

AN EXPERIMENTAL STUDY ON PROPERTIES OF CONCRETE BY USING TOPCRETE

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ABSTRACT: Global warming, which is mainly caused by mankind with the emissions of greenhouse gases, is nowadays one of the major topics in the world. The reduction of these greenhouse gas emissions has become a primary focus of the environmental organizations and the government in many countries. Since Portland cement production is accompanied by a significant CO₂ emission, which is a principle greenhouse gas, it is useful to look for ecological alternatives of cement. Portland cement is normally replaced with fly ash or blast furnace slag cement, which are both byproducts from other industrial waste. The sources of these byproducts are limited. Therefore, seeking for other alternatives of Portland cement is significant from both economic and environmental point of view. In this research the use of thermally activated paper sludge minerals as partial Portland cement replacement is investigated. The replacement of cement with paper sludge minerals is proposed to reduce the environmental pollution that are associated with the concrete productions and with the disposal of paper waste. By calcination and dehydration of paper sludge, which is a by-product of recycled paper waste, a non-poisonous mineral with a highly pozzolanic activity is formed. Besides the pozzolanic activity this mineral also shows hydraulic potential. Therefore, the production and usage of thermally activated paper sludge minerals has attracted increasing interest due to its environmental advantages and excellent cementitious potential.

The use of Top-Crete as a partial cement replacement is a relatively new research area. The cement paste and mortar mixtures have been composed with TC ranging from 0%,10% & 15%, with water to binder ratio of 0.45.

The particle size distribution of TC is slightly finer than that of PC and FA. TC has a specific surface area of 7.2262 m²/g, which is about 27 times higher than PC. The ignition loss of TC is 21%, which mainly arises from the release of carbon dioxide by the thermal decomposition of the large CaCO₃ content. Knowledge on these physical

properties will increase the ability to explain the influence of TC on various properties of blended cement pastes. Experiments have been performed on the cement paste and mortar mixtures to gain more insight in the effect of TC on the properties of blended cement pastes and mortars. The properties include workability, setting time and compressive strength. These properties are tested according to the procedure outlined in the Indian standards.

TC hydrates compensate for the chemical shrinkage. It was also observed that the replacement of PC with 10% & 15% TC substantially increases the autogenous shrinkage of the blended cement paste compared to that of the control paste. This attributed to the acceleration of the PC hydration, the highly pozzolanic reaction of TC and the refinement of porosity by TC. The first two contribute to the removal of water from the specimen which results in self-desiccation of the specimen. While the refinement of the porosity results in the increase of the capillary tension.

Overall, from this research, it has been found that a certain percentage of TC can act as a replacement of cement without significant loss in strength and with a considerable shorter setting time. Replacing cement with 10 % TC in blended pastes.

INTRODUCTION

Concrete is the most commonly used building material in the world. Its huge popularity is a consequence of several advantages, such as general availability, wide applicability and low cost. These advantages are also accompanied by a great environmental burden. The billions of tons of raw materials mined and processed each year leave a mark on the environment. Furthermore, during the production of Portland cement large quantities of CO₂ are released into the atmosphere and enormous

amounts of energy are required. Portland cement is one of the most important ingredient of concrete.

Top-Crete is formed by calcination and dehydration of the minerals in paper sludge, including kaolin clay and calcium carbonate, at a specific temperature and time range. When calcining paper sludge at 700 to 750 degrees after 2 or 5 hours a very reactive metakaolin is created. Metakaolin reacts with CH and creates a dense structure with low porosity, improving the properties of paste, mortar and concrete.

The objectives are:

- To investigate the early age properties and mechanical properties of the cement paste and mortar made with addition of Top-Crete.
- To define the amount of free lime in the cement paste before and after hydration and explain what that will mean for the concrete durability.
- To investigate the chemical and autogenous shrinkage of the blended cement pastes with TC.

Scope

- There are many factors that influence properties of the blended cement paste. The most important parameters that may affect the composition of the samples are the water to powder ratio, the amount of Top-Crete, admixtures, curing temperature and curing age.
- - Water to powder ratio of 0.45
- - Curing temperature of 20 °C.
- All parameters will stay constant, except the amount of cement, Top-Crete, otherwise it would be too complicated and too difficult to compare the experimental results and conclude which parameter is responsible for which effect.

LITERATURE STUDY

Portland cement

Portland cement is a rapid- curing binder which was first fabricated in Great Britain in the early 19th century. The name Portland is derived from the Portland formation, a layer of rocks with the same properties, from which Portland stone was mined. Portland stone is a white sandy limestone.

Portland cement clinker is obtained from the calcination of calcium carbonate (limestone or chalk) and aluminosilicate (clay or shale) in a kiln at a temperature of approximately 1450 °C. Partial fusion occurs and nodules of clinker are produced. The clinker, containing CaO, SiO₂, Al₂O₃, Fe₂O₃ and small quantities of other oxides, is mixed and grinded with gypsum to make the cement. Gypsum will slow down the hydration process and thus improve the workability. The mineral compounds of Portland cement are always the same, but the proportions can be different

Oxides	Percentage content
CaO	60-67
SiO ₂	17-25
Al ₂ O ₃	3.0-8.0
Fe ₂ O ₃	0.5-6.0
MgO	0.1-4.0
Alkalies (K ₂ O,Na ₂ O)	0.4-1.3
SO ₃	1.3-3.0

Clinkers

The components of the clinker consist of dicalcium silicate (C₂S), tricalcium silicate (C₃S), tricalcium aluminate (C₃A) and tetra calcium aluminate ferrite (C₄AF). The relative composition of the clinker depends on the temperature and the relative proportions of the raw materials. This composition can easily be determined with the phase diagram, see figure 2.1, and with the equations given by Boogie. The chemical formula represents the mass percentage of each oxide.

$$C_3S = 4.07 (CaO) - 7.60 (SiO_2) - 6.72 (Al_2O_3) - 1.43(Fe_2O_3) - 2.85(SO_3)$$

$$C_2S = 2.87 (SiO_2) - 0.754 (3CaO.SiO_2)$$

$$C_3A = 2.65 (Al_2O_3) - 1.69 (Fe_2O_3)$$

$$C_4AF = 3.04 (Fe_2O_3)$$

Composition of Top-Crete

After the calcination and dehydration of paper sludge, the resulting ash (top-Crete) is composed mainly of metakaolin (silica and alumina), free lime, calcium carbonate and inert filler. The characterization is carried out by CDEM.

The mineralogical composition of Top-Crete	
Mineralogical composition (%)	
Calcium carbonate	41
Metakaolin	29
Free lime	23
Inert fraction	7

One of the most important properties for a waste product to be accepted and used as a mineral in composite cement is its pozzolanic nature. The metakaolin in TC contributes to the pozzolanic properties of TC, while the free lime in TC contributes to the hydraulic properties of TC.

MATERIALS

CEMENT:

Ordinary Portland cement of 53 grade from a single batch was used for entire work and care has been taken it has to be stored in air tight containers to prevent it from being affected by the atmospheric and monsoon moisture and humidity. The cement produced was tested was physical requirements in accordance with IS: 4032-1977.

Tests:

- Fineness of cement
- Initial and final setting time of cement
- Soundness of cement
- Specific gravity of cement

AGGREGATES:

An Aggregate generally occupies 70% to 80% of the volume of the concrete and can therefore be expected to have an important influence on its properties. They are granular materials derived from the most part from natural rock and sands.

In addition with their use as economical filler, aggregate generally provide concrete with better dimensional stability and wear resistance. Based on their sizes aggregates are divided into coarse and fine aggregates. The coarse aggregate fraction is retained on 4.75mm sieve and, while the fine aggregate fraction is passing through 4.75mm sieve. Based on their origin aggregates are classified into natural aggregates and non- natural aggregates.

NATURAL AGGREGATES:

A mineral aggregate consists of sand and gravel, stones and crushed stones. Construction aggregates make up more than 80% of aggregates market, and are mainly used for road base, rip-rap, cement concrete and asphalt. In 1998 roughly 3500 U.S quarries produced about 1.5 billion tones crushed stone, of which 1.2 billion tones was used in construction applications. The sources of mineral aggregates are by directly extracting from the original basis like river basins or by manufacturing them into a desired shape from a parent rock in a crusher mill. It was also found out that manufactured offers a viable alternative to a natural sand by providing a higher compressive strength and delivering the environmental benefits.

All natural aggregates are originally formed as part of a larger parent mass. This may have been fragmented by natural process of weathering and abrasion or artificially by crushing. Thus, many properties of the aggregates, depend entirely on the properties of the parent rock, e.g. chemical and mineral composition, specific gravity, hardness, strength, physical and chemical stability, pore structure, and color. On the other hand there are some properties possessed by the aggregate but absent in the parent rock those are particle shape, size surface texture and absorption. All these properties may have a considerable effect on the on the quality of the concrete, either in fresh and hardened state.

NON-NATURAL AGGREGATES:

This category consists of aggregates that are artificial in origin. The reasons for their advent in concrete constructions are:

- Environmental considerations are increasingly affecting the supply of aggregate.
- At the same time there are problems with the disposal of construction demolition waste
- At the same time there are problems with the dumping of domestic waste

However these types of wastes can be processed into aggregate for the use in concrete and this is increasingly being done in the number of countries, for example in the Netherlands. A wide variety of

materials comes under the heading of solid wastes. These wastes range from municipal and household garbage

COARSE AGGREGATES:

Throughout the investigation, a cured coarse aggregate of 20mm produced from local crushing plants was used. The aggregate was tested for its physical requirements such as gradation, Fineness modulus, Specific gravity etc. in accordance with IS: 383-1970.

The size of aggregates bigger than 4.75 mm are called coarse aggregates. The aggregates passing through 20mm size sieve and retained on 12.5 mm size sieves. The size of aggregate used for has a great impact on the strength of concrete



Coarse aggregates

Tests:

- Specific gravity of coarse and fine aggregates
- Sieve analysis of fine and coarse aggregate
- Bulking of fine aggregate
- Elongation index for coarse aggregates
- Flakiness index for coarse aggregates

WATER:

Fresh portable water free from organic matter and oil is used in mixing the concrete. Water is required qualities where measured by graduated jar and added

to the concrete and rest of the materials were preparation of the concrete mix were taken by weigh batching. The P^h value should not be less than 6.

TESTS ON FRESH CONCRETE

1. Workability
2. Slump cone test
3. Compaction factor test
4. Vee-bee test

CONVENTIONAL CONCRETE MIX DESIGN

Mix proportion:

Cement: fine aggregate: coarse aggregate

425.74: 480.10 : 923.83

1: 1.12: 2.17

Hence the final mix proportion = water: cement: F.A:
C.A = 0.45: 1: 1.12: 2.17

Fabrication of Specimen

In this project total 20 specimens are prepared in a mould 100 x 100 x 100mm for compression test and 300 x 150mm diameter mould for tensile test & 150 x 150 x 700mm for flexural tests. The specimens are filled by concrete by 3 layers of moulds each layer tamped by 25 strokes using damping rod. After the specimens are stored in a room temperature for 24 hours.

After completion of 1day the specimens are taken out from moulds and placed in a curing tub for 3, 7, 21 and 28 days.



CURING



TESTING OF SPECIMEN

- **COMPRESSIVE STRENGTH TEST ON CONCRETE SPECIMENS**





HEAT OF HYDRATION

Various methods, such as isothermal and adiabatic test setups, are available for measuring the hydration heat. Also different approaches are used to determine the degree of hydration. In this study, an isothermal calorimeter as shown in figure 9 was used to determine the hydration heat and the degree of hydration of the blended cement pastes. The measurements of the heat evolution of the blended cement pastes have been carried out with a thermometric isothermal conduction calorimeter (TAM- Air-314). The calorimeter contains eight separate channels for the hydrated samples and eight channels for the un-hydrated reference sample; each holds a single specimen during the measurement. The reference has been used to correct for minor temperature fluctuations. The calorimeter monitors continually the heat evolved from the specimen with time when the temperature of both the specimen and the surrounding environment are maintained at isothermal conditions



Determination of hydration heat and degree of hydration

After the measurement of 7 days, the rates of heat evolution per gram binder were calculated from every mix design. The average value from four samples of each mix was used for the results. The determination is presented below:

Heat evolution:

The heat that was evolved during a reaction was conducted through the thermo sensors. The heat conduction is converted to a voltage signal (mW/g) proportional to the amount of heat flow as function of time. Knowing the calibration factor used in the beginning of the measurement, allows conversion of the voltage signal to the rate of heat evolution (J/g).

$$\alpha(t) = Q(t) / Q_{pot}; [-]$$

Integration of the areas under the rate of heat evolution curves allowed calculation of the total heat of hydration. \int

Where $Q(t)$ is the total thermal energy [J/g], $P(t)$ is the thermal power [mW/g] and t is time [h].

Degree of hydration

The development of the degree of hydration of the blends at a specific time t can be estimated by the ratio between the generated heat at time t and the total heat of the binder:

Where $\alpha(t)$ is the degree of hydration at age t , $Q(t)$ [J/g] is the accumulated generated heat at time t during the hydration process, t [h] is the time that is converted to an age that expresses the maturity of the

material during the hydration process, and Q_{pot} is the potential heat produced by the different materials separately. This can be defined with the material components and chemical compositions. Consequently, the potential heat is determined using the following formula:

$$Q_{Pot} = P_{cem} \cdot Q_{cem} + P_{poz1} \cdot Q_{poz1} + P_{poz2} \cdot Q_{poz2}$$

Where P_{cem} and $P_{poz1,2}$ (%) are the mass percentages of the materials and Q_{cem} and $Q_{poz1,2}$ are the potential heat of the cement and pozzolana.

Sample preparation and curing

After mixing the samples as described that the cement paste was poured into 500 ml plastic bottles. The bottles were sealed with a plastic lid and placed into a rotational machine to prevent bleeding and segregation in the cement paste. The samples were rotated for 24 hours at a speed of 5 rotations per minute. The environment had a temperature of 22 ± 2 °C. After 24 hours the samples were removed from the rotational machine and restored in a room with a temperature of 20 ± 2 °C until the age of testing. The samples were tested after 3, 14, 28 and 56 days. At the end of each curing period, the plastic bottles were broken and the samples were split up in small pieces of 1 cm³, see figure 12. Only the inner core of the sample was used for testing, because this part of the sample has been least affected by the environment. This preparation procedure is the same for other testing methods, like Thermo Gravimetric Analysis (TGA) and environmental scanning electron microscope (ESEM). Therefore, one sample has been prepared and used for all the three experiment.



Drying procedure:

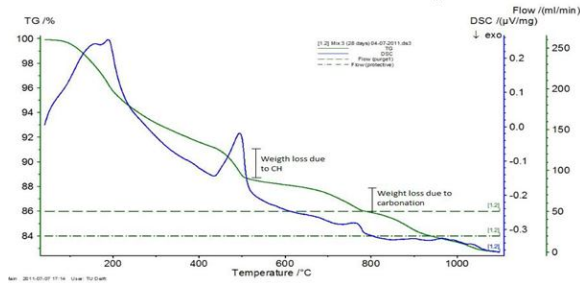
The small pieces were exposed to the air during the breaking of the samples. To minimize the formation of calcium carbonation, these pieces were immediately frozen by immersion in liquid nitrogen for 2 minutes. Freezing of these small pieces is necessary, not only to prevent the formation of carbonation, but also to stop further hydration. The frozen water is removed from the samples by freeze-drying. Ye Guang stated that the freeze-drying method minimizes the damage¹²⁹

on the pore structure in comparison with other drying methods, such as oven drying and vacuum drying. The samples had to be dried in a freeze-dryer with a temperature of -24°C and a vacuum at 0.1 Pa. This drying procedure takes between 10 to 20 days until a stable mass loss of 0.01%/day was reached. Depending on the amount of water used for mixing and the curing age.

Chemical bound water and CH content

Experimental procedure

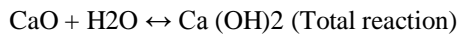
TGA was carried out using a NETZSCH Simultaneous Thermal Analyser TG-449-F3-Jupiter. The experimental apparatus is shown in figure 18. An alumina fine powder and placed in the machine in a micro furnace. Nitrogen has been used



Weight loss as a function of temperature changes. DSC and TG curve measured from thermal gravimetric analysis

Three parameters can be determined from the TG curve. These parameters are the chemical bound water, the weight loss due to dehydration of CH and the weight loss due to calcium carbonation. The loss due to CH dehydration is represented in the figure as an abrupt weight loss between 400°C and 500°C and a peak in the DSC curve. A smaller weight loss between 700°C and 800°C corresponds to the chemically bound water

The CH amount can be defined by the following chemical reaction, after the graphical method has been used:



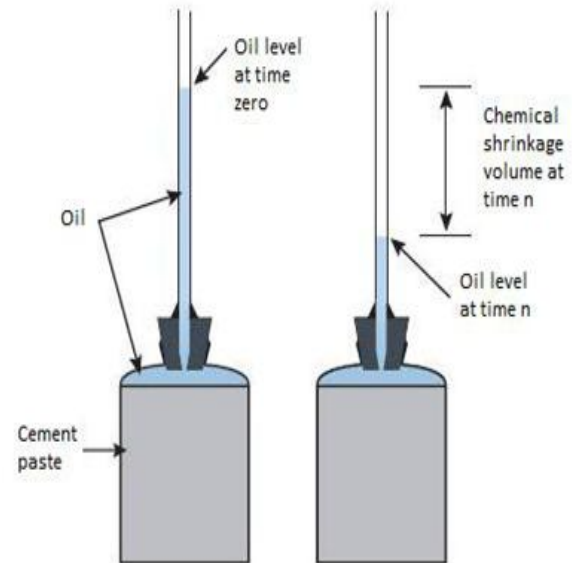
The total reaction shows that for each mole CaO one mol H₂O is added and one mole Ca(OH)₂ is created. The molmass of water is 18,015 g/mol and the molmass of CaOH₂ is 74,1 g/mol. The amount of water that reacted with calcium in the samples is determined with TGA.

Chemical shrinkage

Procedure:

In this method, 50 g of each mixture was blended with water in a cup using a small blender. About 8 g of this freshly blended cement paste was placed at the bottom of a small cylindrical glass jar, with a diameter of 25mm and a height of 60 mm. Three small glass jars were used for each mixture. After the blended cement paste was placed at the bottom, the specimen were vibrated for a couple of seconds to remove air bubbles in the paste and to obtain a uniform thickness spread over the bottom of the jar. The pastes were then covered with 0.8 ml of water to include a water source in contact with the paste. The remaining empty part of the jar was filled with

paraffin oil and enclosed with a rubber stop encasing a pipette graduated in 10 ml increments. The change in oil level within the pipette indicates the volume change due to chemical shrinkage.



Autogenous shrinkage

Procedure

After mixing of the specimen, the blended cement paste was immediately cast under vibration into three corrugated tubes. Each tube was closed with one plug and put with the closed end on the vibration table in a vertical, rigid support tube. The pourable mixtures were slowly poured in the corrugated tubes without stopping, while the semi- fluid mixtures were poured in layers. Each layer was compacted for a couple of seconds by the vibrating table. Then the open ends of the tubes were closed with another plug and placed on a horizontally supporting corrugated plastic sheets to avoid damage to the microstructure (see figure 22). All specimens were stored in a room with controlled environment of $20 \pm 2^\circ\text{C}$ and $60 \pm 5\%$ RH throughout the duration of the tests, to minimize the influence of temperature fluctuations. The restraining amount of the mixture was cast into a setting mold and simultaneously measured with the autogenous strain. The setting of the blended cement paste was determined with the vicat needle test. Only this time the specimen were covered with foil to prevent moisture loss during the experiment.

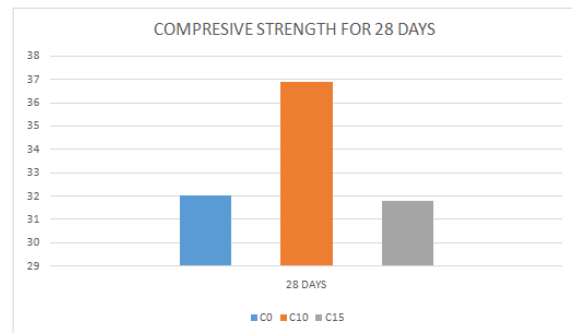
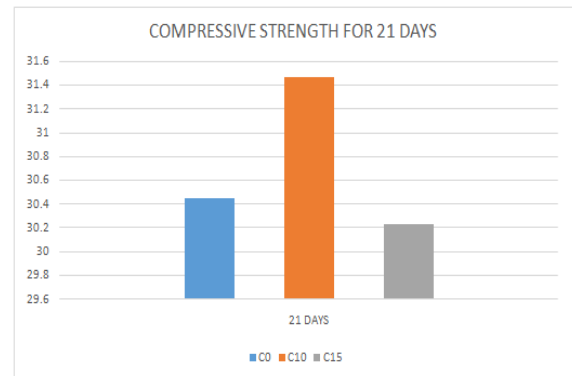
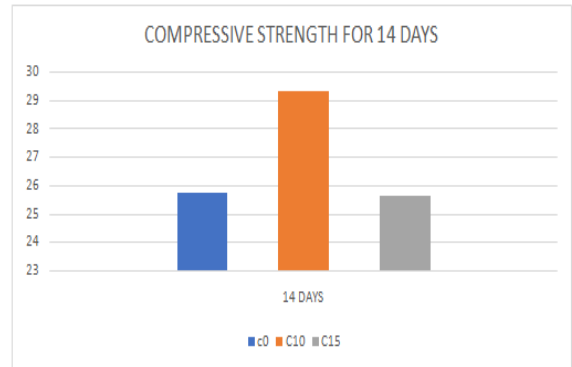
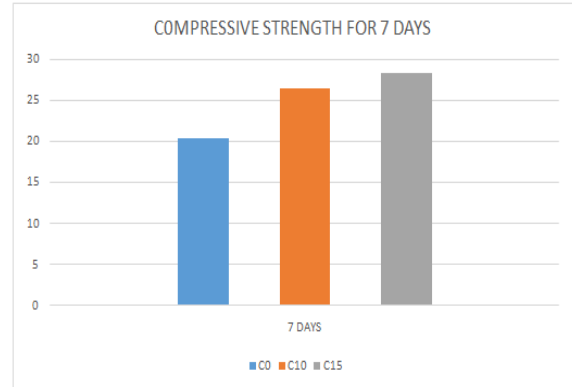
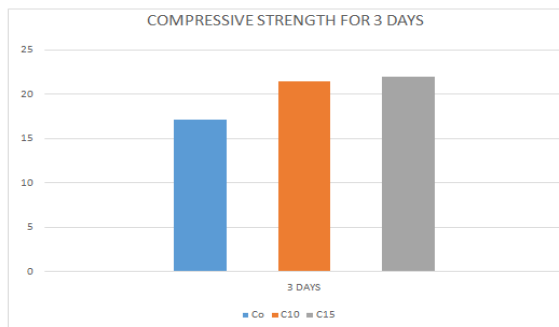


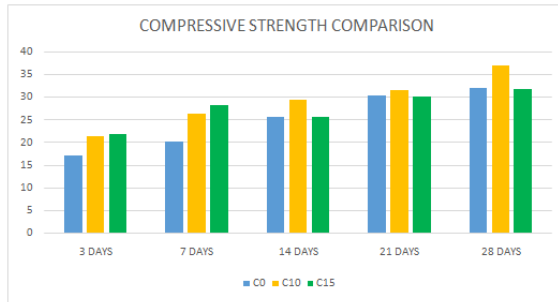
Corrugated plastic sheets supporting the specimen

RESULTS AND ANALYSIS

COMPRESSIVE STRENGTH TEST ON CONCRETE SPECIMENS

NO.OF DAYS	CONCRETE (N/mm ²)	TOPCRETE (10%) (N/mm ²)	TOPCRETE (15%) (N/mm ²)
3 days	17.1	21.4	22
7 days	20.3	26.4	28.3
14 days	25.7	29.3	25.6
21 days	30.45	31.47	30.25
28 days	32.05	36.86	31.8





CONCLUSION

- The specific surface area of topcrete is 27 times greater than cement. The workability of topcrete has decreased compared with ordinary concrete. It is inferred that reduction in workability is due to the large surface area of topcrete.
- The compressive strength of concrete (with 0%, 10%, 15% weight replacement of cement with topcrete) cured in water for 3, 7, & 28 days indicates that the compressive strength is nearly equal to that from the replacement percentage of 15% compared with normal concrete.
- The compressive strength values increase when compared to 3, 7 & 28 days curing in water.
- The replacement of cement with 15% of topcrete makes the cubes act as a brittle material when compared to 5% & 10%.
- The compressive strength of topcrete (15%) increases for 3, 7 days and decreases for 14, 21 & 28 days respectively.
- By using topcrete it acts as an admixture and changes the properties of workability. As per the literature review, the properties of shrinkage and heat of hydration have changed by using topcrete.

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