

Durability of High Strength Self Compacting Concrete with Metakaolin Containing Waste

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Abstract. Metakaolin is considered as one of most promising pozzolanic microfiller material in concrete industry. Metakaolin is a high value product obtained from kaolin clay calcined at high temperatures which also can be effectively used in ceramic industry therefore its application in concrete industry is rather limited. In present research metakaolin containing waste (MKW) by-product was studied as a partial cement replacement in high strength self compacting concrete (SCC). Obtained waste material derives from the foam glass granule production plant where kaolin clay is used as releasing agent during heating process and in the end metakaolin with glass impurities is obtained as by-product. In present research 5 to 15 wt.% of cement was replaced by MKW. A constant water amount was used for all mixtures and workability (>600 mm by cone flow) was ensured by changing the amount of superplasticizer. Compressive strength was tested at the age of 7, 28 and 180 days. To determine durability of SCC the chloride penetration was tested according to NT BUILD 492, freeze-thaw test according to LVS 156-1:2009 annex C and alkali-silica reactivity test according to RILEM TC 106-AAR-2. The results indicate that cement replacement by MKW did not affect the strength of SCC significantly. At the age of 28 days SCC with 15 wt.% of MKW reached compressive strength of 70 MPa comparing to 68 MPa to reference mixture. The chloride penetration test results indicated that the non-steady-state migration coefficient of reference samples was reduced 3.7 times and it was concluded that SCC resistance to chloride penetration can be increased by incorporation of MKW in mixture composition. Freeze-thaw test results indicated that obtained SCC can withstand at least 500 freeze-thaw cycles without surface damage and weight loss. It was concluded that up to 15 wt.% of cement can be replaced by metakaolin containing waste without strength loss and the durability of SCC could be increased.

Introduction

Ordinary self compacting concrete (SCC) is associated with high binder and chemical admixture content therefore there is attempts to produce SCC with high volumes of supplementary cementitious materials like silica fume and metakaolin to make SCC cost effective and more durable [1]. Metakaolin can be obtained by the endothermic reaction of kaolin as dihydroxylation of kaolin begins at 450 °C and continues up to 900 °C and amorphous metakaolin $Al_2Si_2O_7$ is obtained while exceeding 925 to 950 °C the metakaolin transforms to spinel $Si_3Al_4O_{12}$ and mullite at 1050 °C [2]. Metakaolin proves to be one of most effective available pozzolanic material because metakaolin reactivity is 1050 mg $Ca(OH)_2/g$ pozzolan compared to 427 g of silica fume and 875 g of fly ash respectively [3]. However the incorporation of metakaolin in concrete mixture could reduce workability of traditional concrete while comparing to silica fume additive, metakaolin requires from 25 to 35 % less superplasticizers [4].

Metakaolin can be used as pozzolan in lime mortar therefore contributing to the amount of hydrated paste in mortar and besides calcium-silicate hydrates gehlenite C_2ASH_8 was also formed [5]. Metakaolin was used as partial cement replacement together with silica fume and rubber wastes as partial sand replacement and results indicated that high performance concrete can be obtained

with higher resistance to sulphuric acid comparing to reference mix [6]. Artificial kaolinite can be obtained by utilizing paper sludge produced by paper industry and by burning paper sludge at 700 to 800 °C metakaolin and calcium hydroxide can be produced which exhibits pozzolanic reactivity similar to commercially available metakaolin [7]. Metakaolin also can be obtained from coal mining wastes which can contain up to 30 % kaolinite and after generating metakaolin at high temperatures highly pozzolanic product can be obtained [8]. Radonjanin et al has replaced 10 wt.% of ordinary Portland cement with metakaolin and concluded that early strength of concrete was the same as reference but due to reactivity at the age of 28 days compressive strength increased by 13 % and 9 % at 90 days [9].

Ouyang et al. has reported that optimal amount of cement replacement with metakaolin was 15% and this could lead to 20% improvement of compressive strength; however superplasticizers must be used to ensure workability of mortar with cement replaced by metakaolin [10].

Metakaolin greatly affects the pore structure of concrete reducing the permeability and diffusion rates of harmful ions [11]. Therefore durability of concrete could be increased. In cement pastes blended with 10 and 20% metakaolin reduced Cl^- concentration in pore solution was detected [12]. The incorporation of metakaolin in concrete mixture composition reduces the expansion of concrete maintained in 5% sodium sulfate solution and also expansions caused by alkali-aggregate reactions [13] [14]. The freeze-thaw resistance of cement mortar can be increased more than two times for mortar containing waste paper sludge metakaolin from 10 to 20 wt.% of cement compared to reference sample [15]. Metakaolin also finds out to be an alternative pozzolanic additive in ultra high performance concrete effectively replacing silica fume [16].

Present research focuses on the incorporation of metakaolin containing waste by-product as partial cement replacement in self compacting concrete (SCC).

Materials and Methods

High strength self compacting concrete (SCC) mixture composition was designed from CEM I 42.5 N produced by Cemex Ltd (Latvia) with Blaine fineness of 3787 cm^2/g . Natural gravel with fraction 4/11.2mm was used as coarse filler and natural sand with fraction 0/4mm was used as fine filler. Quartz sand fraction 0/0.3mm was used as microfiller to improve workability of SCC and avoid segregation of fresh mixture. In the current study metakaolin containing waste by-product (MKW) with fraction <0.355mm was studied as pozzolanic material in SCC as partial cement replacement. MKW was received from foam glass granule production plant "JSC Stikloporas" Ltd. (Lithuania) where kaolin clay is used as a substance for anti-agglutination in the final stage of expanded glass granule production. During production MKW was calcined at 850 °C for about 40-50 minutes. The X-ray diffraction (XRD) analysis of MKW is given in Fig.1. A halo of amorphous metakaolin was detected in 2θ region from 15 to 30° and quartz (Q) was detected in XRD as impurity present in MKW. Also unreacted kaolin (K) was detected in the structure of MKW and small illite (I) and microcline (M) peaks were detected.

Four SCC mixture compositions were created with different amount of MKW and mixture compositions is given in Table 1. Reference (Ref) mixture composition with cement content 500 kg/m^3 were created. Then cement was replaced from 5 to 15 wt.% with MKW (5%MKW, 10%MKW and 15%MKW). The water to cement and pozzolan (MKW) ratio (W/(C+P)) was constant for all mixture compositions and it was 0.38. The workability of SCC was controlled with superplasticizer and to remain workability of SCC with MKW (>600 mm flow) additional amount of superplasticizer was added.

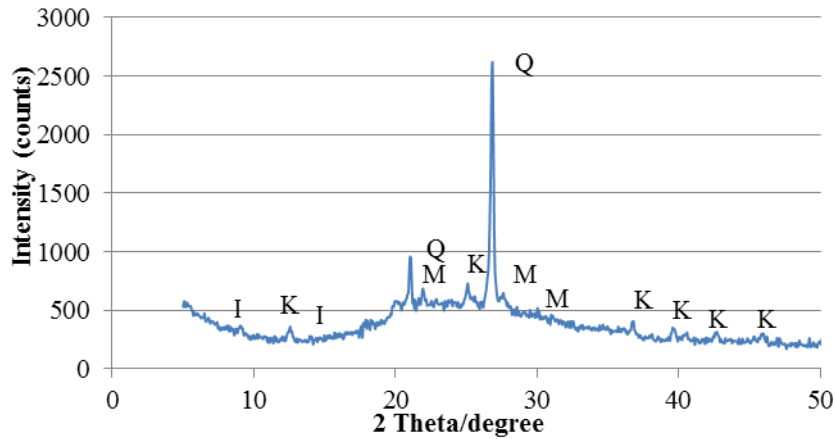


Figure 1. XRD pattern of metakaolin containing waste by-product (MKW). M – microcline, I – illite, K – kaolin, Q – quartz.

Table 1. Mixture composition of self compacting concrete.

Compound	Amount [kg/m ³]			
	Ref	5%MKW	10%MKW	15%MKW
Cemex Latvia Cement CEM I 42.5 N	500	475	450	425
Sand 0/4mm	700	700	700	700
Quartz sand 0/0.3mm	118	118	118	118
Gravel 4/11.2mm	908	908	908	908
Water	190	190	190	190
Superplasticizer Sikament 56	4.0	4.0	4.6	4.8
Metakaolin containing waste (MKW)	0	25	50	75
W/C	0.38	0.40	0.42	0.45
W/(C+P)	0.38	0.38	0.38	0.38

The mixing procedure of SCC was the following: all dry components were mixed together in planetary drum mixer for 120 s to obtain homogenous mixture of dry components. Then half of calculated water was added and mixed for another 120 s. Then rest of water with superplasticizer was added and mixing continued 120 s. Then density of fresh concrete was measured according to LVS EN 12350-6 and workability of SCC was tested according to LVS EN 12350-2. Samples were casted in the 100x100x100 mm and 40x40x160 mm moulds. Compressive strength was determined according to LVS EN 12390-3 and water absorption was carried out by immersing the prismatic SCC specimens in water for 72h. The specimens were weighed and then dried in an oven at 80°C to a constant weight for following measurement of water absorption.

The durability of the chloride penetration for SCC was performed according to NT BUILD 492 and freeze-thaw test was performed according to LVS 156-1:2009. The alkali-silica reactivity was measured according to RILEM TC 106-AAR-2 and prismatic samples 40x40x160mm were used. In mortar mixture composition defined amount of cement was replaced by MKW from 5 to 15 wt.% and workability was ensured with superplasticizer.

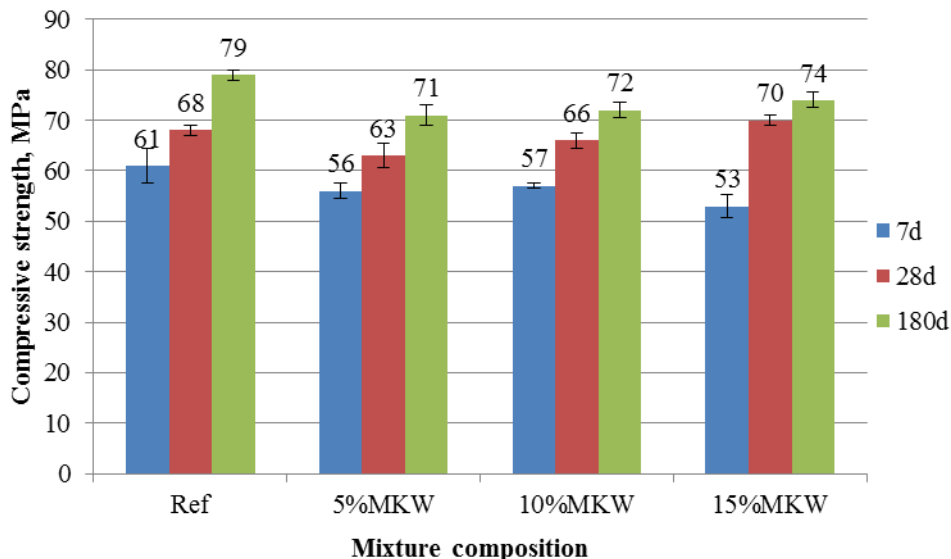
Results and Discussion

The results of fresh SCC containing different amount of MKW is given in Table 2. The fresh SCC density slightly decreased from 2428 kg/m³ for Ref to 2394 kg/m³ for 15%MKW. The partial cement substitution with MKW reduced workability therefore additional amount of superplasticizer should be used to remain the workability properties of SCC (see Table 1). Up to 20% more superplasticizer must be used to ensure workability >600 mm. Also the cone flow diameter was increased by using superplasticizer the flow time of fresh SCC increased from 25 to 34 seconds. Therefore the reduction of workability should be taken into account if MKW is incorporated in SCC as partial cement replacement.

Table 2. Fresh self compacting concrete properties.

Mixture design	Fresh concrete density, [kg/m ³]	Flow time, [s]	Cone flow diameter, [mm]
Ref	2428	25	630
5%MKW	2405	25	600
10%MKW	2403	33	680
15%MKW	2394	34	670

The compressive strength of SCC is given in Fig.2. Results indicate that SCC strength at 7 days was 61 MPa for Ref and reduced to 53 MPa for 15%MKW. The early age strength reduction is associated with slow rate of pozzolanic reactions therefore reduced amount of initial content of cement lead to reduced compressive strength. At the age of 28 days compressive strength for Ref was 68 MPa while for mixture composition 15% MKW it increased to 70 MPa and 5%MKW and 10%MKW 63 and 66 MPa respectively. Strength increase for SCC with MKW could be contributed to the pozzolanic reactions. The increased amount of MKW provided higher compressive strength for SCC at the age of 28 days. The long term curing (180 days) lead to further strength increase. For Ref it was 79 MPa while for SCC with MKW it was from 71 to 74 MPa. The further strength increase was limited for SCC with MKW. This could be contributed to the reduced amount of cement in mixture composition and the pozzolanic reaction could be limited due to rapid strength increase at the age of 28 days.

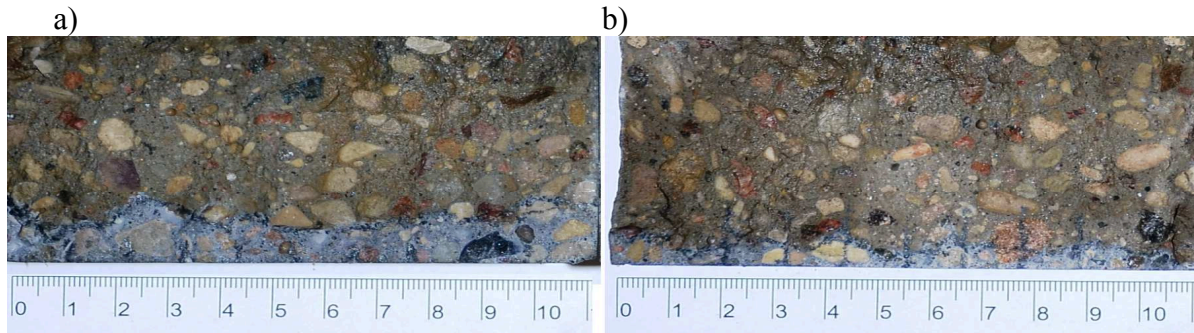
**Figure 2.** Compressive strength of self compacting concrete with metakaolin containing waste as partial cement replacement.

The durability of SCC was affected by the MKW content in the mixture composition. The chloride penetration test results are given in Table 3. Non-steady-state migration coefficient (D_{nssm}) indicates that incorporation of MKW in the mixture composition reduced D_{nssm} by 3.7 times compared to Ref. The assessment of chloride penetration test results for SCC indicate that mixture composition 15%MKW could be evaluated as very good resistance to chloride ingress while SCC with $D_{nssm} < 8 \cdot 10^{-12} \text{m}^2/\text{s}$ has a good resistance against chloride ingress [17]. The specimens Ref and 15%MKW after chloride penetration test is given in Fig. 3 showing the chloride depth of penetration. The water absorption was from 3.3 to 3.5 wt.% and open porosity from 7.6 to 8.2 % and both slightly decreased by replacing cement with MKW which could reduce the permeability of SCC and increase the durability.

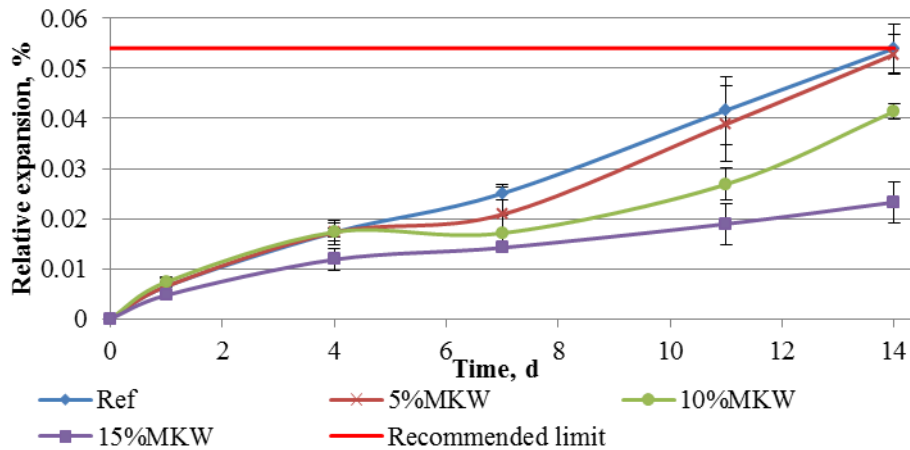
The freeze-thaw test indicates that SCC can withstand more than 500 freeze-thaw cycles in water environment without mechanical or physical damage to the specimens.

Table 3. Chloride penetration test results of SCC (non-steady-state migration coefficient).

Mixture design	D_{nssm} [$10^{-12}m^2/s$]	Standard deviation
Ref	7.70	0.37
5%MKW	5.41	0.11
10%MKW	3.63	0.12
15%MKW	2.08	0.03

**Figure 3.** The depth of chloride migration in reference (a) and SCC containing 15% metakaolin containing waste (b).

The alkali-silica reactivity (ASR) caused by reaction between cement, aggregates and sodium hydroxide solution was reduced by partial cement replacement with MKW (from 5 to 15 wt.%) and results is given in Fig.4. Significant reduction was observed by MKW incorporation from 10 to 15 wt.% of cement (23 and 56 % respectively). The recommended expansion limit defined by RILEM was 0.054% and in this case the Ref and 5%MKW were just within the recommended range while with increased amount of MKW the expansion caused by ASR were well below the recommended limit.

**Figure 4.** The effect of metakaolin containing waste by-product on the expansion caused by alkali-silica reactivity.

Summary

By replacing cement with metakaolin containing waste (MKW) by-product from 5 to 15 wt.% the amount of superplasticizer must be adjusted to ensure the proper mix flow of self compacting concrete (SCC) due to the reduction of workability caused by MKW incorporation in mixture composition. The compressive strength of concrete at the age of 28 days was from 93 to 103 % comparing to reference SCC (63 to 70 MPa) and also strength results was similar comparing reference SCC to samples with MKW the durability against chloride penetration was increased more than 3.7 times which was detected by calculating non-steady-state migration coefficient and deleterious alkali-silica reactions were reduced 2 times. Therefore it is beneficial to replace Portland cement with metakaolin containing waste (MKW) by-product in SCC.

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