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A Review of Optimization of Limestone and Calcined Clay Cement (LC3) Concrete Mixtures for 3d Printing

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Abstract **-** This paper reviews the existing literature on the optimization of Limestone Calcined Clay Cement (LC3) for 3D concrete printing mixtures. The main aim of this paper is to review the impacts of replacing Portland cement with calcined clay and limestone on the properties of 3D concrete mixtures. The paper investigated the property requirements for fresh and hardened concrete for 3D printing, the existing 3D printing concrete mix designs, the effects of substituting calcined clay and limestone for Portland cement and optimization methods for 3D concrete mixtures. In contrast to traditional concrete, the study discovered that 3D printed concrete requires unique properties such as extrudability, flowability, buildability and rapid setting time. Furthermore, the addition of calcined clay and limestone to the concrete mixture enhances buildability, green strength and compressive strength while reducing extrudability and flowability. To optimize 3D concrete printing mixtures, techniques like particle size distribution optimization, the usage of superplasticizers and admixture incorporation were identified in this review. These techniques have been shown to improve 3D printed concrete properties while also reducing its cost and environmental impact. In conclusion, the paper provides an in-depth review of the present state of knowledge regarding the optimization of LC3 for 3D concrete printing and highlights the need for further research to optimize the material for 3D printing.

Keywords**:** Limestone Calcined Clay Cement (LC3), Optimization, Three dimensional (3D) concrete printing, concrete mixtures.

1. Introduction

Over the past two decades, three-dimensional concrete printing (3DCP), alternatively referred to as additive manufacturing of cementitious materials, has been under development. The development of 3DCP reveals numerous benefits over traditional construction techniques. 3DCP technology enables flexibility in geometry and design, shortens the construction process, reduces material waste, and lowers costs by eliminating the requirement for formwork. Additionally, it requires less labour, significantly lowering the dangers to human life connected with construction.

Although 3D concrete printing displays substantial potential benefits, its strict requirements on the concrete mix properties are of major concern. Currently, 3DCP materials are less cost-effective than traditional materials. The price of concrete is increased by the use of unusual ingredients and the rigorous specifications for the qualities of the concrete mix used in 3D concrete printing [1]. According to Panda and Tan (2018), [3], it is challenging to create a material with zero slump and self-compaction, because these are contradictory goals. Compared to traditional concrete, 3DCP requires numerous essential qualities, including flowability, buildability, and extrudability. Previous studies have shown that a combination of the materials' particle size, cement-to-aggregate ratio, quantity of admixtures, and quantity of fibres affects the quality of the printing material [5]. To ensure that a printable cementitious mixture possesses the required properties, it is vital to achieve an appropriate balance among all the concrete mixture components.

Most of the existing 3D printing concrete mixtures contains a large amount of Portland cement (PC) [3]. The reason for this high PC content in 3D printing mixtures is a result of the low aggregate content requirement [4]. High content of PC counters the cost reduction benefits of 3D concrete printing, making it unfavourable in developing countries. Additionally, PC is not environmentally friendly. The production of PC includes crushing and calcining a mixture of limestone and clay to create clinker. This mixture is heated with extremely high temperatures of roughly 1450 °C [5]. This manufacturing process is linked to significant carbon dioxide emissions, generating 900 kg of CO2 for every ton of clinker. According to earlier research, using supplementary cementitious materials (SCMs) can significantly lower cement costs and carbon emissions. Fly ash, silica fume, ground, granulated blast-furnace slag, and low-grade clays are some of these SCMs [6]. Although concrete mixtures containing SCMs like silica fume and fly ash are more environmentally friendly than those containing Portland cement, their supply is under threat in the future. For example, due to coal-based electricity generation, fly ash is abundant in South Africa, however, its long-term supply, is in doubt [6]. This is due to the fact that producing power from coal also results in enormous amounts of carbon emissions, endangering its long-term use [7], [6], [8], [9]. Compared to the typical SCMs, calcined clay and limestone stand out as excellent raw material choice, available worldwide. The availability of clay mineral-rich soils raises discussions about limestone calcined clay cement (LC3) [10]. Figure 1 shows the amounts of supplementary cementitious materials available according to Leo et al (2021) [5].

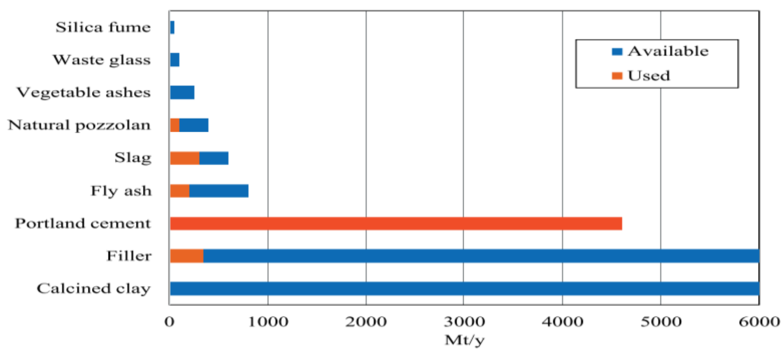

Fig. 1: Amounts of supplementary cementitious materials available and the amount of cements produced [6].

2. Bibliometric Analysis

Bibliometric analysis is a method used to evaluate and quantify available literature on a particular topic. In this study, the analysis was conducted in order identify the research that has been published on 3D printing of concrete and the use of LC3 in 3D printing, which supported the research. The analysis identified key authors, the most cited articles and journals on this topic, which helped with the development of this review. The publications on Scopus were analysed and a trend on the number of publication to date was revealed. The first analysis was performed using the key words 3D concrete printing. The analysis showed that 1,924 studies have been published on 3D concrete printing from 2010 to 2023. A second analysis focused on LC3 in 3D printing, revealing 18 publications dedicated to this topic since 2019. These 18 publications shaped the researcher's review of the impact of LC3 in 3D printing. Figure 2 (a) below shows the trend in research on 3D printing of concrete and 2(b) shows the trend in publications on LC3 in 3D printing of concrete.

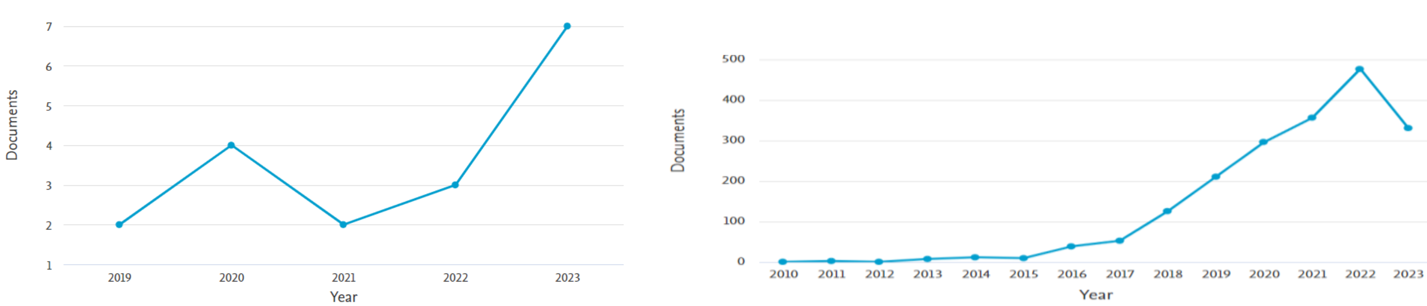

Fig. 2: (a) Trends in research on 3D printing of concrete from 2010 to 2023. (b)Trends in publication on LC3 in 3D concrete printing.

3. 3D Printing of concrete

3.1 Limestone Calcined Clay Cement (LC3) Material Properties

LC3 is a sustainable alternative to traditional Portland cement. It is composed of a blend of limestone, calcined clay, clinker and gypsum [8], [11]. The production of LC3 cement results in lower carbon emissions compared to PC because the calcination of limestone and clay requires less energy than the production of PC. The burning of clay for producing calcined clay requires relatively low temperatures of about 700 to 850°C [12]. The equipment needed for the calcination of clays is not complicated because it requires lower temperatures than clinker manufacturing. Additionally, the use of calcined clay as a replacement for some of the clinker in cement reduces the amount of carbon dioxide released during the production process. Comparatively, 1 kg of CO2 is produced during the production of 1kg of Portland cement, whereas 0.25-0.37 kg of CO2 is emitted during the production of calcined clay [13].

3.2. Calcined clay

Calcined clay and limestone are two key raw materials used in the production of LC3 concrete. According to previous studies, calcined clay improves plastic viscosity, static and dynamic yield stress, initial thixotropy index, and cohesiveness, as well as decreased harmonic distortion [14], [15]. Calcined clay improves the buildability of fresh mixtures. The irregular shape of the clay particles reduces the flowability of the mixtures. Additionally, the improvement in buildability is because metakaolin improves the yield stress and viscosity of the cement paste [16], [7]. With an increase in the metakaolin concentration from 0% to 10%, the yield stress increases 1.75 times while the plastic viscosity doubles [7]. These findings are related to metakaolin's high surface area. In addition, the study showed that increasing calcined clay content sped up the evolution of stiffness. The use of kaolinite in LC3 concrete results in a more homogeneous and crack-free structure, which is important for ensuring the long-term durability of the printed house [7]. Studies have shown that the proportion of kaolinite in the clay affects how reactive it is after being calcined [5], [17]. Kaolinite is a clay mineral which has a chemical composition Al2Si2O5(OH)4 [18]. The addition of kaolinite to LC3 concrete improves the workability, strength of the concrete, and reduces the porosity and water absorption of the printed structure [8]. According to earlier research, an LC3 combination with a 40% kaolinite content will have mechanical qualities similar to PC [12]. According to [19], among all clay classifications, kaolinitic clays have the highest pozzolanic potential. Pozzolans are alumina and siliceous materials that do not have cementitious qualities, but when they react with calcium hydroxide, they create compounds that do [20]. The production of metakaolin during the thermal activation of raw clays is what gives calcined kaolinitic clays their reactivity [21]. The metakaolin reacts with calcium hydroxide, water and sulphate to form C-A-S-H. Whenever limestone is mixed with Portland cement, calcite combines with the clinker's C3A to produce hemi-carboaluminate and monocarboaluminate, while in LC3, aluminate from the metakaolin additionally reacts with calcite to speed up the production of carboaluminate phases [17]. This occupies pores formed in cement thereby reducing the pores and permeability of binder in pores. Reducing the binder permeability reduces corrosion since harmful materials are prevented from entering the structure. The reduction of corrosion thereby increasing durability of concrete structures [20]. Previous studies showed that as the kaolinite content increased, so did the compressive strength [19].

3.3. Limestone

The utilization of limestone as a filler component in the binding material is common. The physical characteristics of limestone influences its effect on the rheological properties [17]. The limestone particles are fine and have a rough surface. These characteristics have an impact on particle packing density, which lowers the need for superplasticizer and water. Limestone's coarser fineness compared to cement's enhances workability by lowering yield stress and plastic viscosity [14], [17]. However, utilizing an ultrafine limestone can lessen the workability. This is due to the increase in inner particle friction and the significant water and superplasticizer absorption [22]. The chemistry of calcite (or dolomite), which permits hydroxyl ions (OH) to localize around the calcite ions (Ca2+) and generate interparticle electrostatic repulsion akin to superplasticizers, also contributes to the improvement in flowability [23].The substitution of limestone for PC4 enhances the nucleation sites, resulting in rapid early age hydration; however, a larger limestone content lowers the mechanical performance of hardened concrete, which is attributed to the dilution effect [24].

3.4 Optimization of LC3 Mix Design

The mix design of concrete mixtures has a huge impact in achieving optimal printability, structural integrity, and desired mechanical properties in 3D-printing. 3D concrete printing needs concrete mixtures with particular properties such as buildability, extrudability, and open time, and creating printable materials is one of the biggest hurdles. These properties are affected by the mix design. Varying the mix designs also affects the compressive and tensile strength.

4 3D printing concrete properties

4.1 Fresh state properties

The fresh properties of 3D printing concrete refer to its behaviour during the printing process before it has fully hardened. These properties are important because they can affect the quality and accuracy of the printed object, as well as its mechanical properties and durability. Some of the key fresh properties of 3D printing concrete include flowability, extrudability, buildability, open time. With the conventional construction method, formwork is used as temporary support. However, in 3DCP the construction material is deposited in layers, one on top of the other to produce the structures, without the use of formwork. The extruded material is supposed to retain the shape and also provide support to the subsequent layers that will be printed. Despite the need to formulate a concrete mixture with enough material strength in its fresh state to maintain the shape, there are other parameters such as flowability and extrudability that should be possessed by the mixture. These properties seem to be a conflicting with the buildability property [25]. Therefore, obtaining a proper balance within these parameters is a huge challenge.

4.1.1 Flowability

Flowability is the capacity of a concrete mixtures to move effortlessly within a printing system. Inadequate mixture compositions and irregular flow velocities can result in issues such as hose obstruction during printing, material seepage, and segregation [17], [26]. According to Malaeb et al., (2015) [25], flowability rate of the material should be within the range 1.0 and 1.2 cm/s, below this range the extrudability of the material is greatly compromised and above the range buildability problems will be encountered during the print. Water-cement ratio affects flowability. However, a higher water-cement ratio results to a greater void content which reduces the mechanical performance of concrete [27]. Therefore, the use of superplasticizer is usually adopted to improve the flowability of the printing material. According to Li et al., (2020) [28], concrete mixtures with water-cement ratio of 0.26 and a superplasticizer-binder ratio of 0.01 demonstrate a good flowability in a print system with a 9mm diameter nozzle. Although superplasticizers provide an increase flowability, an excessive amount reduces the buildability significantly [29]. Another element that affects concrete paste's flowability is the particle grading. A greater packing density is facilitated by a larger variety of particle sizes, which also improves flowability [30]. Li et al., (2020) [28], assert that one effective method for regulating the flowability of fresh paste is the inclusion of mineral admixtures including fly ash, silica fume, and slag. Based on the impact of particle size, shape, pozzolanic nature, and mineral admixture content, this conclusion is drawn. Spherical particles of fine mineral admixtures enhance flowability by reducing inter-particle friction and decreasing fluid demand.

4.1.2 Extrudability

Extrudability refers to the concrete mixture's ability to be smoothly pumped through the nozzle at a specified flow rate without any difficulties. Extrudability of concrete depends on the composition of the mixture, the geometry of the nozzle and the printing rate [31]. It is also affected by the particle sizes of the constituent materials. The addition of a superplasticizers and retarder can greatly increase the flowability and extrudability of the mixtures [32]. Ma et.al (2018) [30], reported that the extrudability of concrete mixtures for 3D printing depends on the particles grading. A wider range of particle size contributes to a greater packing density and results in improved extrudability. Malaeb et al., (2015) [25], reported that printable mixtures consisted of fine aggregate-cement ratio of 1.28 and a fine aggregate- sand ratio of 2.0. The study further showed that the maximum size of an aggregate should be 0.1 of the nozzle diameter. According to Le et al., (2012) [32], a concrete mixture with a maximum size of 2mm aggregates is suitable for a 9mm diameter printing nozzle. To create a printable material Wolfs et al., (2019) [33], used a siliceous aggregate with a maximum particle size of 1 mm. Buswell et al.,

(2018) [26], reported a maximum particle sizes of 2 to 3 mm. The study showed that the optimum mixture design consisted of 3:2 sand - binder ratio.

4.1.3 Buildability

The buildability is the resistance of the fresh concrete to resist deformation under loading [34]. Fresh cementitious materials can be categorized as visco-plastic. They do not flow until an external stress that exceed the material strength is induced. When the cementitious material is deposited there are opposing components that work on it, gravity and material strength. The relationship between these two parameters therefore play a huge role on the buildability aspect. In addition, parameters such as the geometry and length of print, nozzle variables, the time intervals between subsequent layers, nozzle standoff distances and printing speeds, significantly influences the buildability of the material [17]. Increasing the time interval between subsequent layers improves buildability of the concrete mixtures since the stiffness and rigidity of the deposited layer develop with time. The time interval however depends on the printing path and speed.

4.1.4 Open time

Open time is the change of concrete workability over time [25]. Open time is closely related to the setting time of concrete, thus the end goal is to ensure that each layer has the capacity to carry its self-weight, harden when poured in order to carry weight of the subsequent layer and yet stay liquid enough to bond with those subsequent layers above it. Long setting times are required to obtain a consistent flow rate of mixtures for good extrudability, therefore at times retarders are used to control the setting time of concrete. Retarders are absorbed on the surface of cement particles to form an insoluble layer, thereby delaying the hydration of cement [30]. On the contrary, shorter setting time are required to improve buildability. Accelerating admixtures are commonly used to increase early strength development of concrete. They accelerate the early age hydration of cement, thereby shortening the setting time.

4.2 Hardened properties

Compressive strength, flexural strength and interlayer bond strength are the main hardened properties in the printed concrete. More information on these parameters is given below:

4.2.1 Compressive strength

Compressive strength is the ability of hardened concrete to withstand applied loads before failure. Previous studies have shown that the compressive strength of LC3 is much lesser relative to that of PC before 7 days, thereafter the strength become similar or higher than that of PC [8], [14], [35]. The increase in water-cement ratio reduced the strength during the early age hydration. After 14 days of curing the LC3 concrete develops a similar relative strength as PC. According to the research by Patil et al., (2021) [36], a mix of 60% LC3 and 40% OPC has a compressive strength 3.98 percentage higher than that of conventional concrete. The study of Avet and Scrivener, (2018) [19] reported that the early strength of LC3 mixes was linearly correlated to the kaolinite content in the calcined clays. After 7 days the kaolinite content impact on the strength development then became insignificant. According to Scrivener et al., (2019), [8], LC3 shows higher early strength amongst other SCMs because of the presence of kaolinite. Francois et al., (2018) [19] ; Avet et al., (2016) [35], reported that the strength of all LC3 concrete mixtures at 1 day was very low compared to PC, after 3 days of curing LC3 concrete mixtures containing 95% kaolinite develops similar strength as PC and after 7 days, all LC3 concrete mixtures with 40% kaolinite content and higher showed similar or even higher strength than PC. After 28 days of curing, all LC3 concretes with kaolinite content of 17% or higher developed a higher strength than. PC. Although the effect of kaolinite on the compressive strength of concrete has been widely reported there are some conflicting conclusions on the findings. Some studies report that kaolinite has no adverse effect on compressive strength while others report significant improvements of strength. Chen et al., (2021) [37], reported that an increase in the calcined clay and limestone powder results in the decreased compressive strength. Compressive strength was found to improve with increasing clinker content and decreasing clay content [11].

4.2.2 Flexural strength

The testing of flexural strength traditionally is done using a third point loading method as per ASTM C78. In contrast to conventional methods of construction, 3D printing, particularly extrusion-based techniques, involves layer by layer concrete placing which introduces interlayer bonds to the final printed structure. This printing process has the potential to induce mechanical anisotropy to the structure. Previous studies tested specimens both perpendicular and lateral directions [38]. Findings revealed that the flexural strength of the specimens that underwent testing in the perpendicular direction was greater than that of the specimens that underwent testing in the lateral direction. The findings indicate that the flexural strength results of the perpendicular direction showed 3% to 16% increase compared to that observed in the lateral orientation. Additionally, Moelich et al., (2021) [38], reported that the flexural strength values of printed specimens decrease in comparison to the samples cast in moulds. This can be attributed to the effect of compaction. Additionally, the rate of evaporation was reported to impact the flexural strength. Inclusion of polyethylene fibres up to 2% resulted in the increase of the flexural strength [39].

4.2.3 Interlayer bond strength

Interlayer adhesion is a potential weak point in 3DCP due to the layer-by-layer printing process [17]. Interlayer bond strength is the adhesion that results as an interaction of the subsequent printed layers. Printing parameters including the environmental conditions and time intervals between subsequent layers can affect interlayer bond strength [17]. Although buildability can be improved by increasing the time, on the contrary the interlayerbond strength may be significantly reduced resulting in cold joints [26], [40], [41]. Additionally, the time it takes to overlay layers has a significant impact on the interlayer bond strength and may encourage cold jointing [4]. This weak interface may increase the air pores which then allows permeability of corrosive materials therefore time interval should also be sufficiently small in order to eliminate this effect. According to Tay et al., (2016), there is a correlation between the interlayer bond strength and the environmental conditions such as temperature and humidity. Temperature and humidity affect the drying rate of deposited materials. Because the concrete mixtures are usually mixed with accelerator or admixtures to improve their buildability, this results in increase in temperature due to faster chemical reactions (hydration). This increases drying rate and make the material extremely sensitive to the external environment conditions [17], [42].

5 Conclusion

The study reviewed the use of LC3 for 3D printing, concrete properties requirements for 3D printing and optimum LC3 mix designs from previous studies. The review shows that Calcined clay improves plastic viscosity, static and dynamic yield stress, initial thixotropy index, and cohesiveness. Increasing calcined clay improves the buildability of the concrete mixtures while reducing its flowability and extrudability. However, Limestone improves flowability and extrudability of concrete mixtures, hence the combination of limestone and calcined clay (LC3) produce a synergy that support 3DCP properties. Also, the addition of kaolinite to LC3 concrete improves the strength of the concrete. Finally, there is paucity of information with regards to the optimum LC3 mix design for 3D printing. The author recommends additional laboratory works to determine the optimum mix design.

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