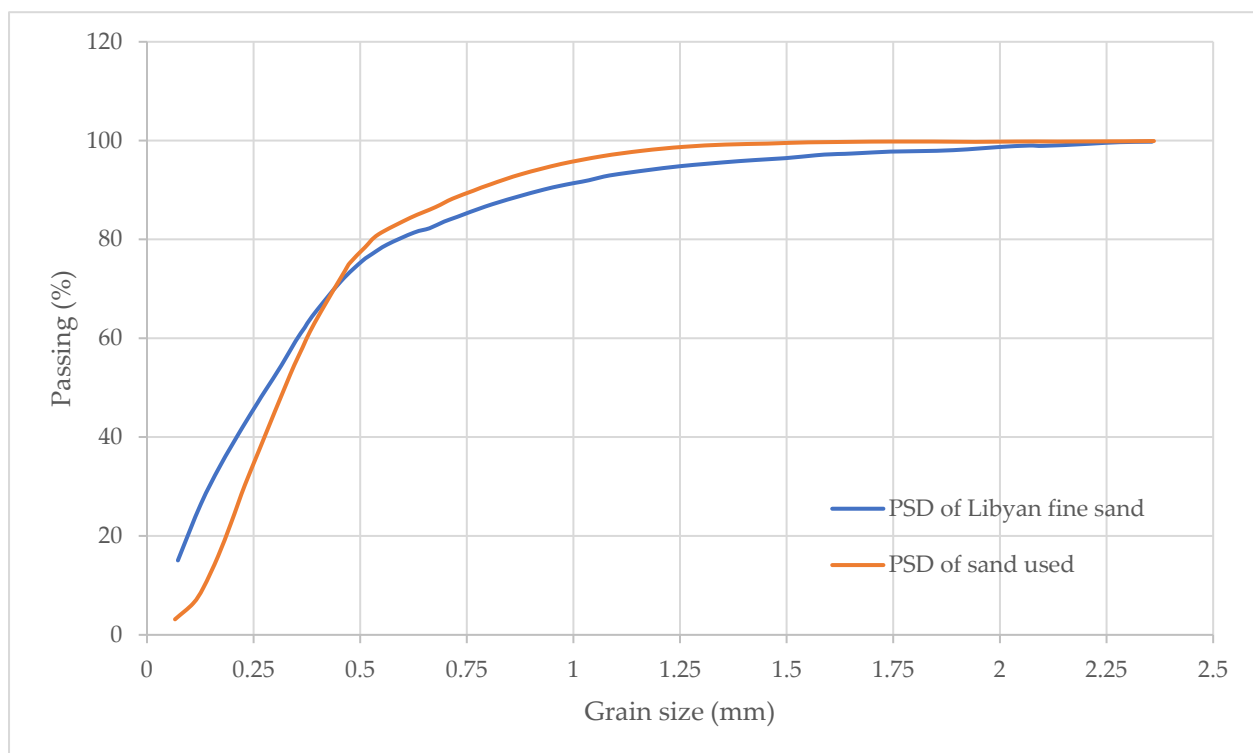


Figure 1. Particle size distribution (PSD) curves of sand used in this study and Libyan fine sand [29].

A polycarboxylate-based high-range water-reducing admixture (HRWRA), set accelerator (SA), and viscosity-modifying admixture (VMA) were incorporated to achieve the rheological properties required for 3DPC. The HRWRA was used to improve flowability, while the SA and VMA were employed to enhance the buildability and dimensional stability of the deposited layers during the printing process.

2.2. Mix Design, Mixing Procedure and 3D Printing System

The mix proportions adopted in this study are presented in Table 2. The binder system consisted of OPC, FA, and SF at proportions of 70%, 20%, and 10%, respectively. The mix design was adopted from previously validated 3DPC mixes reported in the literature [30,31], which achieved satisfactory flowability, extrudability, and buildability. The aggregate-to-binder ratio and water-to-binder ratio were maintained at 0.66 and 0.35, respectively. Three mixes were investigated, including a control mix without oil contamination and two mixes incorporating OCS contamination levels of 0.5% and 1.0%. The selected mixes enabled evaluation of the influence of oil contamination on the fresh behaviour, mechanical characteristics, and microstructural properties of 3DPC.

Table 2. Mix proportions (kg/m³) of the investigated 3DPC containing OCS.

Mix ID	OPC	Fly Ash	Silica Fume	Sand	Fork Oil	Water	HRWRA (%) of Binder	SA (%) of Binder	VMA (%) of Binder
Control	863	246	124	821	0	432	0.1	0.01	0.01
0.5% Mix	863	246	124	821	4.1	432	0.05	0.01	0.01
1% Mix	863	246	124	821	8.2	432	0.05	0.01	0.01

Note: VMA—viscosity modifying agent, SA—set accelerator, HRWRA—high-range water-reducing admixture.

The mixes were prepared using a 10 L Hobart mixer, Sydney, Australia. OPC, FA, SF, sand, and VMA were initially dry-mixed for 3 min. Subsequently, a premixed solution containing water, HRWRA, and SA was gradually introduced and mixed until a homogeneous mix was obtained. The fresh mixes were then subjected to flowability testing before being transferred to the printing system.

A laboratory-scale gantry-type 3D printer was used for specimen fabrication. The printer consisted of a rigid frame measuring 1.4 m × 1.2 m × 1.2 m and was equipped with a circular nozzle with a diameter of 20 mm. The layer height was maintained at 8 mm, while the average filament width was approximately 19.9 mm. A constant printing speed of 50 mm/s was adopted throughout the printing process to ensure stable material deposition and adequate interlayer bonding. Following printing, the specimens were cured under laboratory conditions until the designated testing age.

2.3. Experimental Programme

2.3.1. Fresh-State Properties

The fresh-state performance of the mixes was characterised through flowability, setting behaviour, buildability, extrudability, and shape-retention tests to assess their suitability for 3DPC applications.

The flowability characteristics were determined using the flow table test as per ASTM C1437 [32] standard, as illustrated in Figure 2a. The spread diameter was measured following 25 successive drops on the flow table, and the average value obtained from three replicate tests was reported. The setting behaviour was assessed using a Vicat apparatus following ASTM C191 [33], from which the setting times were determined for assessing the available printing window.

The shape-retention ability was evaluated using a modified mini-slump procedure adapted from ASTM C1437 and the methodology proposed by Nematollahi et al. [34], as illustrated in Figure 2b. Fresh mixes were placed into a truncated cone mould and allowed to rest for 1 min before carefully removing the mould. Subsequently, a static load of 600 g was applied through a steel plate for an additional minute to induce deformation under a controlled loading condition. The deformation of the specimen was quantified by measuring two orthogonal directions, together with the retained specimen height. Lower

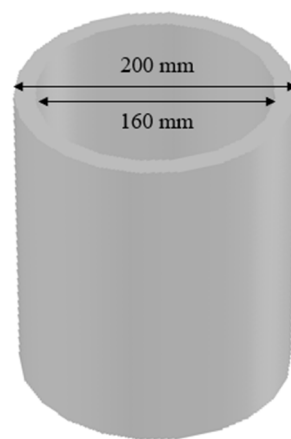
lateral deformation and greater retained height were considered indicative of superior shape-retention ability.



(a) Slump flow test.



(b) Shape retention ability test.



(c) Circular model.



(d) Printed circular structure.

Figure 2. Experimental procedures used for evaluating the fresh-state properties of the mixes: (a) flow table test, (b) shape-retention ability test, (c) circular model for buildability and extrudability test, (d) printed circular structure.

Buildability was assessed following the methodology adopted in previous studies [16,35,36], wherein the mixes were continuously extruded to form circular structures with a diameter of 200 mm until structural instability or collapse occurred, as shown in Figure 2c,d. Each deposited layer possessed a nominal width and height of 20 mm and 10 mm, respectively. The buildability performance was assessed based on the maximum achievable printing height and the geometric stability of the printed specimen. To further quantify the structural stability of the deposited layers, the deformation rate (D) was calculated according to Equation (1), following the approach adopted in previous studies [37,38]:

$$D = \left[\frac{|l - l_0|}{3l} + \frac{|d - d_0|}{3d} + \frac{|h - h_0|}{3h} \right] \times 100\% \quad (1)$$

where D denotes the structural deformation rate; l_0 , d_0 and h_0 represent the designed length, width, and height of the printed specimen, respectively; and l , d and h correspond to the measured length, width, and height immediately after printing, respectively.

The rheological behaviour of the investigated mixes was evaluated using a Kinexus dynamic rheometer manufactured by Selb, Germany equipped with a four-bladed vane geometry. All rheological measurements were performed at a laboratory temperature of

20 °C. Immediately after mixing, the fresh material was transferred into the rheometer measuring cup, and testing commenced immediately thereafter. A thixotropy and structuration protocol was adopted to assess the evolution of static yield stress, thixotropic behaviour, and structural build-up characteristics of the fresh mixes. The test consisted of 12 consecutive measurement cycles separated by progressively increasing resting periods. During each cycle, a constant shear rate of 1 s^{-1} was applied for 60 s, and the corresponding shear stress was recorded at 5 s intervals. The resting periods between successive cycles were 10, 20, 30, 40, 50, 60, 90, 120, 1200, 2400, and 3600 s, resulting in a total testing duration of approximately 138 min. The apparent static stress for each cycle was determined from the peak shear stress recorded at the beginning of the shearing stage. The thixotropic index was calculated to evaluate the structural recovery behaviour of the mixes after repeated shearing, while the structuration rate, A_{thix} , was determined from the relationship between apparent static stress and elapsed resting time. These parameters were used to assess the ability of the mixes to rebuild internal structure during rest, which is critical for maintaining layer stability in 3DPC.

2.3.2. Mechanical Properties

The mechanical performance of the printed mixes was evaluated through compressive and flexural strength tests conducted after 28 days of curing. As illustrated in Figure 3, all specimens were extracted from L-shaped printed panels to ensure consistent specimen geometry and printing conditions. Owing to the geometry of the printed elements, specimen dimensions differed from those specified in the ASTM standards and were selected based on dimensions commonly adopted for the mechanical characterisation of 3DPC [16,39]. Three specimens were tested for each mix and loading direction, and the average value was reported.

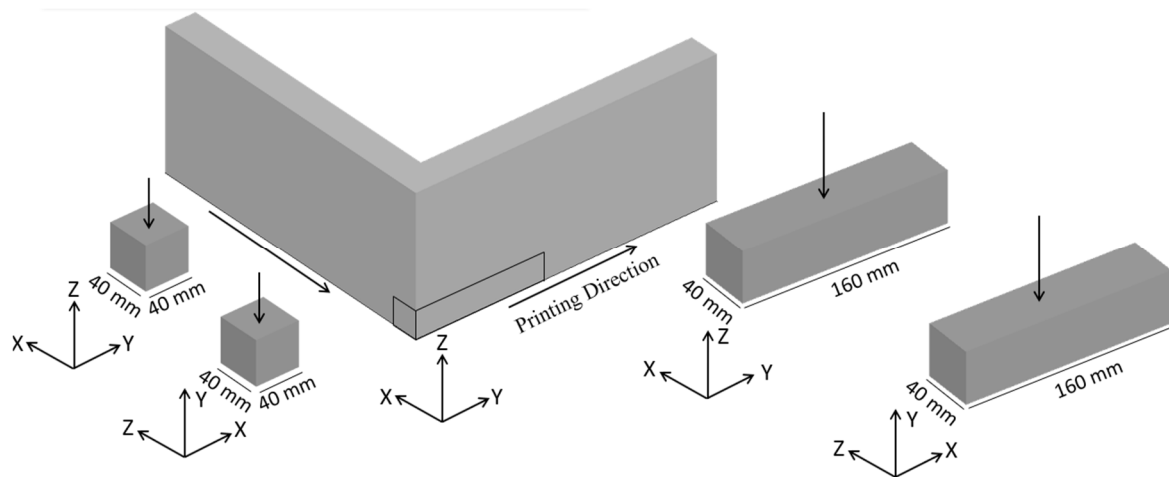


Figure 3. Extraction of compressive and flexural specimens from the L-shaped printed panel and corresponding loading directions adopted for mechanical testing.

For the compressive strength test, cube specimens measuring $40 \text{ mm} \times 40 \text{ mm} \times 40 \text{ mm}$ were extracted from L-shaped printed panels and tested following the loading procedure specified in ASTM C109/C109M [40] using a Shimadzu universal testing machine manufactured by Shimadzu Corporation, Kyoto, Japan. To investigate the influence of printing orientation on load-bearing behaviour, specimens were loaded in the Y-direction, parallel to the printing path, and the Z-direction, perpendicular to the deposited layers.

Flexural strength was assessed in beam specimens using $160 \text{ mm} \times 40 \text{ mm} \times 40 \text{ mm}$ extracted from the same printed panels and tested under three-point bending in accordance with the loading procedure specified in ASTM C78/C78M [41]. The specimens were tested

in both the Y- and Z-directions to evaluate the influence of interlayer interfaces and filament orientation on flexural performance.

The directional dependency of mechanical performance was quantified using the anisotropy degree [42], calculated according to Equation (2):

$$\text{Anisotropic degree} = \frac{\text{Max strength} - \text{Min strength}}{\text{Max strength}} \times 100\% \quad (2)$$

where the maximum and minimum strengths correspond to the highest and lowest strengths obtained from the investigated loading directions.

2.3.3. Microstructural Characterisation

Scanning electron microscopy (SEM) was employed to investigate the influence of oil contamination on aggregate morphology and matrix–aggregate interaction. Representative samples obtained from selected specimens were oven-dried at 50 °C for 24 h, coated with a conductive layer, and subsequently examined using a field-emission scanning electron microscope operating at an accelerating voltage of 15 kV. The obtained micrographs were analysed to identify morphological modifications induced by oil contamination and to provide microstructural interpretation of the observed fresh-state and mechanical performance.

3. Results and Discussion

3.1. Fresh Properties

3.1.1. Flowability and Setting Time

The flowability and setting behaviour of the 3DPC mixes incorporating different levels of OCS are presented in Figure 4. The control mix (C0) exhibited a flowability of 165 mm, which falls within the range of 150–200 mm commonly reported for 3DPC and considered suitable for achieving adequate extrusion while maintaining dimensional stability after deposition [31]. Upon incorporation of OCS, only marginal changes in flowability were observed. The mix containing 0.5% OCS exhibited a flowability of 163 mm, whereas the mix incorporating 1% OCS exhibited a slightly higher value of 167 mm. The maximum variation among the investigated mixes was less than 3%, indicating that the incorporation of OCS at the selected contamination levels exerted only a limited influence on flowability. The negligible variation in flowability suggests that the low levels of oil contamination adopted in this study were insufficient to significantly alter the water demand of the cementitious system. Although the OCS mixes contained a lower dosage of HRWRA than the control mix, comparable flowability values were achieved. This behaviour may be attributed to the formation of a thin oil film on the surface of the sand particles, which reduces interparticle friction and facilitates particle movement within the fresh matrix [43,44]. Consequently, the lubricating effect of the residual oil appears to compensate for the reduced superplasticizer dosage, resulting in flowability values comparable to those of the control mix [45]. This observation is further supported by the rheological results presented in Section 3.1.5, where the OCS-modified mixes exhibited comparable thixotropic behaviour despite the presence of residual hydrocarbons. These results indicate that incorporating OCS up to 1% maintained adequate flowability for 3DPC.

In contrast to the limited influence on flowability, OCS significantly affected the setting behaviour of the mixes. The control mix exhibited initial and final setting times of 92 min and 123 min, respectively. Incorporation of 0.5% OCS increased the initial and final setting times to 151 min and 170 min, respectively, while the corresponding values for the 1% OCS mix were 155 min and 172 min. Relative to the control mix, the initial setting time increased by approximately 64.1% and 68.5% for the 0.5% and 1% OCS mixes, respectively, whereas the final setting time increased by approximately 38.2% and 39.8%.

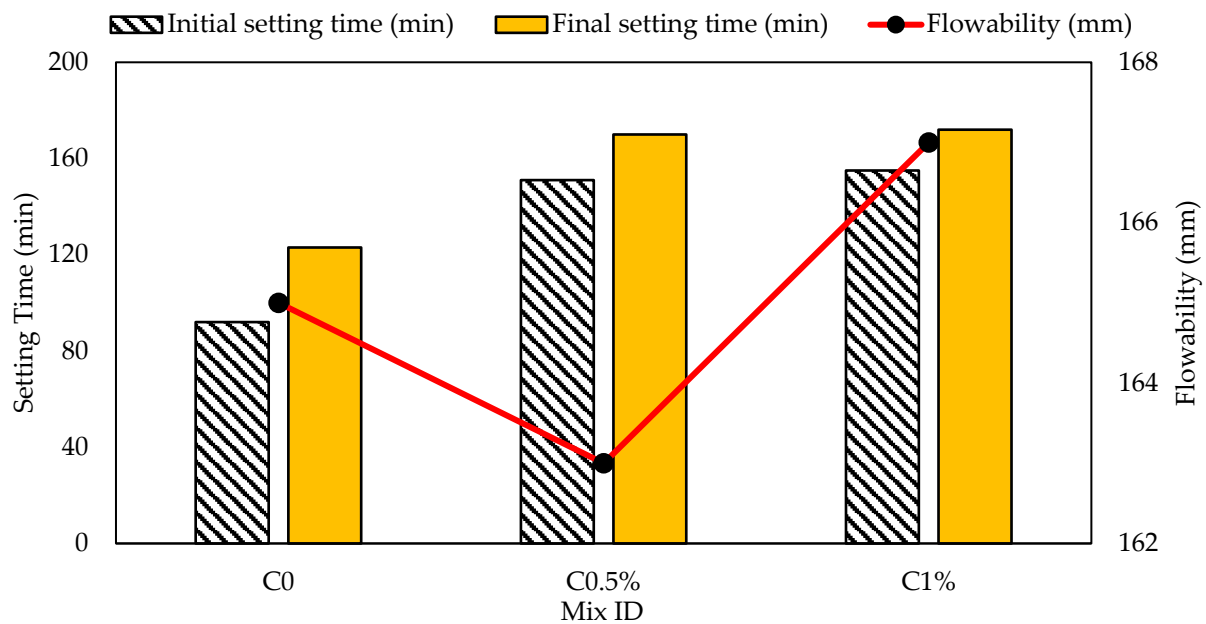


Figure 4. Effect of OCS incorporation on the flowability, initial setting time, and final setting time of 3DPC mixes.

The pronounced increase in setting time indicates that oil contamination primarily influences the hydration kinetics rather than the flow characteristics of the fresh mixes [46]. The residual hydrocarbons coating the aggregate particles may form a physical barrier that restricts direct contact between water, cement particles, and aggregate surfaces, thereby reducing the rate of dissolution of cement phases and delaying the formation of hydration products [25]. Furthermore, the hydrophobic nature of the hydrocarbon film may hinder moisture transport within the cementitious matrix and retard the development of a continuous hydration network [47]. Similar observations have been reported by Abousnina et al. [25], who observed that hydrocarbon residues associated with oil-contaminated aggregates interfered with cement hydration and prolonged the setting process. Despite the delayed setting behaviour, the rheological results demonstrated that the OCS-modified mixes developed higher static yield stresses and substantially greater structuration rates than the control mix. This suggests that the enhanced fresh-state stability of the OCS mixes was governed primarily by accelerated structural build-up of the particle network rather than hydration-induced stiffening. The prolonged setting times may increase the available printing window and facilitate material deposition during printing operations. However, excessive retardation of hydration may adversely affect the rate of early-age strength development. Therefore, although the incorporation of OCS had a negligible effect on flowability, its influence on hydration kinetics and setting behaviour should be carefully considered when designing 3DPC.

3.1.2. Buildability

Figure 5 presents the maximum printable layers achieved by the investigated mixes, while representative printed cylindrical structures obtained during the buildability test are shown in Figure 6. As shown in Figure 5, the maximum achievable printing height increased progressively with increasing OCS content. The control mix (C0) achieved a maximum of 19 printed layers, whereas the mixes incorporating 0.5% and 1% OCS achieved 22 and 26 layers, respectively. Compared with the control mix, the maximum printable layers increased by approximately 15.8% and 36.8% for the 0.5% and 1% OCS mixes, respectively. The improved ability of the OCS-modified mixes to sustain a greater number of deposited layers without collapse indicates enhanced resistance to the accumulation

of vertical stresses generated during layer-by-layer deposition. Similar observations have been reported in previous studies, where mixes exhibiting enhanced structural build-up and green-state stability achieved greater printable heights and improved buildability performance [39,48].

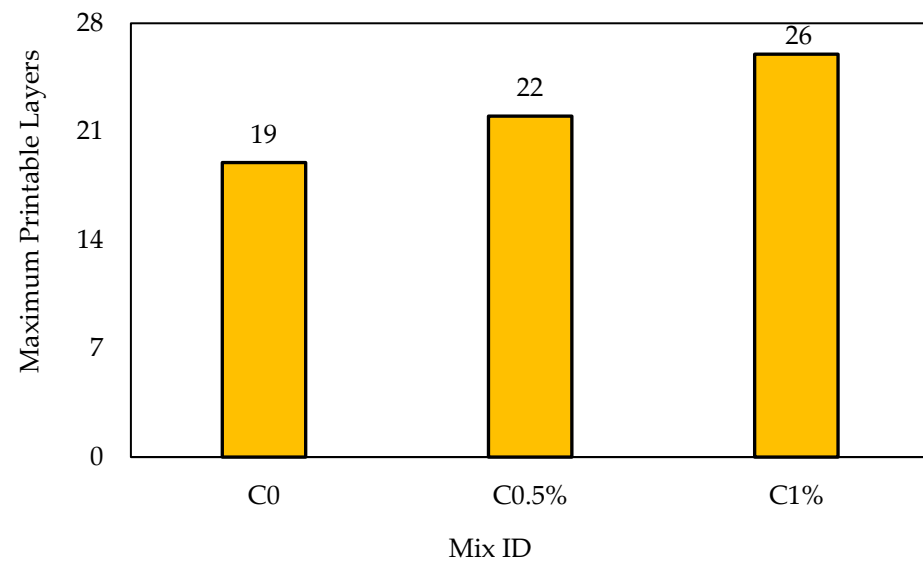


Figure 5. Maximum printable layers achieved by 3DPC mixes incorporating different levels of OCS.



Figure 6. Representative printed cylindrical structures obtained during the buildability assessment.

The printed cylindrical structures shown in Figure 6 further demonstrate the influence of OCS on the buildability performance of the mixes. Compared with the control mix, the OCS-modified mixes maintained taller printed geometries with reduced visible deformation and improved shape stability. This behaviour indicates that the fresh mixes containing OCS possessed greater resistance to plastic deformation during printing. The improved shape stability may be associated with modifications in interparticle interactions induced by the residual hydrocarbons coating the aggregate surfaces. These modifications may facilitate the development of a more coherent particle network with enhanced structural build-up, as reflected by the higher static yield stress and structuration rate obtained from the rheological tests. Consequently, the fresh matrix was better able to resist deformation under the self-weight of the deposited layers, resulting in improved buildability and geometric stability during printing.

Figure 7 presents the settlement behaviour of the printed specimens immediately after printing and after hardening. The control mix exhibited a height reduction from 195 mm to 193 mm after hardening, corresponding to a settlement of approximately 1.03%. Similarly, the 0.5% OCS mix showed a reduction from 235 mm to 234 mm, while the 1% OCS mix exhibited a reduction from 265 mm to 263 mm, corresponding to settlements of approximately 0.43% and 0.75%, respectively. Although all mixes exhibited relatively small reductions in height, the OCS-modified mixes maintained substantially greater retained heights after hardening. The lower settlement values indicate an enhanced ability of the fresh material to resist self-weight-induced deformation during the early stages of hardening. This behaviour suggests that incorporation of OCS improved the stability of the deposited filaments and reduced their susceptibility to progressive compression under the cumulative stresses imposed during layer-by-layer deposition.

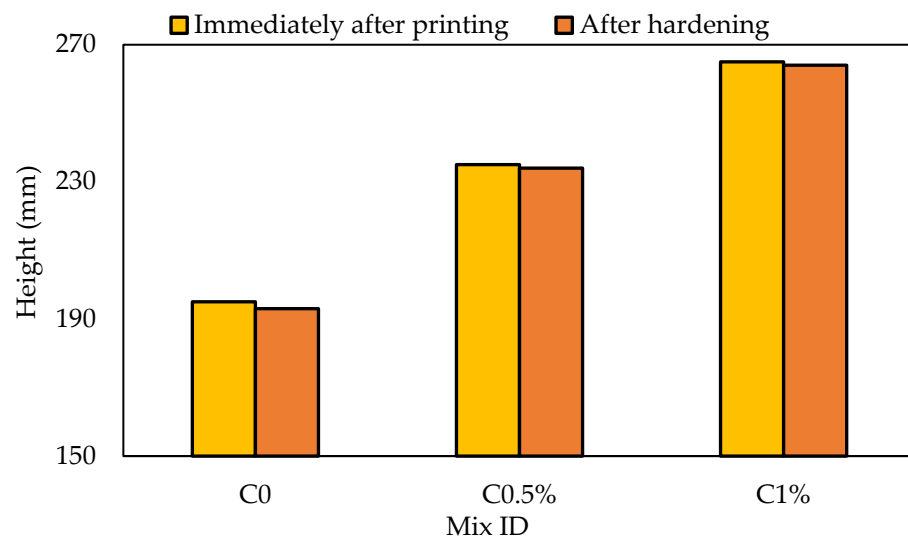


Figure 7. Comparison of layer heights immediately after printing and after hardening for 3DPC mixes containing OCS.

Further evidence of the influence of OCS on buildability is provided by the structural deformation rate (D as defined by Equation (1)) results presented in Figure 8. The structural deformation rate decreased from 11.33% for the control mix to 10.54% for the 0.5% OCS mix and was further reduced to 5.71% for the 1% OCS mix. Relative to the control mix, incorporation of 1% OCS reduced the structural deformation by approximately 49.6%, demonstrating a substantial improvement in geometric stability. The reduction in deformation rate indicates that the OCS-modified mixes possessed a greater ability to retain their intended geometry under the stresses generated during continuous layer deposition. The enhanced deformation resistance may be associated with the development of a stronger internal structure within the fresh matrix, enabling the deposited layers to better withstand geometric distortion and maintain dimensional stability throughout the printing process.

3.1.3. Shape Retention Ability

The shape-retention ability was assessed by determining the spread diameter of the mixes under a static loading condition, with the results presented in Figure 9. As shown in the figure, the control mix (C0) exhibited a spread diameter of 96 mm, whereas both the 0.5% and 1% OCS mixes exhibited slightly higher spread diameters of 99 mm. The spread diameter is approximately 3.1% higher than that of the control mix, indicating a marginal increase in lateral deformation under the applied load. The slightly larger spread diameters observed for the OCS-modified mixes may be attributed to the presence of residual hydrocarbons coating the aggregate particles, which can reduce interparticle

friction and facilitate particle movement within the fresh matrix [49]. Consequently, the mixes exhibited a slightly greater tendency to deform laterally when subjected to the applied load. Similar behaviour has been reported in oil-contaminated cementitious systems, where hydrocarbon residues influenced the fresh-state deformation characteristics of cement-based materials [25,50].

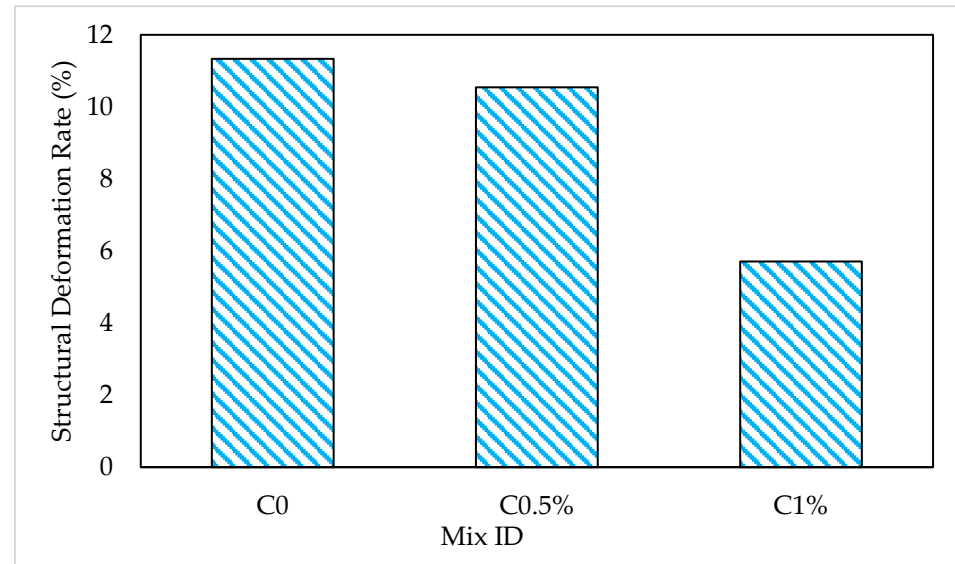


Figure 8. Structural deformation rate of 3DPC mixes incorporating different levels of OCS.

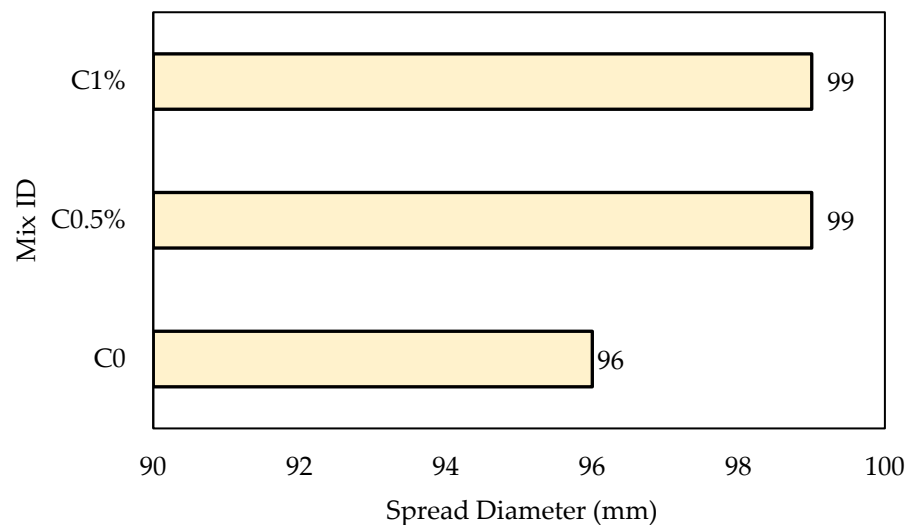


Figure 9. Shape-retention performance of 3DPC mixes incorporating different levels of OCS.

Despite the slight increase in spread diameter, the overall variation among the investigated mixes remained relatively small, indicating that incorporation of OCS at the selected contamination levels exerted only a limited influence on shape-retention ability. This observation is consistent with the flowability results presented in Section 3.1.1, where only marginal differences in flowability were observed following OCS incorporation. Furthermore, although the OCS-modified mixes exhibited slightly greater lateral deformation under static loading, they simultaneously demonstrated improved buildability and greater maximum printable heights. These results suggest that the minor increase in spread diameter was insufficient to adversely affect the overall printing performance of the mixes. The mechanisms governing this behaviour are further clarified through the rheological analysis presented in Section 3.1.5.

3.1.4. Extrudability

Figure 10 presents the extrudability performance of the investigated mixes based on visual assessment of the printed filaments. The control mix (C0) exhibited smooth, continuous, and well-defined extruded layers with no visible discontinuities, indicating stable material flow through the nozzle during the printing process. In contrast, the mix containing 0.5% OCS exhibited visible surface cracks and small voids along the extruded filaments, suggesting a reduction in extrusion quality compared with the control mix. However, increasing the OCS content to 1% resulted in noticeably smoother and more continuous filaments, with a substantial reduction in the number and severity of surface defects. Although minor cracks remained visible, the overall filament quality was markedly improved compared with the 0.5% OCS mix. These observations indicate that the incorporation of OCS influenced the extrusion behaviour of the mixes, with the 0.5% OCS mix exhibiting reduced extrusion stability, whereas increasing the OCS content to 1% improved filament continuity and surface quality.



Figure 10. Extrudability assessment of 3DPC mixes incorporating different levels of OCS.

The observed differences in extrusion quality may be attributed to changes in particle interaction and flow characteristics within the fresh matrix, which may also have been influenced by the reduced HRWRA dosage used in the OCS-modified mixes compared with the control mixture. However, the different extrusion behaviours observed between the 0.5% and 1% OCS mixes are more likely associated with differences in the continuity and thickness of the residual oil film coating the aggregate particles. At the lower OCS content (0.5%), the residual oil film was likely relatively thin and discontinuous, which may have resulted in inconsistent lubrication and localised variations in interparticle friction during extrusion. These localised variations may have disrupted particle flow through the nozzle, leading to the formation of surface cracks and small voids in the extruded filaments. In contrast, increasing the OCS content to 1% likely produced a thicker and more continuous oil film around the aggregate particles, providing more uniform lubrication throughout the fresh mixture. This more consistent lubrication may have reduced interparticle friction and facilitated smoother particle movement through the nozzle, resulting in improved filament continuity and surface quality.

3.1.5. Rheology

The rheological responses of the investigated mixes are presented in Figures 11–14. The incorporation of OCS significantly influenced the structural build-up characteristics of the fresh mixes, as evidenced by the higher static shear stresses and increased structuration rates observed for the OCS-modified mixes.

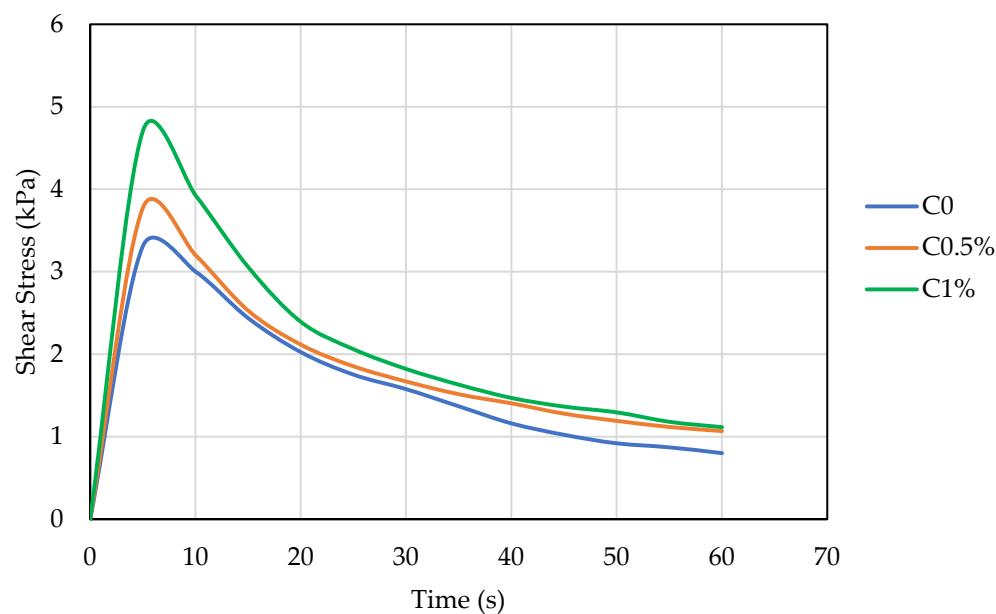


Figure 11. Evolution of shear stress with time under a constant shear rate of 1 s^{-1} for 3DPC mixes incorporating different levels of OCS.

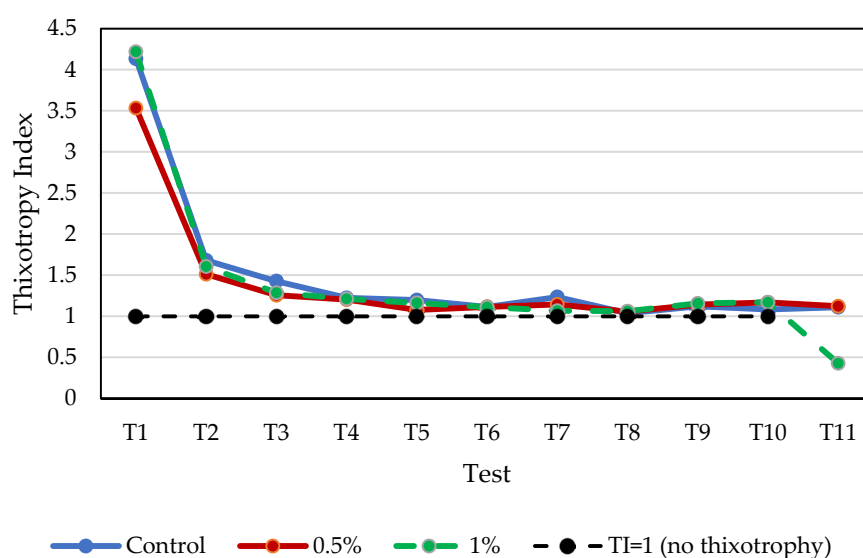


Figure 12. Thixotropic index of 3DPC mixes incorporating different levels of OCS.

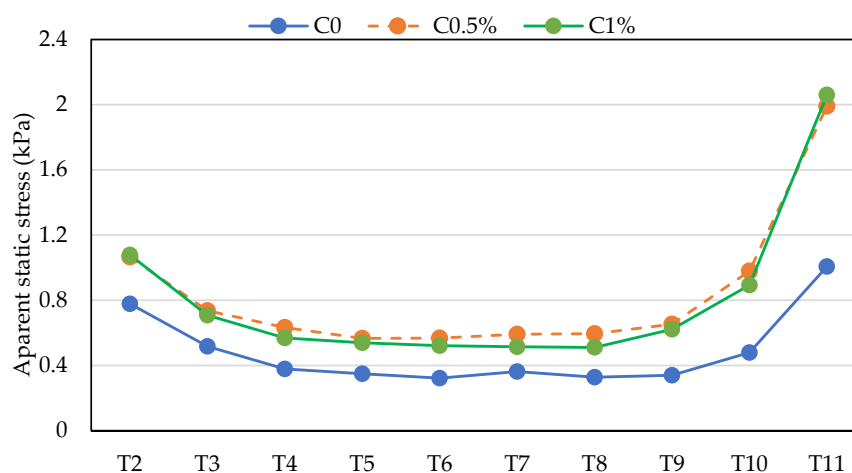


Figure 13. Structuration curve for 3DPC mixes incorporating different levels of OCS.

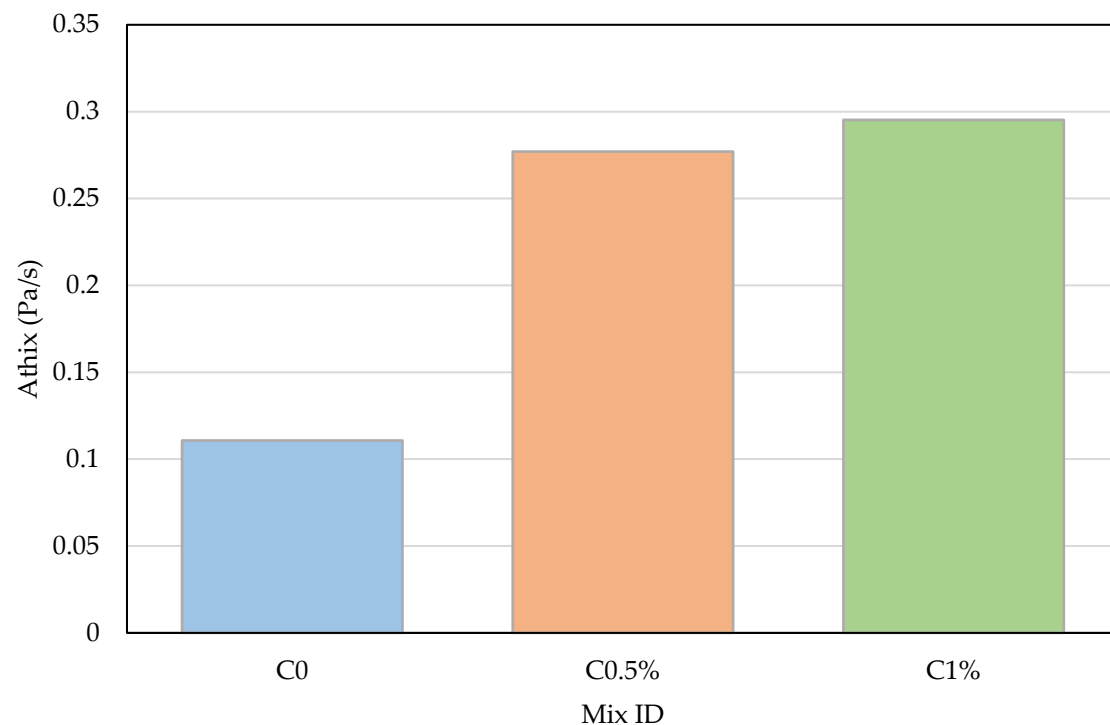


Figure 14. Comparison of the structuration rate (A_{thix}) of 3DPC mixes incorporating different levels of OCS.

Figure 11 presents the variation in shear stress with time under a constant shear rate of 1 s^{-1} . All mixes exhibited a characteristic rheological response comprising an initial increase in shear stress followed by a gradual reduction towards a quasi-equilibrium state. The initial peak represents the static yield stress necessary to break down the internal structure developed during the resting period and initiate flow [12]. Compared with the control mix, the OCS-modified mixtures exhibited consistently higher peak static stresses, increasing from approximately 3.4 kPa for the control mix to 3.9 kPa and 4.8 kPa for the 0.5% and 1% OCS mixes, respectively. The increase in static yield stress indicates that the incorporation of OCS enhanced the resistance of the fresh mixes to flow initiation. This behaviour may be attributed to modifications in interparticle interactions caused by the residual hydrocarbons coating the aggregate particles. The oil film may modify interparticle interactions and facilitate particle rearrangement within the fresh matrix, promoting the development of a more interconnected particle network capable of resisting external deformation. Consequently, greater shear stress was required to disrupt the internal structure and initiate material flow.

The thixotropic indices obtained from successive testing cycles are presented in Figure 12. Despite the increase in static yield stress, all mixes exhibited comparable thixotropic indices throughout the testing sequence. Following an initial reduction during the early cycles, the thixotropic index stabilised at values close to unity for all mixes, indicating similar structural recovery behaviour after repeated shearing. The absence of significant differences suggests that OCS did not substantially alter the reversible breakdown and rebuilding mechanisms of the cementitious network. Instead, the results indicate that OCS primarily affected the magnitude of the internal structural strength rather than the fundamental thixotropic nature of the mixes.

The evolution of apparent static stress with increasing resting time is shown in Figure 13. The OCS-modified mixes consistently exhibited higher apparent static stresses than the control mix throughout most of the testing period, indicating enhanced structural rebuilding during rest. The higher apparent static stresses suggest that the internal structure

of the OCS mixes developed greater rigidity during rest, thereby improving the ability of the fresh material to resist deformation under self-weight and externally applied loads. The difference between the control and OCS mixes became increasingly pronounced at longer resting periods, demonstrating that the beneficial influence of OCS on structural build-up persisted throughout the resting process.

To further quantify the structural build-up behaviour, the structuration rate (A_{thix}) was calculated and is presented in Figure 14. The A_{thix} value increased from 0.111 Pa/s for the control mix to 0.278 Pa/s and 0.295 Pa/s for the 0.5% and 1% OCS mixes, respectively. Compared with the control mix, the structuration rate increased by approximately 150% and 166% for the 0.5% and 1% OCS mixes. The significantly higher A_{thix} values indicate that the OCS-modified mixes developed structural rigidity at a substantially faster rate during rest. Such behaviour is particularly beneficial for 3DPC because a higher structuration rate enables freshly deposited layers to rapidly gain resistance against deformation and sustain the weight of subsequently printed layers.

The simultaneous increase in static yield stress and structuration rate provides a mechanistic explanation for the enhanced buildability and dimensional stability observed in previous sections. Although OCS increased the setting times, the rheological results indicate that the OCS-modified mixes developed a stronger load-bearing structure during the fresh state, thereby improving their resistance to layer compression and geometric distortion during printing. This interpretation is consistent with the higher maximum printable layers, reduced settlement, and lower structural deformation rates observed for the OCS mixes. The enhanced structural build-up may be attributed to modifications in particle interaction and packing characteristics induced by the residual hydrocarbons coating the aggregate surfaces, which facilitated the formation of a more stable particle skeleton during rest.

Overall, the rheological results demonstrate that incorporation of OCS significantly enhanced the structural build-up behaviour while maintaining comparable thixotropic characteristics. The higher static yield stresses and substantially increased structuration rates enhanced the ability of the deposited filaments to resist deformation under self-weight and the stresses imposed by subsequently deposited layers, thereby improving buildability and geometric stability during the printing process.

3.1.6. Hydration Behaviour

Figure 15 presents the heat evolution curves of the control mix (0% OCS) and the mix incorporating 1% OCS. Both mixes exhibited similar hydration profiles, characterised by the typical stages of cement hydration, including the initial dissolution period, dormant period, acceleration stage, and subsequent deceleration stage. A pronounced main hydration peak associated with the formation of C-S-H gel and calcium hydroxide was observed between approximately 500 and 800 min for both mixes. The 1% OCS mix exhibited a slightly lower peak heat evolution rate compared with the control mix, indicating a marginal reduction in the rate of hydration. However, the overall similarity of the hydration curves suggests that incorporation of 1% OCS exerted only a limited influence on the hydration kinetics of the cementitious system.

The slight reduction in heat evolution observed for the OCS-modified mix may be attributed to the presence of residual hydrocarbons coating the aggregate particles, which can partially restrict water accessibility and delay interactions between cement particles and the surrounding pore solution [25]. Nevertheless, the difference between the two curves remained negligible throughout the monitoring period, indicating that the low level of oil contamination adopted in this study was insufficient to significantly alter the hydration process. This observation is consistent with the mechanical performance results, in which

the mix containing 1% OCS exhibited compressive strengths higher than the control mix despite the marginal reduction in hydration intensity. The negligible difference between the hydration profiles indicates that the performance variations observed in the OCS-modified mixes are unlikely to be governed primarily by changes in cement hydration kinetics.

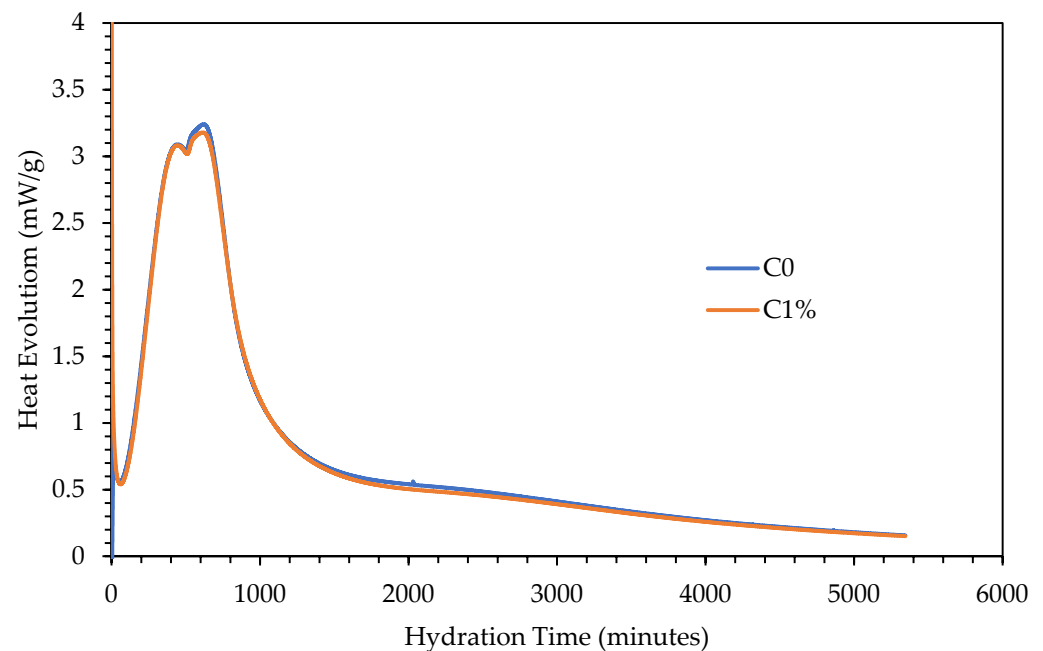


Figure 15. Heat evolution rate of mixes incorporating 0% and 1% OCS obtained from isothermal calorimetry.

3.2. Mechanical Properties

3.2.1. Compressive Strength

The compressive strength of the 3DPC mixes incorporating different levels of OCS tested in the Y- and Z-directions is shown in Figure 16. The results demonstrate that the incorporation of OCS significantly influenced the compressive performance of the printed specimens. The control mix exhibited compressive strengths of 21.47 MPa and 27.16 MPa in the Y- and Z-directions, respectively. Incorporation of 0.5% OCS increased the compressive strength to 33.35 MPa in the Y-direction and 34.65 MPa in the Z-direction, corresponding to improvements of approximately 55.3% and 27.6%, respectively, compared with the control mix. Increasing the OCS content to 1% resulted in compressive strengths of 28.68 MPa and 31.67 MPa in the Y- and Z-directions, respectively. Although these values were lower than those of the C0.5% mix, they remained higher than those of the control mix, representing increases of approximately 33.6% and 16.6% in the Y- and Z-directions, respectively.

The improvement in compressive strength observed for the OCS-modified mixes may be attributed to changes in matrix development and internal structure resulting from the presence of residual hydrocarbons on aggregate surfaces [25]. The C0.5% mix exhibited the highest compressive strength, which is consistent with the SEM observations showing a denser and more homogeneous microstructure with fewer visible pores and improved matrix continuity. In addition, the rheological results demonstrated enhanced structural build-up and higher static yield stresses for the OCS-modified mixes, while the buildability assessment revealed greater maximum printable heights and reduced structural deformation. These characteristics may have contributed to improved filament stability during printing, reducing geometric deformation during layer deposition and producing a denser and more homogeneous hardened matrix, which consequently improved the compressive load-bearing capacity of the printed material. However, a slight reduction

in compressive strength was observed when the OCS content increased from 0.5% to 1%. This behaviour is consistent with the SEM observations of the C1% mix, where a slightly greater number of pores and isolated microcracks were identified. These microstructural defects can act as stress concentration sites and reduce the efficiency of load transfer within the hardened matrix [51], resulting in lower compressive strength compared with the C0.5% mix. Nevertheless, the C1% mix continued to exhibit higher compressive strengths than the control mix, demonstrating that the beneficial effect of low OCS can enhance the compressive performance of 3DPC.

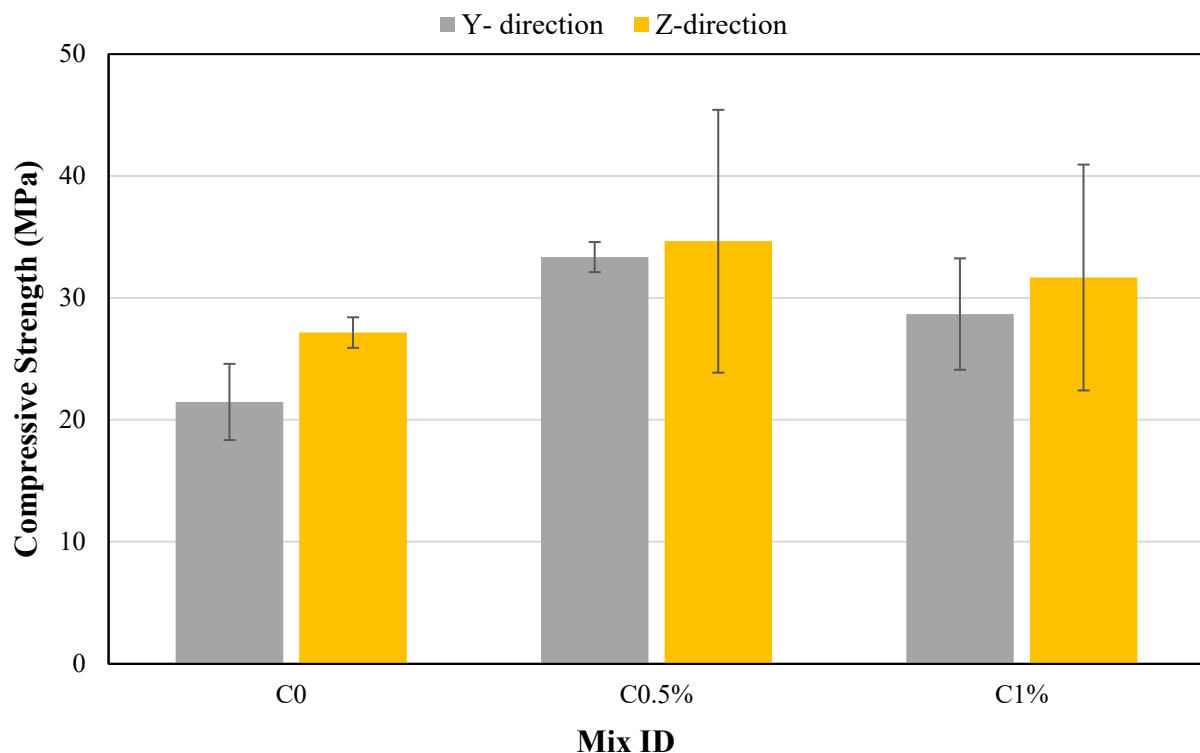


Figure 16. Compressive strength of 3DPC mixes incorporating different levels of OCS measured in the Y- and Z-directions.

For all mixes, compressive strengths recorded in the Z-direction exceeded those obtained in the Y-direction. Such directional differences are commonly observed in extrusion-based 3DPC and are associated with the influence of layer orientation, interlayer interfaces, and anisotropic load-transfer mechanisms developed during the printing process [52,53]. The influence of OCS incorporation on the directional dependency of compressive behaviour is further discussed in Section 3.2.3 through anisotropic degree analysis.

3.2.2. Flexural Strength

The flexural strength of the 3DPC mixes incorporating different levels of OCS tested in the Y- and Z-directions is presented in Figure 17.

The flexural strength test shows a clear trend of gradual reduction with increasing OCS content in both loading directions. The control mix exhibited the highest flexural strengths of 7.66 MPa and 7.29 MPa in the Y- and Z-directions, respectively. Incorporation of 0.5% OCS reduced the flexural strength to 6.98 MPa and 6.22 MPa, corresponding to reductions of approximately 8.9% and 14.7%, respectively, relative to the control mix. A further decrease was observed for the 1% OCS mix, which exhibited flexural strengths of 6.31 MPa and 6.19 MPa in the Y- and Z-directions, representing reductions of approximately 17.6% and 15.1%, respectively. These results indicate that the increasing OCS content

progressively reduced the flexural resistance of the printed mixes, although all specimens maintained flexural strengths exceeding 6 MPa.

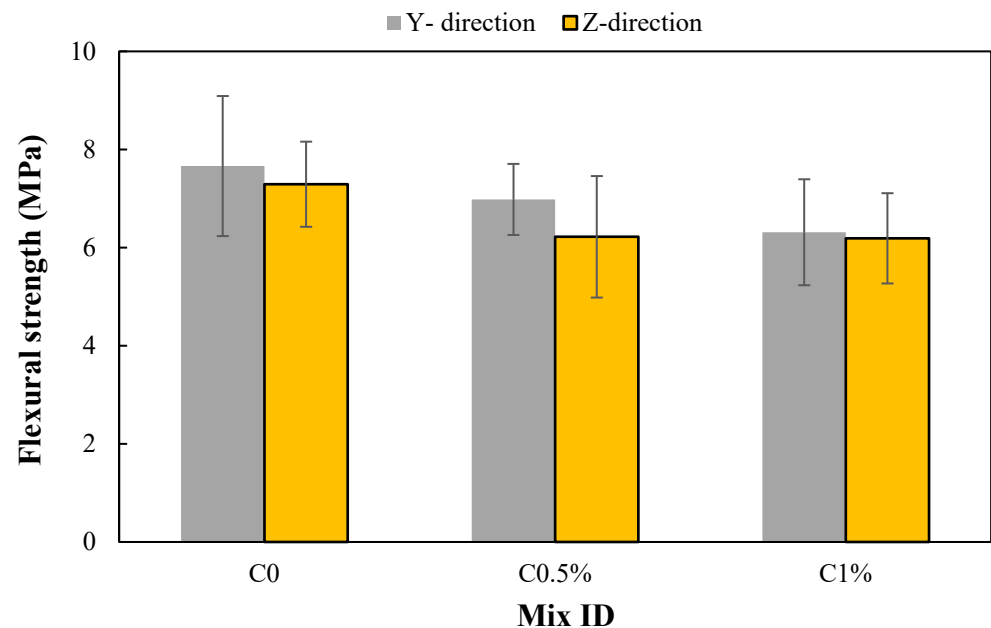


Figure 17. Flexural strength of 3DPC mixes incorporating different levels of OCS measured in the Y- and Z-directions.

The reduction in flexural strength suggests that OCS exerted a greater influence on the tensile load-carrying capacity of the printed matrix than on its compressive resistance. Unlike compressive failure, flexural failure is governed primarily by crack initiation and propagation within the tensile zone of the specimen [54]. Therefore, although OCS promoted a denser microstructure and improved compressive performance, the presence of residual hydrocarbons on the aggregate surfaces may have influenced the aggregate–paste interface and local bonding characteristics within the matrix, reducing the efficiency of stress transfer under tensile loading. These localised weak regions can facilitate crack initiation under tensile stresses and promote crack propagation during loading, resulting in lower flexural strengths. Furthermore, the slightly greater number of pores and isolated microcracks observed in the C1% mix may have contributed to stress concentration and accelerated crack growth, leading to lower flexural strength compared with the C0.5% mix. Nevertheless, the reduction in flexural strength remained moderate, indicating that the incorporation of low levels of OCS did not cause severe deterioration of the structural integrity of the printed matrix.

3.2.3. Anisotropic Degree

The anisotropic degrees calculated from the compressive and flexural strength results are presented in Figure 18. For compressive strength, the control mix exhibited the highest anisotropic degree of 20.96%, indicating a pronounced difference between the Y- and Z-direction strengths. Incorporation of 0.5% OCS significantly reduced the anisotropic degree to 3.76%, representing an approximately 82.1% reduction relative to the control (C0) mix. However, when the OCS content was increased to 1%, the anisotropic degree increased to 9.46%, although this value remained substantially below the value recorded for the control mix. The marked decrease in anisotropy exhibited by the C0.5% mix suggests that low-level incorporation of OCS improved the uniformity of mechanical properties within the printed structure. This behaviour is consistent with the rheological results, which demonstrated enhanced structural build-up and higher static yield stress, as well as the SEM observations

showing a denser and more homogeneous microstructure. Together, these factors may have promoted more uniform filament deposition and reduced structural heterogeneity between adjacent layers, resulting in more uniform stress transfer across the printed structure and consequently reducing the directional dependence of compressive behaviour. The increase in anisotropy observed for the C1% mix may be associated with the greater number of pores and isolated microcracks identified in the SEM micrographs, which can introduce localised heterogeneity within the printed matrix.

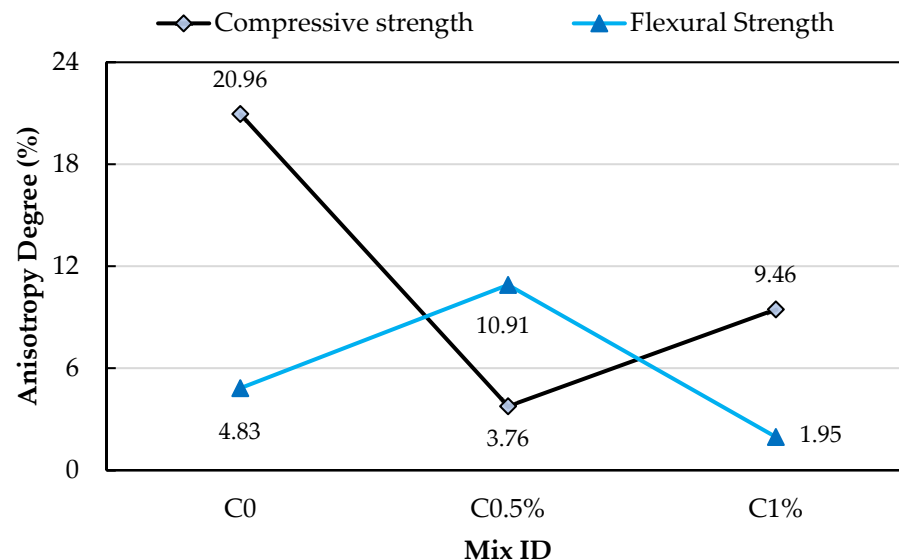


Figure 18. Anisotropic degree of compressive and flexural strengths of 3DPC mixes incorporating different levels of OCS.

A different trend was observed for flexural strength. The anisotropic degree increased from 4.83% for the control mix to 10.91% for the C0.5% mix before decreasing substantially to 1.95% for the C1% mix. Despite these variations, the flexural anisotropy values remained relatively low for all mixes, indicating limited directional dependence of flexural performance. The lower anisotropy observed for flexural strength compared with compressive strength suggests that the influence of layer orientation on flexural behaviour was less pronounced under the adopted testing configuration.

3.3. Microstructural Properties

Figure 19 presents the SEM micrographs of the 3DPC mixes incorporating different levels of OCS as fine aggregates. The control mix exhibited a typical cementitious microstructure consisting of hydration products distributed throughout the matrix together with several visible pores and microvoids. Although a relatively continuous hydration network was observed, localised voids and discontinuities were present within the matrix, indicating the existence of capillary pores formed during hydration. These pores may act as potential weak zones under loading and contribute to the comparatively lower mechanical performance observed for the control mix.

The incorporation of 0.5% OCS resulted in a noticeably denser and more compact microstructure. The matrix appeared more homogeneous, with fewer visible pores and a greater degree of continuity between hydration products. The reduction in pore size and pore frequency suggests improved particle packing and a more efficient arrangement of solid constituents within the cementitious matrix. This observation is consistent with the superior compressive strength exhibited by the C0.5% mix. The improved microstructural compactness may be attributed to the thin hydrocarbon film coating the aggregate particles, which modified interparticle interactions and contributed to a more coherent internal

structure. Similar observations were reported by Abousnina et al. [25], who found that low levels of oil-contaminated sand produced a denser microstructure with a finer pore structure and no significant deterioration of the interfacial transition zone.

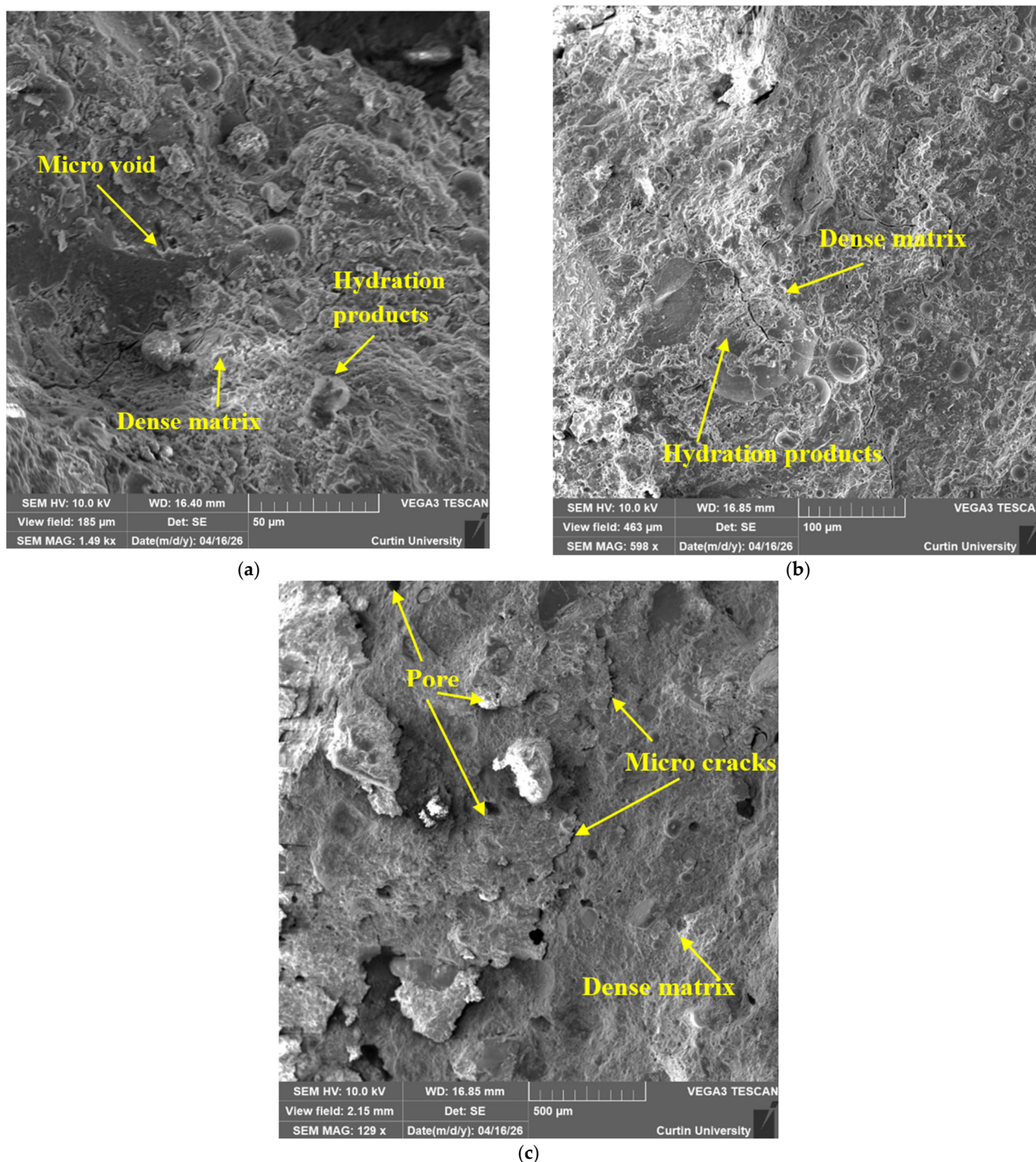


Figure 19. SEM micrographs of 3DPC incorporating OCS: (a) C0, (b) C0.5%, and (c) C1%.

For the C1% mix, the matrix remained relatively compact and continuous; however, a slightly greater number of pores, localised discontinuities, and isolated microcracks were observed compared with the C0.5% mix. Although these features were not sufficiently severe to compromise the overall integrity of the matrix, they indicate the onset of microstructural changes associated with increasing oil contamination. The presence of residual hydrocarbons on aggregate surfaces may partially interfere with particle packing

and matrix development, resulting in the formation of isolated pores and microstructural defects within the hardened matrix. The appearance of microcracks may further contribute to local stress concentration and reduce the efficiency of load transfer under compressive loading. Nevertheless, the overall microstructure of the C1% mix remained denser than that of the control mix, which is consistent with its higher compressive strength and improved buildability performance. The SEM observations, therefore, suggest that the enhanced mechanical performance of the OCS-modified mixes is primarily associated with microstructural densification and reduced pore content rather than significant changes in cement hydration kinetics, which is consistent with the calorimetry results presented in Section 3.1.6.

Although long-term durability was not investigated in the present study, the observed microstructural characteristics may have implications for the long-term performance of OCS-modified 3DPC. The denser and more homogeneous microstructure observed for the 0.5% OCS mix may contribute to improved resistance against the ingress of aggressive agents, potentially enhancing durability. However, residual oil may also influence the long-term bond between the cement matrix and aggregate particles, particularly under prolonged environmental exposure. Furthermore, the durability behaviour of OCS-modified 3DPC may be affected by factors such as moisture transport, freeze–thaw resistance, chloride penetration, and chemical attack, which require dedicated long-term investigation. Therefore, comprehensive durability studies are recommended before the widespread practical application of OCS in 3DPC.

4. Conclusions

This study investigates the feasibility of incorporating oil-contaminated sand (OCS) as a partial replacement of natural sand in 3DPC. The effects of OCS incorporation on fresh-state behaviour, printability, rheological response, hydration behaviour, mechanical performance, and microstructural characteristics are evaluated. It is found that OCS can enhance buildability and improve compressive performance of 3DPC, with the 0.5% OCS mixture resulting in the most balanced overall performance. The improved behaviour can be attributed to enhanced structural build-up and a denser cementitious matrix rather than significant changes in cement hydration.

The experimental results support the findings that demonstrate the potential for valorising oil-contaminated sand as an alternative fine aggregate in additive manufacturing for construction. The test results support sustainable construction by reducing the demand for natural sand while providing a circular economy for contaminated industrial waste that would otherwise require costly disposal or remediation. The results also provide preliminary results for OCS replacement levels in 3DPC concrete applications, contributing to the development of more resource-efficient and environmentally responsible construction materials.

5. Limitations and Future Research

The present study was limited to laboratory-simulated OCS prepared using a single motorcycle oil at contamination levels of 0.5% and 1% under controlled laboratory conditions. Therefore, the findings should not be directly extrapolated to field-contaminated sands, which may contain aged hydrocarbons and other contaminants with different physicochemical characteristics.

Moreover, durability assessment was beyond the scope of this study and future research should therefore investigate long-term durability, contaminant leaching behaviour and compliance with relevant environmental regulations, and pilot- and industrial-scale implementation to facilitate the practical application of OCS in 3DPC.

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