The influence of viscosity-modifying agent and calcium carbonate on 3D printing mortar characteristics

The influence of viscosity-modifying agent and calcium carbonate on 3D printing mortar characteristics

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Abstract. 3D printing or additive manufacturing is an example of technology development in the construction sector. In 3D printing mortar, many things need to be considered regarding the characteristics of the mortar, such as workability, initial setting time, compressive strength, extrudability, and buildability. In previous studies, there were problems in the printing process, such as a lousy extrusion process and cracks in the 3D printing mortar sample. This study used calcium carbonate and a viscosity-modifying agent (VMA) to modify the mixture to achieve the desired characteristics. The extrudability and buildability were tested by observing the extrusion process and measuring the thickness and width of each layer printed. Based on the research, adding calcium carbonate and VMA can reduce workability and accelerate the initial setting time of the mixture. The use of VMA can reduce the compressive strength of the mixture. Using calcium carbonate and VMA can also increase the buildability of the mixture. From the compressive strength test, there was a decrease of 39.26% in the 3D printing mortar sample compared to the mortar cube sample. In addition, it was found that the 3D printing mortar is anisotropic, so the compressive force's direction affects the compressive strength produced.

1. Introduction

In recent years, the construction industry has started to adopt digital technology in its application, and this industry is currently entering the fourth industrial revolution [1]. Additive manufacturing, usually known as 3D printing, is one of the technological advancements that is now being developed in the construction industry. The advantage of 3D printing construction is that it can reduce construction and is relatively eco-friendly because of the absence of formwork in its process [2]. Furthermore, complex-shaped concrete structures can be achieved by using this method [3].

In 3D printing, there are many things to be considered regarding the characteristic of the mortar used, such as workability, initial setting time, compressive strength, extrudability, and buildability. Some additives are needed in the 3D printing mortar mix to get a mortar mixture that meets the 3D printing material criteria mentioned earlier. For example, the additives that can be used are calcium carbonate, calcium oxide, accelerator viscosity-modifying agent (VMA), and superplasticizer [4].

In recent years, the usage of nano-$CaCO_3$ has been introduced in concrete [5]. Calcium carbonate is a material that can be used to manufacture 3D printing mortar. Calcium carbonate reacts with tricalcium silicate (C_3S), produces higher initial compressive strength, and speeds up the setting time [6]. According to Valcuende et al. [7], calcium carbonate can be used as a replacement for cement to speed up the initial hydration of cement and improve the early modulus elasticity, along with the increase of

calcium carbonate. The higher the calcium carbonate content, the shorter the initial and final setting time. With a faster initial setting time, the 3D printing mortar mix hardens faster to increase the buildability of the 3D printing mortar. Even according to Vergara & Colorado [8], calcium carbonate does not affect the characteristics of the 3D printing mortar mix. However, using calcium carbonate in 3D printing mortar is still not widely used. Therefore, this study aims to evaluate the effect of calcium carbonate content on the characteristics of the 3D printing mortar mix.

The addition of viscosity-modifying admixture (VMA) can affect the viscosity of the mortar mixture by absorbing water in the mixture [9]. The mortar used in 3D printing construction needs to have the ability to maintain its shape after being deposited from the nozzle. In the recent research by Chaves Figueiredo et al. [10], VMA with a mass ratio of 0.14% - 0.48% from binder is used. The higher the content of VMA can increase the yield stress and shape stability of the mortar coming out of the pipe. However, more pressure is needed to extrude the mixture from the pipe because the mixture becomes thicker and affects the extrudability of the mixture.

This study aims to develop the previous research by Antoni et al., where cracks were still found in the molded mortar and could be repaired using synthetic macro fiber. [11,12]. As an alternative, viscosity-modifying admixture as an additive was proven to reduce cracks in mortars, and the mixture's buildability could also increase [10]. The mortar extrusion tool used in the previous research to examine extrudability and buildability characteristics from 3D printing mortar was a traditional printing instrument using PVC pipes and a manual mortar pump [11,12]. The problem was that the mixture was challenging to extrude from the pipe because manual human power could not apply enough pressure. Therefore, this research used a tool with the help of electric power that can put more pressure on the end of the pipe so that the mixture can come out quickly.

Another objective of this research was to determine the composition of the mixture suitable for 3D printing using calcium carbonate, calcium oxide, and an accelerator to speed up the initial setting time and improve buildability. In addition, the use of viscosity-modifying admixture (VMA) and superplasticizer aim to maintain the rheology of the mixture so that it can be extruded continuously without being obstructed by the nozzle with the help of a pump that uses electric power. The effect of adding calcium carbonate and viscosity-modifying admixture (VMA) with varying contents was also investigated to analyze the effect of these materials on the characteristics of 3D printing mortar. This study also compared the compressive strength of a 5cm cube sample against the compressive strength of 3D printing samples with various directions of compressive force. The 3D printed specimen is a mortar printed through a 3.5 cm width and 0.6 cm nozzle, printed in a straight line, in a total of 5 layers.

2. Materials and Testing Procedures

2.1. Mix Design

The materials used in this research were PCC cement, silica sand, water, superplasticizer, VMA, calcium carbonate ($CaCO_3$), and calcium oxide (CaO). The silica sand used was the one that passed the No. 50 sieve and was retained on the No. 100 sieve. Polycarboxylate-based superplasticizer, VMA, and accelerator were obtained from Sika. The calcium carbonate used was calcium carbonate mesh 2000. Meanwhile, calcium oxide was obtained from a limestone factory in Tuban.

The content of each material was tested at three different ratios, keeping the other materials the same to examine the influence of VMA and calcium carbonate. The mix design obtained by trial and error can be seen in Table 1. "V" letters in the table represent the VMA contents in the mix designs, while "C" letters represent the calcium carbonate content. V0C0, V0C0.1, and V0C0.2 were made to examine the effect of calcium carbonate, while V0.00030C0, V0.00035C0, and V0.00040C0 were made for the VMA influence. The effect of the combination of the two can be seen in V0.00030C0.1 and V0.00030C0.2.

Table 1. Mix design in a mass ratio to cement.

Mix	Cement	Sand	W/C	SP	VMA	Accelerator	CaO	$CaCO_3$
V0C0								0
V0C0.1	1	1.75	0.4	0.004	0	0.04	0.15	0.1
V0C0.2								0.2
V0.0030C0					0.003			
V0.0035C0	1	1.75	0.4	0.004	0.0035	0.04	0.15	0
V0.0040C0					0.004			
V0.0030C0.1	1	1.75	0.4	0.004	0.003	0.04	0.15	0.1
V0.0030C0.2								0.2

2.2. Mixing methods

Cement, sand, and water were mixed and stirred first. Then, the superplasticizer diluted with water was added to the mixture. Accelerator and VMA were added later to speed up the initial setting time and increase mix viscosity. After that, CaO was added first, then followed by the addition of calcium carbonate.

2.3. Testing procedures

The test was divided into two stages. The initial characteristic was examined from the flow table test, initial setting time testing, and compressive strength testing. The advanced characterize stage was in the form of testing the extrudability, buildability, and compressive strength of 3D printing samples.

Workability testing with flow table test refers to ASTM C1437 [13]. The mortar was poured into the mold in three stages and compacted by pounding it at each stage. Then, the mold was lifted, and the flow table lever was rotated 25 times in 15 seconds. The diameter of the mortar mixture on the flow table was then recorded.

Initial setting time testing was carried out according to ASTM C403/403M [14] that was applied on molding mortar in 15cm-rib cube formwork. The mortar was inserted into the formwork until the thickness reached 5 cm. The measurement was conducted every 15 minutes. Then, the pressure reading on the penetrometer was recorded. The mortar has reached the initial setting when the penetrometer has reached 500 psi.

The mortar was extruded using an electric mortar pump, as shown in Figure 1, to maintain the extrusion rate. The nozzle was modified using a plastic hose to form a flat form of nozzle, with a width of 3.6cm and thickness of 0.6 cm. The printing bed was made of multiplex, 30 cm width, and 60 cm length. The printing bed had 4 wheels so that the printing bed could move in one direction of motion. The printing mechanism used the mortar pump to extrude the mortar, and the printing bed would be moved manually in one direction. As soon as 1 layer had been printed, the next layer would be printed until it reached a total of 5 layers.

The mortar compressive strength test was started by molding the mortar in the 5cm-rib cube formwork. The compressive strength of mortar was tested at the age of 3, 7, 14, and 28 days using the Universal Testing Machine. The hardened printed sample was cut to form a 5cm cube to measure the compressive strength of the 3D printed mortar sample. The compressive strength result was the average of three samples from each mix design.

Extrudability and buildability tests were carried out visually by measuring the width and thickness of the hardened layer using a ruler. The extrudability examination was done by direct observation, as the printing process of 5 layers of mortar. The mixture was said to have good extrudability if the mortar extruded from the nozzle could be extruded continuously and had no cracks visible on the surface. A mixture with good buildability is a mixture that can maintain its shape and withstand the load of the layer above it. The buildability test shows the width dimensions change across the layers in one

specimen [15]. Consistency of the dimension between the layers in one specimen was the goal of a buildable mortar.

Figure 1. A mortar extruder, nozzle, and printing bed are used for mortar extrusion.

3D printing mortar with excellent quality meets the following characteristics:

- The mortar does not change shape when removed from the mold and has a flow diameter value ranging from 15 - 19 cm [16]
- The initial setting time is below 90 minutes [17]
- Good extrudability, mortar can be printed out of the nozzle and be placed on the printing bed continuously without visible cracks [15]
- With good buildability, mortar can maintain its shape (not having any deformations) when loaded with another layer, measured with a change in the dimensions of the width of the cross-section of the layers [18]

The compressive strength test of the 3D printing layer sample was carried out by cutting the 3D printed sample to resemble a cube shape with 5 cm ribs. The compressive strength test was carried out when the sample was 28 days old. The compressive strength test was carried out by applying a compressive force in the direction perpendicular to layer (A), parallel to the cross-section in a transverse direction (B), and perpendicular to the cross-section (C), as shown in Figure 2.

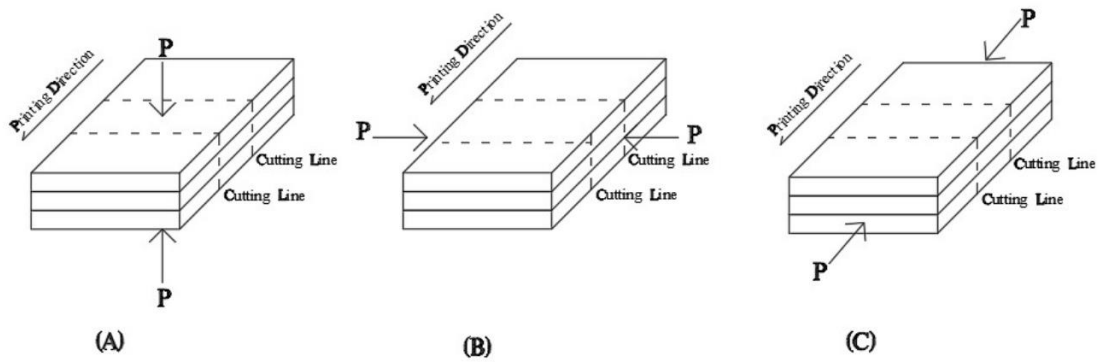

Figure 2. Compressive test directions.

3. Results and Discussion

3.1. Mortar basic characteristic testing

3.1.1. *Workability test.* From Figure 3, the results of the workability test, V0C0, V0C0.1, and V0C0.2 test results show that the higher the $CaCO_3$ content in the mixture, the lower the flow diameter. Adding $CaCO_3$ can fill the air voids in the mixture so that the mixture becomes denser [19]. The addition of VMA also has a similar impact. The flow table diameter from the workability test results on V0.0040C0 shows lower results than Mix V0.0030C0 and V0.0035C0, which have the same flow diameter. The results were because adding VMA can increase the bond between materials so that the material becomes denser and becomes a compact unit.

Mix V0.0030C0.1 and V0.0030C0.2 were combined with the addition of VMA with a ratio of 0.3% and $CaCO_3$ with a ratio of 10% and 20%. This combination aims to get a mixture with a flow diameter that matches the desired parameters (15-19 cm) because the Mix V0C0.1, V0.0030C0, and V0.0035C0 flow diameters do not meet the parameters. The addition of VMA and CaCO3 succeeded in reducing the diameter of the flow so that the results followed the parameters.

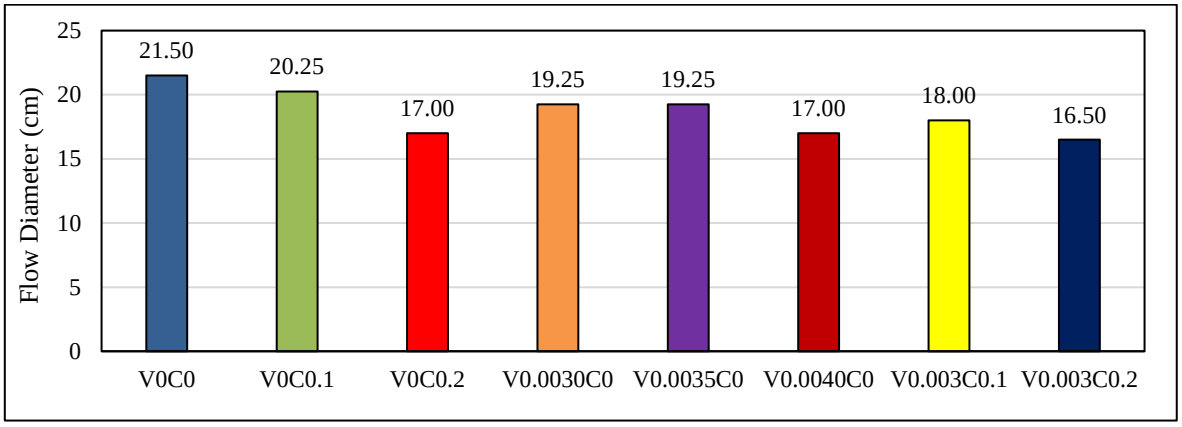

Figure 3. Result of flow table test.

3.1.2. *Initial setting time.* The test results for Mix V0C0, V0C0.1, and Mix V0C0.2 show that the higher the $CaCO_3$ content in the mixture, the faster the initial setting time (Figure 4 and Figure 5). The faster setting was because adding $CaCO_3$ could accelerate the initial hydration process of the mixture. Moreover, adding $CaCO_3$ causes the mixture to become denser so that the initial setting time becomes faster [7]. In Mix V0.0030C0, V0.0035C0, and V0.0040C0, the result was that the higher the VMA content in the mixture, the faster the initial setting time. The faster setting was because VMA can increase the bond between materials, so a faster initial setting time was obtained [20].

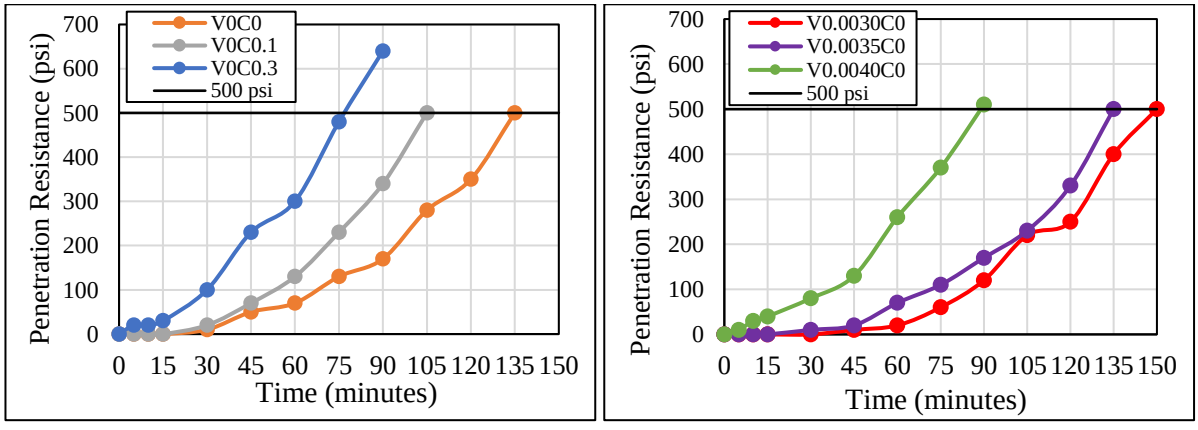

Figure 4. Initial setting time of mixture with the variation of (a) $CaCO_3$ and (b) VMA dosage.

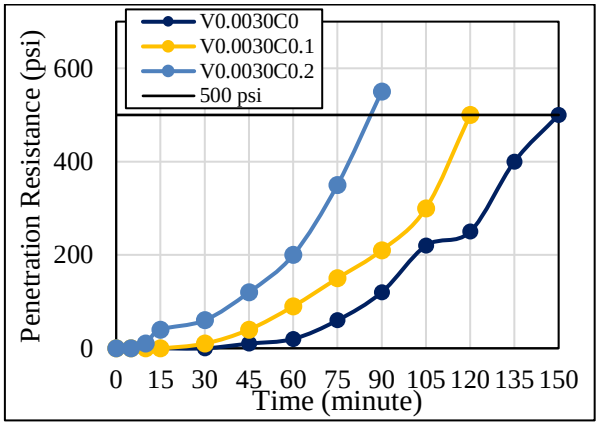

Figure 5. Initial setting time of combined $CaCO_3$ and VMA.

In Mix V0.0030C0, V0.0030C0.1, and V0.0030C0.2, the results showed that the higher content of $CaCO_3$ combined with VMA at a ratio of 0.3% resulted in a faster initial setting time. However, using a combination of $CaCO_3$ and VMA resulted in a slower initial setting time when compared to adding $CaCO_3$ without VMA. The slower setting can be seen in Mix V0.0030C0.1. The combination of VMA and $CaCO_3$ produces a slower initial setting time compared to the addition of $CaCO_3$ alone because VMA can slow down the C-S-H reaction in the mixture [21].

From the initial setting time test, only Mix V0C0.2 and Mix V0.0040C0 meet the requirement of an initial setting time under 90 minutes. However, it should be noted that a faster initial setting time can reduce the time for the printing process. Therefore, in making a 3D printing mix, the required initial setting time must be adjusted to the pump and the time available during the printing process.

3.1.3. *Compressive strength test*. The compressive strength test results are shown in Figure 6 and Figure 7. The addition of $CaCO_3$ didn't impact the compressive strength of the 3D printing mortar. From the results of testing the compressive strength of the Mix V0C0, V0C0.1, and V0C0.2 cube samples, it was found that the variation of $CaCO_3$ in the mixture didn't have a significant impact on the compressive strength. In contrast to $CaCO_3$, the addition of VMA caused a decrease in the compressive strength of the 3D printing mortar. In Mix V0.0030C0, V0.0035C0, and V0.0040C0, the results show that the higher the VMA content in the mixture, the compressive strength got lower. The use of VMA causes the hydration reaction to slow down and provides a water retention effect on the mortar to prevent bleeding, which was one of the significant factors in reducing compressive strength [22].

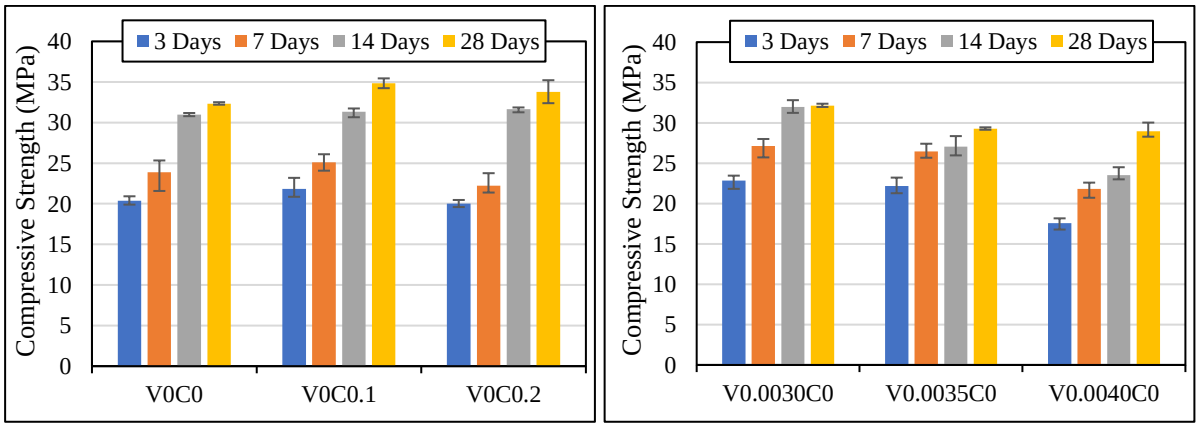

Figure 6. Compressive strength for the mixture with the variation of (a) $CaCO_3$ and (b) VMA dosage.

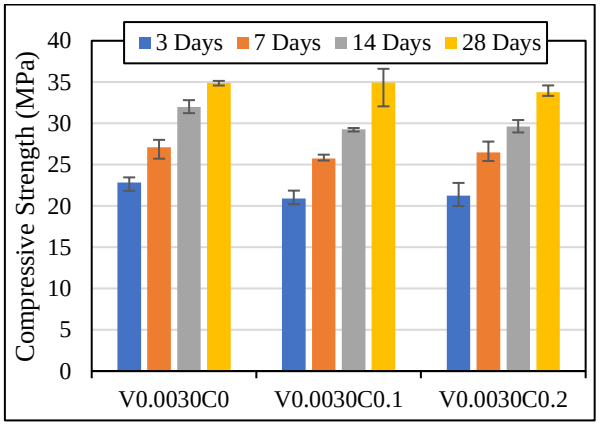

Figure 7. Compressive strength of combined $CaCO_3$ and VMA.

3.2. Initial characteristic test

3.2.1. Extrudability and buildability test. From the results of extrudability and buildability testing, it was found that in Mix V0C0, V0C0.1, and V0C0.2. It was seen that the variations of $CaCO_3$ contents influence the extrudability and buildability of the sample. The more $CaCO_3$ content added to the mixture, the increased extrudability, and buildability of the mixture. The measurements of the layer's width and thickness are shown in Table 2, showing that the higher the $CaCO_3$ content, the width of the layer that comes out decreases and is closer to the width of the nozzle. The $CaCO_3$ filled the voids in the mixture so that the density of the mixture increased. In addition, $CaCO_3$ also absorbs the water content in the mixture so it can maintain its shape well.

Table 2. Width and thickness of each printed layer.

Layer	V0C0 $CaCO_3$ 0% VMA 0%		V0C0.1 $CaCO_3$ 10% VMA 0%		V0C0.2 $CaCO_3$20% VMA 0%		V0.0030C0 $CaCO_3$ 0% VMA 0.3%	
	Thickness (cm)	Width (cm)	Thickness (cm)	Width (cm)	Thickness (cm)	Width (cm)	Thickness (cm)	Width (cm)
Layer 5	0.7	4.5	0.8	4.0	0.7	3.8	0.6	4.2
Layer 4	0.7	5.0	0.9	4.3	0.7	4.0	0.6	4.7
Layer 3	0.8	5.0	1.0	4.7	0.6	4.3	0.8	4.5
Layer 2	0.6	5.0	0.7	4.5	0.8	4.5	0.7	5.5
Layer 1	1.0	5.0	0.8	5.0	0.5	5.0	0.6	5.0
Layer	V0.0035C0 $CaCO_3$ 0% VMA 0.35%		V0.0040C0 $CaCO_3$ 0% VMA 0.4%		V0.0030C0.1 $CaCO_3$ 10% VMA 0.3%		V0.0030C0.2 $CaCO_3$ 20% VMA 0.3%	
	Thickness (cm)	Width (cm)	Thickness (cm)	Width (cm)	Thickness (cm)	Width (cm)	Thickness (cm)	Width (cm)
Layer 5	0.6	3.8	0.5	3.5	0.5	3.5	0.8	3.1
Layer 4	0.6	3.8	0.6	3.7	0.6	4.0	1.0	4.0
Layer 3	0.6	4.3	0.6	4.3	0.8	4.3	1.0	4.1
Layer 2	0.7	4.5	0.6	4.5	0.8	4.3	1.0	4.0
Layer 1	0.6	4.7	0.6	4.6	0.8	4.4	0.9	4.0

In Mix V0.0030C0, V0.0035C0, and V0.0040C0, adding VMA content to the mixture increases extrudability and buildability. The result can be seen from the layer width getting closer to the nozzle width. The use of VMA modifies the characteristics of the mixture so that it becomes more viscous and

increases the bond between the particles in the mixture. In contrast to $CaCO_3$, VMA does not absorb water content but maintains water content so that it remains in the mixture and does not come out to the surface of the mixture [22]. In addition, using VMA can prevent cracks from the 3D printing mortar layer [7].

In Mix V0.0030C0.1 and V0.0030C0.2, a combination of $CaCO_3$ and VMA was used, giving more optimal extrudability and buildability results. The printing results from Mix V0.0030C0.2 show the best results compared to other mixtures. It can be seen from Table 2, Figure 8, and Figure 9 that Mix V0.0030C0.2 had the best buildability characterized by a regular cross-sectional shape and a layer that can maintain its shape even though it was loaded with the other subsequent layers.

Figure 8. Mortar printing results.

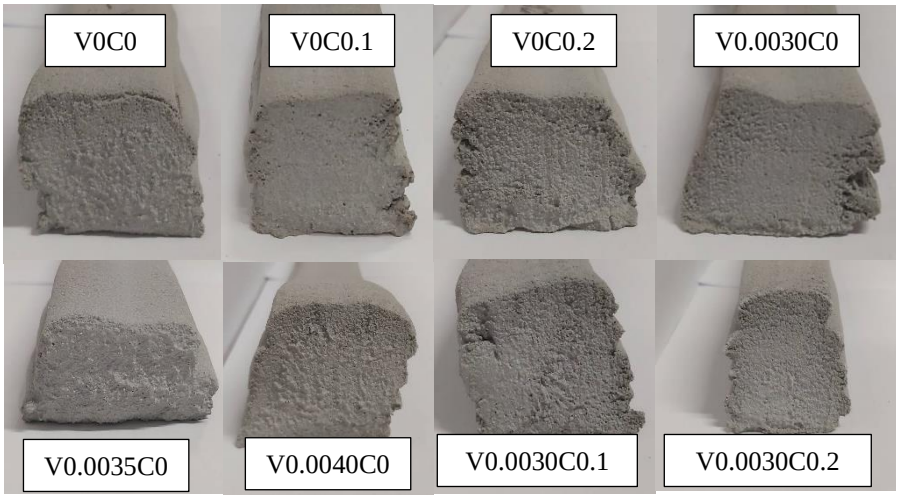

Figure 9. Cross sections of the printed mix designs.

Table 2 shows that the base layer has a larger layer width than the other layers, while the top layer has a smaller layer width than the other layers. The base layer received the heaviest layer load, so the layer was deformed, which caused the layer width to change from the original. The top layer received no load, so the layer width was maintained the same as when the mixture was extruded. However, The printing process can be optimized by the uniformity of the movement speed of the printing bed and the volume of mortar leaving the pump.

All the mixtures shown in this study can come out of the pump properly without any obstacles. However, it should be noted that in this research, the printing time gap between layers was relatively short. As soon as one layer had been printed, the subsequent layer was immediately printed. Over time, the mixture may lose its workability and cause the mixture to fail to exit the pump. Therefore, the printing process must pay attention to the period from mixing water and cement until the mixture cannot be removed from the pump. This period is known as open time. In this study, all mixtures had an open time of approximately 50% of the initial setting time of each mixture.

3.2.2. Compressive strength test of 3D printing mortar. Testing the compressive strength of the 3D printing layer sample was carried out by cutting the layered sample to resemble a cube shape with 5 cm ribs. The corrugated side of the layers was also trimmed, so the compressive test on the direction parallel to the cross-section in the transverse direction (B) could be done. The compressive strength test was carried out when the sample was 28 days old. The results of testing the compressive strength of the 3D printing sample, which are compared with the compressive strength of the 5 cm cube sample, can be seen in Figure 10 and Table 3**.**

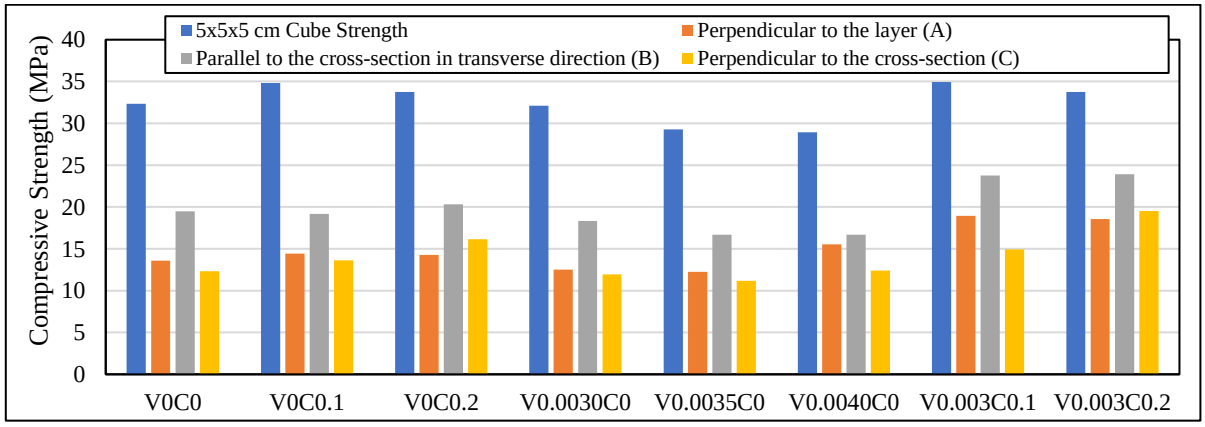

Figure 10 Compressive strength of 5cm cube and from 3 different directions.

Table 3. Compressive strength reduction of 3D printed mortars in comparison with 5cm cube sample.

Mix	Compressive Strength of 5x5x5cm Cube	Compressive Strength 3D mortar parallel to the cross-section in the transverse direction	Strength Reduction
	MPa	MPa	%
V0C0	32.33	19.48	39.76
V0C0.1	34.83	19.16	44.99
V0C0.2	33.77	20.32	39.82
V0.0030C0	32.12	18.32	42.95
V0.0035C0	29.29	16.7	43
V0.0040C0	28.95	16.67	42.39
V0.003C0.1	34.94	23.77	31.98
V0.003C0.2	33.77	23.91	29.21
Strength Reduction Average in Percentage			39.26

The results of the compressive strength of the 3D printing sample show that the sample tested for compressive strength in the direction parallel to the cross-section in the transverse direction (B) provides the highest compressive strength compared to the direction perpendicular to the layer (A) and the direction perpendicular to the cross-section (C). The increase in strength was because 3D printing samples have a strong bond between layers after 28 days. Meanwhile, in the compressive strength test in the direction perpendicular to layer (A), the compressive strength was lower due to the non-uniformity

of the axles between layers due to the manually moving printing bed process. For the compressive strength test, the direction perpendicular to the cross-section (C) has a lower compressive strength due to the irregular shape of the cross-section, so there were difficulties in finding the middle points of the samples when they were tested. Meanwhile, finding the middle points of the sample is crucial because the samples needed to be placed right in the middle of the Universal Testing Machine.

It can be concluded that the 3D printing mortar sample has anisotropic properties, that was, different characteristics depending on the direction of the applied force [22]. From testing the compressive strength of the 3D printing sample, it was found that there was a reduction in compressive strength compared to the compressive strength of the 5 cm rib cube sample, with an average of 39.26%, which can be seen in Table 3. Several factors cause the reduction in compressive strength, including 3D printing samples, which are not compacted, while 5 cm edge cube samples are compacted when cast. The next factor was an irregularity in the cross-section that was tested for compressive strength, thus affecting the compressive strength results. Another factor that caused the difference in compressive strength was that the 3D printing sample did not undergo a curing process like the 5 cm rib cube sample [23].

4. Conclusion

- Adding 20% $CaCO_3$ to the mixture reduces workability, speeds up the initial setting time, and increases buildability and extrudability. However, the use of variations in the contents of $CaCO_3$ in the mixture did not significantly impact the compressive strength of the mortar.
- The addition of VMA from 0.3% to 0.35% in the mixture did not change the flow diameter. However, the addition of the VMA content to 0.4% causes the flow diameter to decrease. Increasing the VMA usage rate speeds up the initial setting time and improves the extrudability and buildability of the mix. However, the higher the VMA content used can reduce the compressive strength. In addition, VMA can prevent cracks from the 3D printing mortar layer.
- Using a combination of 20% $CaCO_3$ and 0.3% VMA can improve extrudability, and buildability does not affect compressive strength and reduces workability. Using a combination of CaCO3 and VMA produces a slower initial setting time when compared to adding $CaCO_3$ without VMA.
- From the results of the overall compressive strength test of the 3D printing sample, adding $CaCO_3$ and VMA gave the same impact as the compressive strength of the 5 cm rib sample. The results of the compressive strength of the 3D printing sample show that the sample tested for compressive strength in the direction parallel to the cross-section (B) provides the highest compressive strength. This compressive strength result proves that the 3D printing sample is an anisotropic material.
- The compressive strength of the cube sample with the 3D printing sample with the compressive force parallel to the cross-section (B) showed a reduction in compressive strength with an average reduction of 39.26%.
- In this research, the mixture used as a 3D printing mortar was V0.0030C0.2. This mixture produces 3D printing samples with initial and advanced characteristics that meet the parameters.

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