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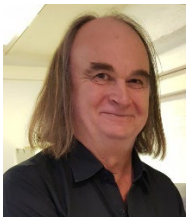
Durability of Mortar and Concrete with Composite Cements Containing Novel SCMs



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ABSTRACT

The simplest way to reduce global CO_2 emissions linked to cement productions is to reduce the amount of clinker by partially replacing it with supplementary cementitious materials (SCMs). The availability of the most common used SCMs (e.g. fly ash), is, however, expected to decrease in the coming years. Extensive research is conducted to find new alternatives and ensuring long-term durability of concrete incorporating new materials is crucial for implementation. The impact of novel SCMs from Nordic countries on fresh and hardened properties as well as durability performance in mortar and concrete was studied. The composite cements analysed had a composition of 78 wt.% Portland cement, 18 wt.% SCM and 4 wt.% limestone. The investigated SCMs include natural materials such as clays and volcanic rocks (rhyolite and hyaloclastite), and industrial products such as fly ash, slag, oil shale ash, bio-fuel ash or bio-coal fly ash. All SCMs

except oil shale ash had comparable performance to fly ash in terms of resistance to carbonation and chloride ingress, resistance to freeze-thaw scaling and mitigation of ASR. Concretes prepared with oil shale ash had considerably higher chloride ingress and no mitigating effect on ASR expansion.

Key words: SCM, concrete, chloride ingress, carbonation, ASR, frost.

1. INTRODUCTION

The world's annual cement production is roughly 4 billion tonnes [1] meaning about 24 billion tonnes concrete or a volume of 10^{10} m^3 concrete. Portland cement clinker production causes 5-8% of all man-made CO_2-emmision. By producing composite cements in which part of the clinker is replaced with supplementary cementitious materials (SCMs), the CO_2 emission and energy consumption associated with the end-product can be decreased [2]. SCMs have been used in blended cements for quite a long time [3,4], in particular ground granulated blast-furnace slag (GGBFS) [5] and coal fly ash (FA) [6]. Silica fume is another common SCM in Norway but is more often added to the concrete mix as a quality enhancer [7]. Since most of the GGBFS already is in use and FA is becoming obsolete as coal fired energy plants producing it are progressively shutting down to cut CO_2 emissions, there is a need for alternative SCMs.

It is well known that SCMs can improve the long-term compressive strength and durability of concrete by pore refinement [3]. Besides lower permeability of concretes with blended cements, the chloride binding in hydration products can be increased [8–10]. Both lower permeability and improved chloride binding improve the resistance against chloride ingress. Many SCMs have also been shown to mitigate alkali silica reactions [11–13]. Similar to chlorides, increased binding of alkali metal ions was observed in blended cements and thus improved resistance to alkali silica reactions [14–16]. In general, a faster carbonation rate is often observed in blended cements with pozzolanic SCMs due to a reduced amount of portlandite [17]. In general, resistance to carbonation is often observed to decrease with increased levels of cement substitution [18]. Resistance to salt frost scaling also remains a challenge for concrete with high amount of SCMs and is often reduced, potentially also as consequence of increased carbonation [19].

As a follow up to the article by Zuschlag et al. on hydration reactions [20], this paper documents the durability performance of alternative Nordic SCMs in binders with 78% PC, 18% SCM and 4% limestone (LL) in comparison to Norwegian Standard Fly ash cement. Zuschlag et al. showed that the different novel SCMs had only limited influence on hydration reactions at low replacement levels of cement. In this paper, the durability performance of the SCMs has been investigated regarding carbonation in mortars, chloride ingress, freeze-thaw resistance and mitigating effect on expansion due to alkali-silica reactions (ASR) in concrete. For all mixes, fresh and hardened properties of mortar and concrete were tested.

2. EXPERIMENTAL

2.1 Materials

The raw materials used are listed in Table 1. All materials were fully characterised, and information on mineralogical and chemical composition, particle size distribution and specific surface area (BET and Blaine) are published earlier [20]. Materials for the durability study were chosen based on an initial screening of reactivity, but also final potential future availability of the materials. All materials would be available in sufficient amounts, i.e., 50.000 to 200.000 t/year in a minimum 10-year perspective. Furthermore, costs and CO_2 emissions related to transport from the materials origin to the Norwegian cement plant are considered acceptable.

Table 1 – Overview of the materials used, the abbreviations and type of materials.

Material	Acronym	Type
Portland cement	PC	CEM I 52.5 R according to EN 206
limestone	LL	limestone
fly ash	FA	ash (by-product)
bio-fuel ash	Bio	ash (by-product) from incineration of biomass with some addition of coal fly ash addition
bio-coal fly ash	BCFA	ash (by-product), see Bio
oil shale ash	T	ash (by-product) from incineration of oil shale
rhyolite	Rhy	extrusive rock equivalent to granite
hyaloclastite	HYA	extrusive rock resulting from subglacial volcanic eruption
calcined illite	Q	calcined clay based on illite rich raw material
slag	SI	metallurgical slag by product

Portland cement, fly ash and limestone were received directly from the cement factory. The new types of SCMs were received in batches of 20 to 100 kg. The materials were homogenized in a rotating drum before representative samples were collected. The SCMs were received in varying fineness from very coarse stones to fine powders. Where necessary, materials were crushed with a jaw or cone crusher prior to milling in a ball mill. Before milling, the SCMs were dried at 105ºC. A grinding aid (NOR02) was used to prevent clogging of the materials in the mill. During milling, samples were taken for analysis of the surface area according to the Blaine method to achieve the desired fineness. After milling, the SCMs were homogenized in a rotating drum. The illite clay was calcined at 900ºC with a residence time of 20 min, in a pilot scale direct natural gas fired rotary kiln of 7 m length and 0.3 m diameter at IBU-Tec in Germany. Before calcination the clay was dried at 105ºC and homogenised.

Model cements were produced by blending Portland cement, limestone, and the other pre-ground SCMs in a rotating drum. The composite cements had a composition of 78% PC/ 18% SCM/ 4% LL. Since FA is the standard SCM used in current Norwegian cement, it was used as reference to compare the performance of new types of SCMs. As the SCMs were identified and received at different times during the project, samples had to be cast in two different series with two different

batches of cement (PC1 and PC2). Differences in composition and particle size were however negligible.

Table 2 – Chemical composition (%), Blaine surface area, median particle size (d_{50}) and of the two cement batches used during the project for producing model cements

	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	Na_2O	MgO	SO_3	TiO_2	LOI *	Blaine (m^2/kg)	d_{50} (µm)
PC1	19.6	4.7	63.1	3.2	1.1	0.4	2.3	3.7	0.3	1.3	453	12.4
PC2	19.7	4.9	62.2	3.2	1.1	0.5	2.4	3.9	0.3	1.3	436	11.5

Table 3 – Mineralogical composition (%) of the two cement batches determined by powder X-ray diffraction and Rietveld analysis

	C_3S	β-C_2S	C_3A	C_4AF	$CaCO_3$	MgO	CaO	$Ca(OH)_2$	Ca-Sulfates [1]	Alkali Sulfates [2]
PC1	60.4	14.4	7.7	8.8	0.4	1.2	0.4	0.6	4.1	1.7
PC2	59.2	13.6	6.6	10.4	-	1.5	0.1	1.5	4.5	2.5

[1] Sum of anhydrite, gypsum and bassanite
[2] Sum of Aphthitalite and arcanite

2.2 Mortar and concrete tests

Resistance to carbonation was tested according to EN 13295 [21], while mortar prisms were cast according to EN 196-1 [22]. The water/binder ratio (w/b) was increased from 0.50 (standard procedure) to 0.55 by adding more water. A superplasticizer (SP) was added to the mortars with the goal to achieve similar flow as the reference mortar. Except for biofuel ash, SP had to be used in all mortars. Mortar recipes are provided as supplementary data file [23]. The amount of SP used is shown in the Results section in Table 4. Prisms were demoulded after 24 h, cured wrapped in plastic for 13 days and another 21 days unwrapped at 20°C and 60% RH. At the age of 35 days, two prisms of each mix were placed in a CO_2 cabinet at 1% CO_2, 20°C and 60% RH. The third prism was left for natural carbonation in the laboratory. The carbonation depth was measured before placing the prisms in the CO_2 cabinet (35 days - Start) and after 4 and 8 weeks of exposure to 1% CO_2. Readings were taken on freshly split surfaces earliest one hour after application of the pH-indicator (1% thymolphthalein solution). The carbonation depths were measured visually with an accuracy of 0.5 mm, in 5 points for each of the four sides, omitting the corners of the uncarbonated zone as well as aggregate grains and pores. Another set of mortar prisms was cast for compressive strength testing after 28 days according to EN 196-1 [22].

For testing the resistance to chloride ingress and frost salt scaling, samples were cast from the same concrete mix (batch size 35 l). Concretes were made with 410-420 kg cement/m^3 concrete, a water to cement ratio of 0.45 and fine (0/8 mm) and coarse (8/16 mm) granite aggregate from Årdal, Norway. An air entraining agent (AEA) was added to obtain an air content of 5 ± 1 vol%. SP was used to obtain sufficient flow for casting the concrete. All concrete recipes are provided in [23]. The freshly mixed concrete was tested according to EN 12350-2 for slump [24], 12350-6 density [25] and 12350-7 air content [26]. A total of nine 100 mm cubes were cast for density, compressive strength and resistivity measurements. Three cubes were used for testing density and

compressive strength each, according to EN 12390-7 [27] and -3 [28], respectively, after 28- and 90-days curing. Electrical resistivity was measured on water saturated samples. Three concrete cubes per recipe were stored together in a small longitudinal plastic box of approx. 12 x 12 x 35 cm. The boxes were filled with water, so that samples were submerged but using minimum amount of water to avoid excessive leaching. Measurements were done with steel discs placed on parallel surfaces and connected to a LCR meter.

Resistance to chloride ingress was tested on concrete cores according to NS-EN 12390-11 [29]. This involved 90 days curing and 90 days exposure (full immersion) to 6% NaCl. Chloride ingress was evaluated by powder grinding of 9 layers from the surface to the bulk (0-1, 1-3, 3-6, 6-9, 9-12, 12-15, 15-20, 20-25, 25-30 mm). The chloride content for each layer was determined by dissolving 5 g powder dried at 105°C in 1:10 HNO_3 at 80°C. The resulting filtrate was analysed by potentiometric titration using $AgNO_3$ with a Methrom automatic titrator with silver electrode. The apparent surface chloride concentration (C_s) and chloride diffusion coefficient (D_{app}) were calculated by fitting the chloride profiles to the mathematical solution of Fick's 2nd law of diffusion in an infinite half space, and the regression coefficient (R^2) was in general > 0.996. The first point (1 mm depth) in the profile was discarded for calculation of D_{app}. The results are given together with the total intrusion of chlorides per surface unit and the depth (mm) for chloride content of 0.07% by weight of dry concrete, d(0.07), also calculated from the chloride profiles. The initial background chloride content was close to 0.01 wt% in all cases. The test was performed on three parallel concrete cylinders of 100 mm diameter and 200 mm length.

Resistance to frost salt scaling was performed according to CEN/TS 12390-9 [30], including 56 freeze thaw cycles from +20 to -20 °C with 3% NaCl solution on top of a cut concrete surface. The scaled mass from the test surface is collected and weighed after 7, 14, 28, 42 and 56 cycles. The tests are performed on four parallel samples from four 150 mm cubes.

For a selection of concretes, four additional 150 mm cubes were cast for porosity and air void analyses. Air void analyses were done on half cubes according to EN-480-11 [31]. Additionally, capillary and macro porosity was determined with the PF method [32]. After water saturation (28 days immersion), five 25 mm thick slices were cut from the cubes, dried for 7 days at 105°C and noting the weight (m_{dry}). Followed by this, the slices were again immersed in water for 7 days with subsequent weighing above (m_{air}) and below water (m_{water}). In a last step, the slices were water saturated at 50 atm water pressure for 2 days and weighed again ($m_{pressure}$). The following parameters have been calculated from the recorded weights (m_x). The PF-factor (pore protective factor) gives the volume percent of pores that are not filled with water by capillary suction.

$$\text{Capillary porosity} = 100 \cdot (m_{air} - m_{dry})/(m_{air} - m_{water}) \text{ [vol\%]}$$

$$\text{Macro porosity (air)} = 100 \cdot (m_{pressure} - m_{air})/(m_{air} - m_{water}) \text{ [vol\%]}$$

$$\text{PF-factor} = (\text{Macro porosity})/(\text{Macro porosity} + \text{capillary porosity})$$

To test the mitigation of alkali-silica reaction by the novel SCMs a new batch (batch size 25 l) of concrete was mixed. Concrete mixes with 445-450 kg cement per m^3 concrete and water/cement ration of 0.48 were made with Årdal innocuous sand (0/4 mm, 405-420 kg/m^3) mixed with Søberg reactive sand (0/10 mm, 335-350 kg/m^3) and Ottersbo reactive gravel (4-16 mm, 945-980 kg/m^3). To check the concrete quality, the freshly mixed concrete was tested according to EN 12350 [24-26] for slump, density and air content. Three 100 mm cubes for determination of 28-day compressive strength were cast for each mix and tested according to EN 12390-3 [28].

Three 100x100x450 mm^3 prisms with cast-in measuring studs were prepared for testing according to Norwegian Concrete Prism Test (NCPT) [33]. This method is comparable with the recently published RILEM AAR-10 concrete performance test [34] (except small deviations in the concrete composition). The prisms were stored in containers over water with 100% RH at 38 ± 2°C for 2 years. Weight and length measurements were performed after 1, 4, 8, 16, 26, 52, 78 and 104 weeks of exposure. The length change compared to the reference length was calculated. All length measurements were performed at 20°C, and the prisms were kept in the containers at 20°C for 16 ± 4 hours prior to each measurement.

3. RESULTS AND DISCUSSION

3.1 Carbonation

Table 4 shows the mechanical and fresh properties of mortar prisms. All results should be compared to the FA reference mixes which is similar to the standard cement type used in Norway today. A superplasticizer (SP) was added to the mortars with the goal to achieve similar flow as the reference mortar. Except for biofuel ash, SP had to be used in all mortars. Calcined clay (Q) had as expected the highest water demand and hence highest amount of SP had to be used to maintain good flow. Although, the two cement batches (PC1 and PC2) were very similar (Table 2 and Table 3) and the same fly ash batch was used, there was a considerable difference in strength between the two reference FA binders. The 28-day strength of the second reference was only about 90% of the first reference. The difference in density and weight of the mortar samples was small and cannot explain the difference in strength development. There are however minor differences in the mineralogical composition of the cements that might play a role. PC2 has a somewhat lower content of the fast-reacting phases C_3S (59.2 vs. 60.4%) and C_3A (6.6 vs. 7.7%), as well as a higher content of the slowly reacting phase C_4AF (10.3 vs. 8.8%). The slightly higher amount of $Ca(OH_2)$ (CH) is a sign of minor pre-hydration. Furthermore, PC2 has a lower Blaine value (436 vs. 453 m^2/kg) then PC1. Even though the individual differences are not large, they all pull in the same direction of lower 28-day strength. Only mortars with oil shale ash (T) and hyaloclastite volcanic pozzolan (HYA) achieved higher 28-day strength compared to FA mortars, while all other SCMs gave a lower strength (max 10 % reduction for SI).

Figure 1 shows example pictures of a carbonated mortar samples sprayed with pH indicator. It is often observed that the carbonation depth varies between top, bottom and the sides of the prisms. Measured on the top surface the carbonation depth is often higher while it may be lower when

measured at the bottom. In this study, only the average values measured on left and right side of two mortar prisms per model cement (i.e. 20 points) are considered in agreement with [35].

Table 4 – Fresh and hardened properties (28 days) of mortar prisms for carbonation experiments.

Binder	Compressive strength (MPa)	Flexural strength (MPa)	Weight (g)	Density (g/cm^3) *	Flow (mm)	SP (% of PC)
FA-PC1	49.0 ± 1.2	7.9 ± 0.3	574 ± 3	2.24	202	-
Bio-PC1	47.8 ± 1.6	7.7 ± 0.4	574 ± 5	2.20	200	-
T-PC1	50.7 ± 1.5	7.3 ± 0.2	566 ± 4	2.21	196	1.4
Q-PC1	47.9 ± 1.4	7.4 ± 0.2	566 ± 1	2.24	193	1.8
Rhy-PC1	45.1 ± 0.7	7.3 ± 0.0	562 ± 1	2.21	191	0.45
SI-PC1	44.4 ± 0.9	7.6 ± 0.4	572 ± 2	2.23	198	0.9
FA-PC2	44.6 ± 0.9	7.5 ± 0.1	570 ± 0	2.23	212	-
HYA-PC2	51.0 ± 0.7	7.8 ± 0.2	569 ± 1	2.22	203	0.9
BCFA-PC2	46.2 ± 0.5	7.6 ± 0.1	567 ± 1	2.22	210	0.9

* Variations within ± 0.01 in all samples

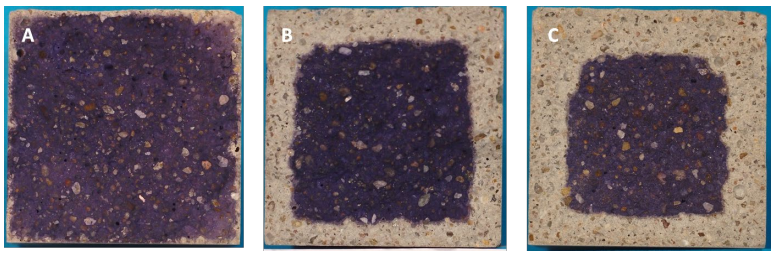

Figure 1 – Example of carbonation depth A: Start = after 35 days (21 days exposure to air), B: 4 weeks exposure to 1% CO_2 and C: 8 weeks exposure 1% CO_2

Figure 2 shows the carbonation depth in mortars after 4- and 8-weeks exposure to 1% CO_2, where mortars with HYA and BCFA had the lowest carbonation depth, i.e. best resistance towards carbonation. However, in general there were only minor differences considering the error bars (standard deviation of 5x4 points measured on two sides of two mortar prisms). There were also only small differences between the two fly ash references. The initial (start) carbonation depth was higher in mortars of the series cast with cement batch 2 (PC2). A possible reason is that a different plastic foil was used for wrapping the samples during preconditioning.

The influence of SCMs on carbonation is three-fold [36]. The physical dilution effect with less cement clinker per volume unit will result in less CH produced. The chemical and pozzolanic activity of the added SCMs will on the hand also reduce the amount of CH and on the other hand result in pore refinement. Reduction of CH per volume unit will cause a faster carbonation rate, but C-S-H gel will also carbonate [37]. SCMs containing reactive alumina (e.g. fly ash) combined with limestone filler as in this study will form more AFm-phases (mostly calcium hemi-and mono-carboaluminate hydrate) and at the same time stabilize ettringite [38]. This results in more water binding, reduced porosity and increased strength. However, these calcium aluminates with a lot of crystal water will also carbonate and then turn this crystal water back into liquid water and

thereby porosity. The permeability of a fly ash blended cement after carbonation increases therefore much more than a CEM I [39]. This can also explain why carbonation proceeds faster in cements blended with alumina containing reactive pozzolana, particularly in combination with limestone filler. In the presented results, there is no correlation between the alumina content of the studied SCMs [20] and the carbonation depth after 8 weeks.

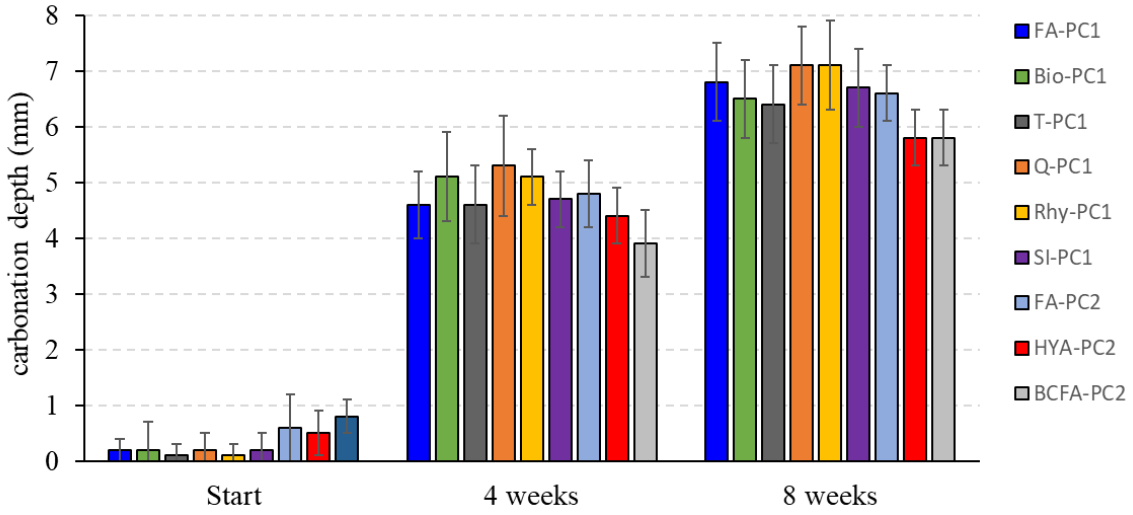

Figure 2 – Carbonation depth in mortars with a binder composition of 78% PC, 18% SCM and 4% LL after 4- and 8-weeks exposure to 1% CO_2 atmosphere

3.2 Chloride ingress

Table 5 shows the fresh properties of concrete batches cast for chloride ingress and frost resistance testing. The concrete with biofuel ash showed the lowest slump value, despite adding higher amount of SP compared to FA. This effect on flow was not observed in mortar mixes. Relatively high addition of SP was used in mixes with T, Q and Rhy, in all cases successfully increasing flow to similar or higher values than one of the FA mixes. The use of air entraining agent (AEA) was successfully applied in all mixes to reach the target of around 5% air. The highest amount of AEA had to be used with biofuel ash. For volcanic pozzolan (HYA), RHY and Q, 50% less AEA compared FA was necessary to reach the 5% goal.

Table 6 shows the results of compressive strength, density, and electrical resistivity after 28- and 90- days hydration. Note that there is a large difference between the FA-PC1 and FA-PC2 as observed for mortar mixes in section 3.1. Relative to its respective references (FA-PC2), HYA reaches the highest relative mortar strength increase, resulting in 20% more than FA after 28 days. For all SCMs there is a considerable strength increase between 28 and 90 days. Interestingly, for T, Q and HYA the relative compressive strength to FA strength decreases considerably from 28 to 91 days but is still equal or higher than the FA strength after 91 days. This indicates, that these new SCMs are reacting much faster than FA during the first 28 days and that the pozzolanic contribution of FA comes later.

Most of the concretes follow the expected trend of higher electrical resistivity with higher compressive strength. In most cases the electrical resistivity also almost doubles from 28 to 90 days. The exception is the mix with oil shale ash (T), that shows low electrical resistivity despite good compressive strength. Also, it has only a small increase from 28 to 90 days with T. Of all SCMs, oil shale ash had the lowest content of reactive silica that could contribute to a pozzolanic reaction, and a relative high alkali content [20]. As shown in the next section, T had also the poorest resistance to chloride ingress. The low resistivity might hence be explained by high alkalinity in the pore solution, and open porosity.

Table 5 – Fresh properties of concrete cast for testing against chloride ingress and frost-salt scaling, and the amount of related admixture used. SP=superplasticizer, AEA=air entraining agent.

		SP (kg/m^3)	Slump (mm)	Fresh Density (kg/m^3)	AEA (kg/m^3)	Air (%)
PC1	FA	0.67	190	2310	1.02	4.9
	Bio	0.89	160	2310	1.76	5.0
	T	1.26	210	2320	0.72	5.3
	Q	1.59	220	2310	0.59	5.0
	Rhy	1.36	195	2300	0.51	5.3
	SI	1.01	215	2320	0.66	4.9
PC2	FA	0.64	180	2300	1.09	4.8
	HYA	0.61	180	2330	0.46	4.2
	BCFA	0.93	180	2290	1.05	5.0
	LL	0.84	190	2300	0.48	5.2
	PC	1.13	195	2300	0.59	5.9

Table 6 – Properties of hardened concrete cast for testing against chloride ingress and frost-salt scaling.

		28 d			91 d		
		Cube strength (MPa)	Density (kg/m^3)	Resistivity (Ωm)	Cube strength (MPa)	Density (kg/m^3)	Resistivity (Ωm)
PC1	FA	52.3 ± 0.7	2370	87.3	63.0 ± 0.7	2350	198.1
	Bio	51.5 ± 0.2	2360	81.0	62.2 ± 0.9	2370	156.3
	T	56.4 ± 2.1	2360	59.8	63.0 ± 0.8	2370	82.7
	Q	57.6 ± 1.3	2340	100.0	64.2 ± 0.5	2350	164.9
	Rhy	47.8 ± 0.7	2330	76.8	56.8 ± 0.6	2330	149.5
	SI	53.8 ± 0.6	2380	86.8	65.7 ± 0.5	2390	159.0
PC2	FA	44.8 ± 1.0	2370	89.7	57.0 ± 1.0	2360	239.7
	HYA	53.5 ± 1.3	2380	133.7	63.5 ± 1.1	2380	265.9
	BCFA	44.6 ± 0.1	2350	80.0	56.0 ± 0.4	2340	195.3
	LL	42.7 ± 0.3	2310	57.8	47.8 ± 1.0	2330	77.8
	PC	54.6 ± 0.4	2330	58.0	59.6 ± 0.6	2330	73.5

Figure 3 shows the chloride ingress curves from accelerated chloride ingress experiments. For better visibility, the figure is divided for concretes cast with PC1 (left) and PC2 (right). Where no error bar is visible in the figures, the standard deviation between three parallel samples was less than the size of the symbols. The calculated apparent non-steady state chloride diffusion

coefficients (D_{app}) and total amount of intruded chloride, together with the interpolated surface concentrations are given in Table 7. The two reference cements, FA-PC1 and FA-PC2, have very different chloride profiles for some reason giving apparent diffusion coefficients of 4.6 ± 0.6 and 1.9 ± 0.6 10^{-12} m^2/s, while the difference in intruded chlorides were 70±3 and 78±6 g Cl^-/m^2 within the standard deviation of the two parallel samples. Table 7 also lists the depth (d) at which the chloride concentration reached 0.07% (d(0.07)), which is often regarded as the concentration to initiate reinforcement corrosion in the field in Norway [40].

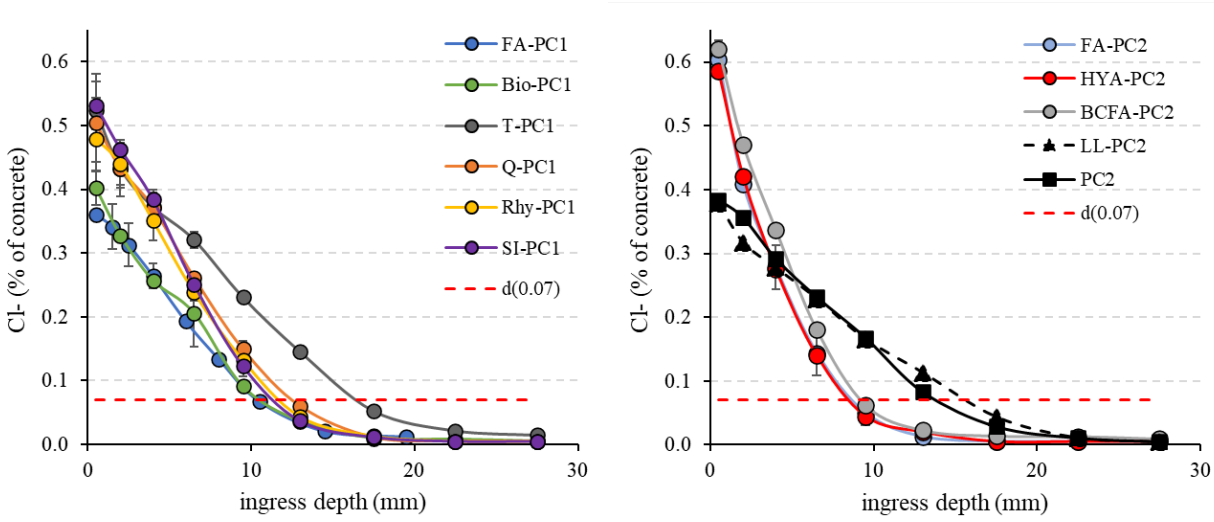

Figure 3 – Chloride ingress profiles for concretes with 78% PC, 18% SCM and 4% LL as binder mix. Left: Concretes cast with cement batch PC1. Right: Concretes cast with cement batch PC2

Table 7 – Resistance against chloride ingress of concretes with 78% PC, 18% SCM and 4% LL as binder mix and a reference with 78% PC and 22% LL (Cs = Surface concentration, D_{app} = apparent diffusion coefficient, d(0.07%) = depth at chloride concentration < 0.07%)

		C_s (% conc)	D_{app} (10^{-12} m^2/s)	Intrusion (g/m^2)	d(0.07) (mm)
PC1	FA	0.42 ± 0.05	4.6 ± 0.6	70 ± 3	10.4 ± 0.2
	Bio	0.42 ± 0.01	3.6 ± 0.4	77 ± 3	10.5 ± 0.7
	T	0.53 ± 0.03	7.8 ± 0.3	127 ± 5	16.3 ± 0.1
	Q	0.60 ± 0.09	4.1 ± 0.8	100 ± 0	12.2 ± 1.0
	Rhy	0.56 ± 0.05	3.8 ± 0.2	96 ± 6	11.8 ± 0.4
	SI	0.60 ± 0.00	3.6 ± 0.1	101 ± 1	11.3 ± 0.2
PC2	FA	0.58 ± 0.04	1.9 ± 0.6	78 ± 6	8.6 ± 2.1
	HYA	0.60 ± 0.18	1.9 ± 0.4	77 ± 12	8.4 ± 0.2
	BCFA	0.65 ± 0.06	2.2 ± 0.2	92 ± 11	9.2 ± 1.3
	LL	0.38 ± 0.07	9.3 ± 2.3	94 ± 3	15.7 ± 0.9
	PC	0.43 ± 0.04	6.8 ± 0.5	92 ± 4.7	13.9 ± 0.3

Considering the chloride profiles in Figure 3, all SCMs, except for T, considerably improve the chloride ingress resistance for the concrete compared to the PC and LL references. The concretes containing Bio and HYA had about the same performance as their respective FA reference concrete with similar chloride profiles. The concrete with BCFA had only a slightly higher

chloride ingress compared to its FA reference. The surface chloride concentration as well as concentration measured in the outer 10 mm was higher in concretes containing Rhy, Q, SI. The depth at chloride concentration of 0.07% and the amount of intruded chlorides was also increased. FA and Bio showed a similar amount of intruded chlorides. Calculated apparent chloride diffusion coefficients were similar for FA, Bio, Q, SI and Rhy. The only SCM that showed significantly worse chloride ingress resistance was T. This is reflected in the diffusion coefficient as well the intrusion value. Also, the depth at which the chloride concentration is below 0.07 wt% of concrete was considerably higher than the reference with FA.

3.3 Frost-salt scaling

Frost resistance of concrete with SCMs has been studied intensively the last decades, and it remains challenging when more than 30-35% of PC is replaced by SCMs, particularly when tested by accelerated methods involving de-icing salts. Aside from the critical dosage of the SCMs and concrete proportioning, pre-conditioning of the specimens and proper air entrainment also play very important roles. A comprehensive overview of current European, Russian and North American standard requirements and recommendation for frost resistant concrete has been summarised in [41]. The investigated concretes in this study were all designed with the goal to achieve frost resistant concrete. Special care was taken to obtain required values for air content and porosity parameters, i.e., specific pore surface area and pore spacing factor.

Figure 4 shows the frost scaling of concrete with 22% replacement of PC in the binder by SCMs after 56 freeze-thaw cycles. According to the Norwegian standard [33], scaling has to be equal or less than 0.50 kg/m^3 concrete to pass the test. Hence, all concretes with the new SCMs are within the acceptance limit for frost scaling. Furthermore, the concretes containing new SCMs all perform better than their respective reference with fly ash. All curves showed a similar behaviour. Scaling started already after 7 cycles and in most cases increased steeply up to 14 days before levelling off. The fly ash reference cast with PC2, had a lower compressive strength compared to the reference with PC1. The frost resistance, however, appeared to be better for FA-PC2. Amongst the new SCMs, the best performance was shown by Rhy and BCFA followed SI and T. There was little difference in the frost resistance of concretes containing Bio, Q and HYA. As opposed to general expectations, concretes with lowest compressive strength had the best frost resistance, e.g. Rhy, BCFA.

Frost resistance is related to the air content and distribution of air voids as well as to porosity in the concrete. Air void and porosity analyses were performed on a selection of concretes with the most interesting SCMs. Results are shown in Table 8. As specified in [42], to obtain frost resistant concrete a minimum air content of 4-5%, minimum specific pore surface area of 25 mm^{-1} and maximum pore spacing factor of 0.25 mm is required. All concretes met the requirements for air content. The concrete with Q and SI appear to require a lower dosage of AEA to get similar or higher air content. The reason might be the absence of carbon typically present in fly ashes. The air content was for all mixes within the required range. On the other hand, the air void analyses did not meet the requirements, which does not correlate to the good results from performance

testing. For all concretes the specific surface of air voids was below the recommended minimum of 25 mm^{-1}. Furthermore, except BCFA, the spacing factor was above 0.25 mm in all concretes showing insignificant differences. Still, in terms of scaling, SI performed better than FA, HYA and Q. On the other hand, the highest PF factor was measured with the concrete containing SI and BCFA. An increasing PF factor (>0.20 for fresh water and >0.25 for saltwater) is correlated to better frost resistance [43] which is in agreement with the measured material scaling.

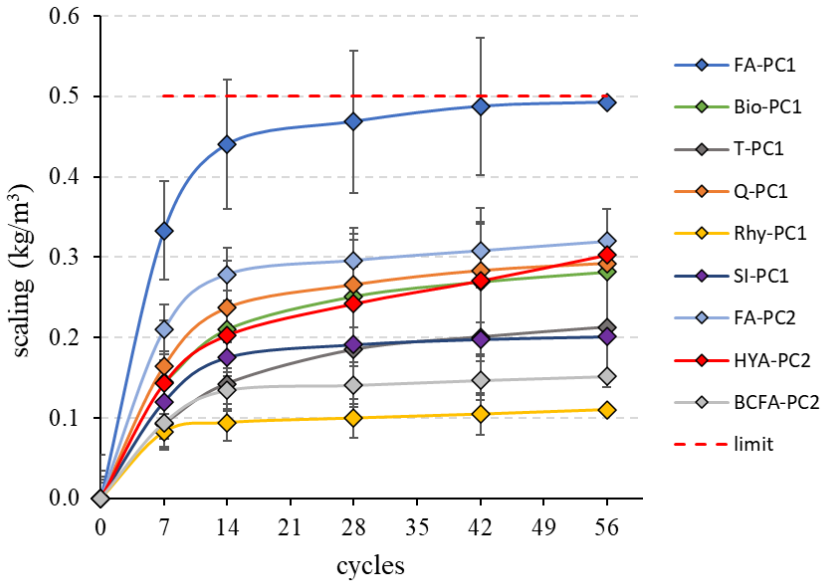

Figure 4 – Frost scaling between 7 and 56 freeze-thaw cycles from -20 to +20ºC of concrete with binders containing 78% PC, 18% SCM and 4% LL

Table 8 – Air void analyses and porosity of selected concretes from Figure 4 (same concretes as in the chloride ingress tests)

		78PC/18SCM/4LL				
		FA-PC2	HYA	BCFA	Q	SI
	Air entraining agent (kg/m^3)	1.1	0.5	1.1	0.5	0.7
Air void analyses	Air content (Vol.%)	5.0	4.5	5.1	5.9	5.7
	Specific surface area (mm^{-1})	20	20	21	18	16
	Spacing factor (mm)	0.26	0.28	0.25	0.27	0.30
	Micro Air A300 (Vol.%)	1.69	1.19	1.79	1.81	1.28
Porosity	Capillary porosity (Vol.%)	14.5	14.1	14.2	14.7	14.2
	Macro Porosity (Vol.%)	4.0	3.6	4.1	3.9	4.3
	PF-factor	0.21	0.20	0.22	0.21	0.23

3.4 Alkali silica reaction

Table 9 shows the fresh properties and 28-day compressive strength of concretes cast for experiments on alkali-silica reactions. In all mixtures, the alkali content in the binder originating from the clinker is 3.9 kg $Na_2O_{eq.}/m^3$. The total alkali content including the alkalis originating from the SCMs varies from 4.7 (SI) to 7.5 (Q and Bio) kg Na_2O_{eq}/m^3 depending on the SCM. In contradiction to all previous concrete and mortar mixes there is no difference in strength between the two FA references. The other trends of higher or lower strength between the different SCMs are similar as earlier.

Table 9 – Alkali content, fresh properties and 28-day compressive strength of concrete mixes for ASR testing.

		$Na_2O_{eq.}$ in binder (kg/ m^3)	Slump (mm)	Fresh Density (kg/m^3)	Air (%)	28-day Compressive strength (MPa)
PC1	FA	5.9	155	2390	1.2	50.5
	Bio	7.5	140	2410	1.2	52.1
	T	6.2	120	2410	1.8	57.5
	Q	7.5	95	2410	1.7	53.3
	Rhy	7.0	95	2400	1.6	52.8
	SI	4.7	140	2410	1.6	50.0
PC2	FA	5.9	190	2400	1.2	50.5
	HYA	5.6	200	2400	1.7	56.5
	BCFA	6.5	195	2390	1.5	49.4

Figure 5 shows the expansion due to ASR up to 2 years of exposure at 38ºC and 100% RH for concrete prisms containing binders with 22% replacement of PC. The red dotted lines show the limits for acceptable expansion according to the Norwegian ASR regulation, i.e., maximum 0.030% expansion after 1 year and maximum 0.060% expansion after 2 years.

The two reference concretes; one with 100% PC (alkali content 1.2 wt% Na_2O_{eq}) and one with 78% PC and 22% LL (alkali content 1.1 wt% Na_2O_{eq}) showed high expansion. The two fly ash concretes with PC batch 1 (PC1) and 2 (PC2) obtained almost identical expansions, documenting the good repeatability of the Norwegian performance test method as previously reported [44]. Moreover, it enables the comparison of various SCMs even though two different batches of PC were used in the model cements. Compared to the two references, all new SCMs except T appear to mitigate ASR in combination with the highly reactive aggregates used. However, the new SCMs appear not to have the same mitigation potential as fly ash (FA). BCFA, Rhy and HYA show slightly more expansion than FA, but are below the acceptable expansion limits in the Norwegian ASR regulations. The total alkali content in these mixtures when including the alkalis originating from the SCMs was 5.6 (HYA), 6.5 (BCFA) and 7.0 (Rhy) kg $Na_2O_{eq.}/m^3$. Bio, Q and SI did not meet the Norwegian requirements as they showed expansion above the acceptance limits after 1- and 2-year of exposure. The three SCMs reveal similar expansion despite having very different total alkali content when including the alkalis originating from the SCMs, varying

from 4.7 (SI) to 7.5 (Bio and Q) kg Na_2O_e / m^3. The availability of alkalis and thus the potential alkali contribution from the SCMs is dependent on the mineralogical composition. The majority of alkalis present in the SCMs is strongly bound in minerals like feldspars. ASR mitigating ability is roughly proportional to the ratio of reactive SiO_2/(alkalis*CaO). SCMs with high amount of reactive SiO_2 will contribute to formation of C-S-H with lower Ca/Si, increasing the capacity of the paste to bind alkalis [45].

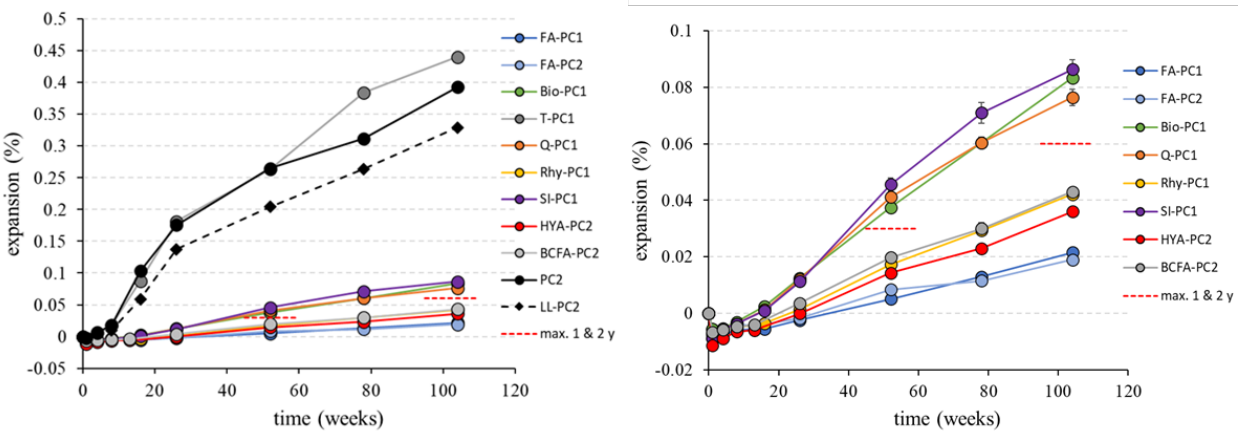

Figure 5 – Expansion due to ASR up to 2 years of exposure at 38 ºC for concretes containing binders with 78% PC, 18% SCM and 4% LL, including two references; one with 100% PC and one with 78% PC and 22% LL. The Norwegian acceptance limits are included as red dotted lines. Right: Close up to expansion limits

The slightly lower expansion of concrete with the inert limestone reference can be attributed to the diluting effect, i.e., reducing the amount of available alkalis by reducing the cement content. The additional reduced expansion by the SCMs is a result of alkali binding in cement hydrate phases, i.e. C-S-H, produced from the pozzolanic reaction [14].

4. SUMMARY AND CONCLUSIONS

Several model cements have been prepared consisting of 18% SCM, 4% LL and 78% PC or, in addition to references with 100% PC and 78% PC, 22% LL. Siliceous fly ash has been used as a reference SCM and seven other SCMs available in Northern Europe have been tested both in mortars and concrete for the most common durability issues in Norway. The performances of the new SCMs were characterized in terms of strength evolution (EN 196-1; EN 12390-3) and the resistance against carbonation (EN 13295), chloride ingress (EN 12390-11), freeze-thaw scaling (CEN/TS 12390-9) and the potential for mitigating alkali-silica reactions (Norwegian concrete prism test – NCPT). The novel SCMs were two types of fly ash (bio coal and bio fuel fly ash), oil shale ash, rhyolite, hyaloclastite, slag and an illitic calcined clay.

Table 10 – Summary of durability performance of the new SCMs compared to FA; = similar performance (within standard deviation), < and > slightly lower or higher, << and >> lower or higher, <<< and >>> very much lower or higher; (+) and (-) for passing or not passing the national requirements

	Carbonation depth	Chloride ingress D_{app}	Frost scaling 56 cycles	ASR 2y expansion
Bio	=	<	< / (+)	>> / (-)
T	=	>>	<< / (+)	>>> / (-)
Q	=	<=	< / (+)	>> / (-)
Rhy	=	<	<<< / (+)	> / (+)
SI	=	<	<< / (+)	>> / (-)
HYA	<	<=	< / (+)	> / (+)
BCFA	<	=	<<< / (+)	> / (+)

All SCMs had a similar performance with regard to carbonation. Considering chloride ingress, all SCMs except oil shale ash had better or similar performance than FA. All SCMs were below acceptable limits for frost-salt scaling after 56 freeze-thaw cycles and showed better performance than the FA reference. On the other hand, all SCMs, showed higher ASR expansion compared to FA, with the calcined clay, bio ash and slag slightly exceeding the expansion limit values after 1 and 2 years. Oil shale ash had significantly worse performance than the other SCMs, even showing higher expansion than 100% Portland cement.

5. OUTLOOK

There are several parallel activities in Norway and Sweden led by Heidelberg Materials Northern Europe to further test the durability of concrete with the most promising SCMs for future implementation, i.e. volcanic pozzolan, natural calcined clay and slag. In 2022, field samples for exposure to chloride ingress (Sotra, Norway), natural carbonation (Brevik, Norway), ASR (Trondheim, Norway and Lisboa, Portugal) as well as frost salt scaling (Borås, Sweden) were produced of full scale produced binders investigated in this paper. Within the ZeroCarbCon project (supported by NRC, No.: 336953) extensive testing of carbonation and chloride ingress on concrete with binders with up to 50% replacement of cement are conducted. The activity on carbonation is supported by a PhD work. Extensive performance tests with regard to alkali silica reactions are underway in Norway and Sweden. Furthermore, initiatives are started to establish an own research project dealing with frost-salt scaling of binders with high replacement of cement.

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