Influence of Illitic Calcined Clay on Properties of Mixtures for 3D Concrete Printing

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Challenges in Cement Industry

- Severe influence of **CO_2 factor** on feasibility of clinker production

Opportunities

- Capturing and depositing of emitted CO_2
- **Reduction** of cement **consumption** in concrete
- Use of **SMCs** to replace OPC in blended cements
  - Natural (pozzolanas, calcined clay, limestone)
  - Side-products from other industries (Blast furnace slags, silica fume, fly ash)

Calcined clay as an optional SCM

Abundant raw material, clay, available throughout the world, which after thermal treatment at 600^o to 900^o C at the process of dehydroxylation give a material with pozzolanic properties. Different clays show different pozzolanic activity.

- Kaolinite clays, having good pozzolanic activity, have proven as a component in LC3
- Illitic clays, having less pozzolanic activity, need to be investigated

The aim of the research

Analyse the influence of different amounts and fineness of calcined clay additive in the ternary binder with OPC and LF on the properties of 3DCP mixtures.

Materials used in the study

A. Binder’s components

- C4 - Portland cement, CEM I 42,5R, Blaine 410 m^2/kg , C_3S – 58%, C_2S – 15%, C_3A – 8%, C_4AF – 10%
- LF - Limestone filler R0, Blaine 250 m^2/kg, D_{50} – 25 μm)
- CC - Calcined illitic clay at 900° C, Blaine 464 m^2/kg D_{50} – 33 μm (CC) and 1033 m^2/kg D_{50} – 8 μm (CCP)

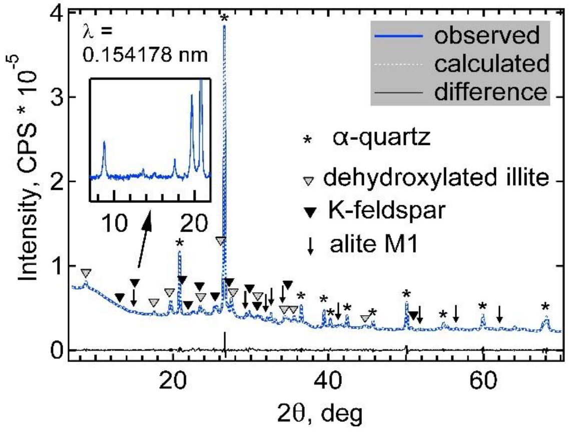
Chemical composition by XRF

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	L.O.I.
C4	19.2	4.7	2.9	61.9	3.9	3.4	0.9	0.3	2.2
CC	56.1	19.1	8.4	4.4	2.6	1.0	6.8	0.1	na
LF	11.4	2.6	1.6	41.0	3.1	0.7	1.3	0.1	37.9

Phase composition of calcined clay by XRD, Smart Lab TM (Rigaku)

Crystalline (30%) – Quartz (18.0%); Dehydroxylated illite (3.0%); K-feldspar (5.0%);
Iron containing minerals (2.0%); Anhydrite (1.0%); Alite/belite (1.0%)

Amorphous (70%)



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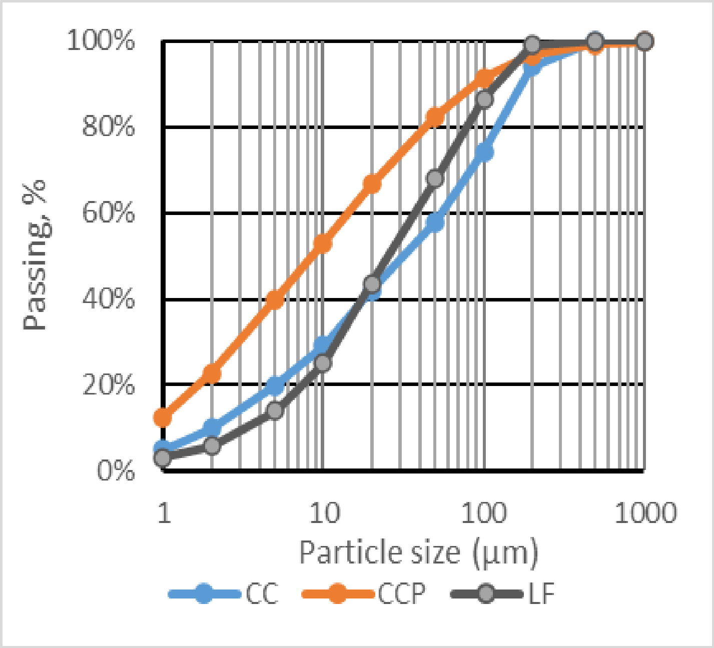
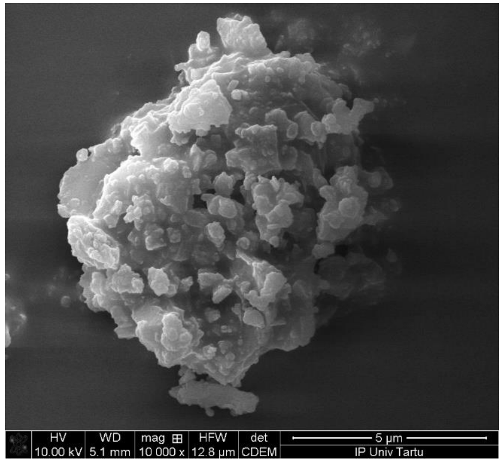
Particle size distribution of components by Laser Diffractometry Mastersizer 3000



Morphology and microstructure of calcined clay by SEM Helios Nanolab 600 (FEI)



Agglomerated particle of CCP

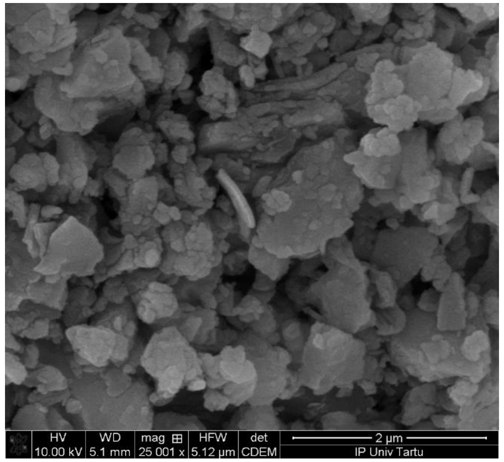


Plate-like particles of meta-illite

Materials used in the study

B. Aggregates and admixtures to the dry mix

- Quartz sand from EMG Kuiv Liiv, fraction 0-0.5mm (SF) and 0.5-1.2mm (SC)
- Printing mix modifying admixtures
  - Superplasticizer (SP) - Polycarboxylate ether PCE (up to 0.05% from the binder mass)
  - Viscosity modifying admixture (VMA) – hydroxypropyl methylcellulose Hpmc (up to 0.1% from the binder mass)
  - Fibres (PF) - Polypropylene fibres 6mm (up to 0.1% from the binder mass)

A semi-industrial 3DCP mixture produced with C4, LF, two fractions of quartz sand SF/SC at 60/40% and printing modifying admixtures in Mira Ehitusmaterjalid OÜ was used as a basic mixture.

Dry mixes with different amounts of calcined clay of two fineness (CC and CCP) were prepared in 2,5 kg batches in Hobart planetary mixer by homogenizing 4 min. at low speed.

Testing methods

Mixing in batches of 2,5 kg with water at W/M 18% took place in Hobart planetary mixer for 2 min. at low speed, and after pause for scraping the walls, for another 1.5 min. at low speed.

Compositions of ternary blends and water ratios of the concrete mixes at W/M of 18%

Symbol	Binder (B), %				Sand (S), %		Water ratios		Sand/binder ratio
	OPC	LF	CC	CCP	SF	SC	W/B	W/C	
MC4L15	62.5	37.5	0	0	60	40	0.5	0.72	1.5
MC4L15+CC5	55	35	10		60	40	0.42	0.76	1.33
MC4L15+CC10	50	30	20		60	40	0.39	0.80	1.17
MC4L15+CC15	44	26	30		60	40	0.37	0.84	1,04
MC4L15+CCP5	55	35		10	60	40	0.42	0.76	1.33
MC4L15+CCP10	50	30		20	60	40	0.39	0.80	1.17
MC4L15+CCP15	44	26		30	60	40	0.37	0.84	1.04

A Hägermann cone flow of fresh mixture after 15 jolts was performed according to EN 1015-3.

Specimens 40x40x160mm for estimating flexural and compressive strength were prepared and tested at the age of 1 day and 28 days of curing at 20^{o}C and 90% RH according to EN 1015-11.

Mixtures for technological tests were prepared using printing facility set up in TTK UAS.

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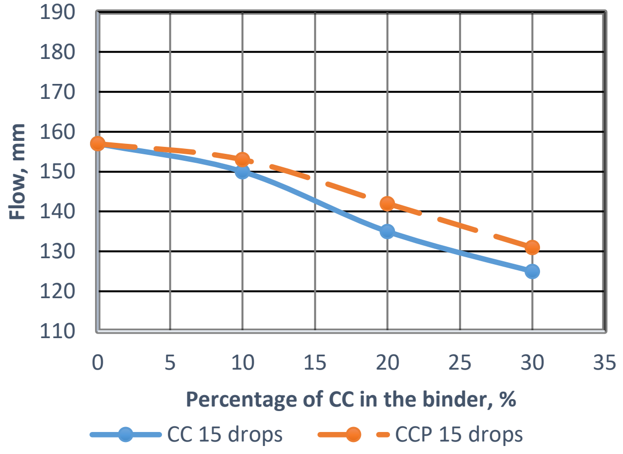
Performance of calcined clay in combination with limestone filler in ternary binders on properties of 3D concrete printing (3DCP) mixtures

Properties of fresh mixtures

Flowability – expresses plastic viscosity, shear stress, static and dynamic yield stress of fresh mixture. Measured by the flow of the Hägermann cone (EN 1013-5) after 15 drops. W/M ratio 18%.

Influence of calcined clay of two fineness classes CC and CCP on the flow of fresh mixture



Flow decreases with the increase of amount of calcined clay in the binder. It can be explained by:

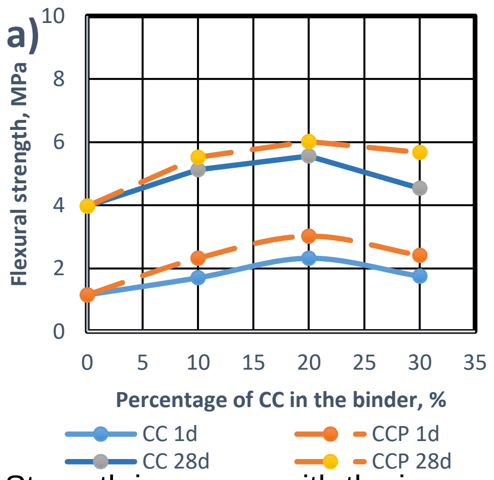
- Increase of content of fine particles in the mixture (decrease of S/B ratio)
- Enhanced absorption of mixing water by CC particles of flaky morphology

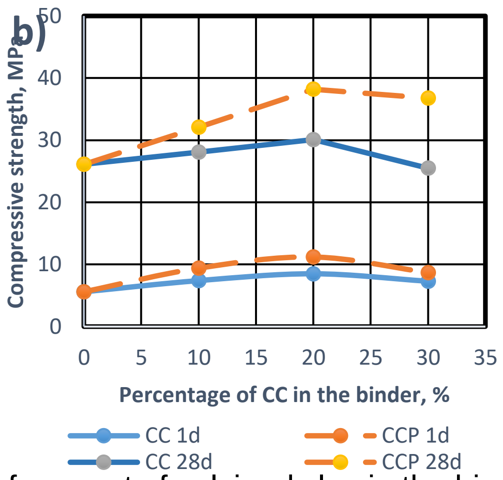
Properties of hardened mixtures at the age of 1 day and 28 days after curing at 90%RH and 20^{0}C

Specimens of 40x40x160mm, prepared by filling the moulds in 2 equal portions and compacting after each filling by dropping the mould from the height of about 20mm 10 times against a concrete floor.

Influence of calcined clay of two fineness classes CC and CCP on the strength of mixtures at the age of 1 day and 28 days

a) Flexural strength, MPa b) Compressive strength, MPa





Strength increases with the increase of amount of calcined clay in the binder and after maximum is achieved (at 20%), starts to decrease, caused by:

- Decrease of W/B ratio with the increase of binder content in the mixture. CC over 20% dilutes the mixture (share of OPC decreases)
- Better packing density in case of finer calcined clay (CCP)

Technological tests were conducted at TTK UAS, where a Gantry-type of printing facility is set up.

Influence of calcined clay on technological parameters of mixtures for 3D concrete printing

Mixtures MC4L15+CC5 and MC4L15+CC10 were prepared in Soroto 100 L-30 mixer by dry-homogenizing of 75 kg batches of basic semi-industrial 3DCP mixture and calcined clay during 4 min and following mixing with water (W/M – 18%) during 4 min before poured into Spraytec conveying pump and delivered to the printing nozzle by a 11,5m long hose of DN 38mm. Flow of the mixture was measured and kept between 140-150mm which was suitable for fluent printing.

The following parameters were analysed:

- Extrudability – seamless filament production, expressed by **solidity ratio (SR),** dependant on material flow rate Q (ml/s), nozzle size A (mm^2) and speed v (mm/s). At measured flow rate of 108 ml/s, nozzle size of 804mm^2 and printing speed of 133 mm/s the solidity ratio was 1,01 which ensured required filament quality. Resulted filament dimensions were measured - width b=47mm and height h=14,5mm.
- Buildability – ability of deposited filaments to retain its geometry under the load from upper layers. A wall element with a contour length of 9688mm was printed and its parameters measured before elastic buckling occurred. The height of the wall element (H = 236mm) and number of layers (n = 16) was achieved in case of the mixture MC4L15+CC10. In case of MC4L15+CC5 corresponding figures were H = 189 and n =13.

Mixture with higher content of calcined clay showed somewhat better buildability because of additional flocculation centres in the mixture, provided by finely ground calcined clay with flaky morphology.

Printed wall element for experimental 3D printed building

Concluding remarks

The study leads to the following findings

- The flow of the fresh mixture decreased with the increase of the content of calcined clay in the binder. It can be attributed to the enhanced absorption of mixing water by the fine-grained particles of calcined clay of flaky morphology and increase of finer fractions in the mixture (expressed lower by S/B ratio).
- The strength of the hardened mixture increased with the increase of content of calcined clay in the binder up to 20% after which it starts to decrease. It can be explained by the decrease of W/B ratio due to the increase of binder content in the mixture. CC content over 20% dilutes the mixture (share of OPC decreases). Higher strength, especially at the age of 28 days can be attributed to the better packing density in case of finer calcined clay (CCP).
- Suitability illitic calcined clay with less pozzolanic activity in 3DCP mixes is based on availability of additional flocculation centers, provided by finely ground calcined clay of flaky morphology.

Thank you !