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# Effect of Set Accelerator on Properties of Wet Sprayed Concrete



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## ABSTRACT

This paper describes sprayed concrete experiments varying the set accelerator dose. Literature on the hydration of cement with modern alkali-free set accelerators is reviewed and two full scale wet spraying experiments have been conducted, varying the dose of set accelerator in each. The effects on the properties of the hardening and hardened sprayed concrete were investigated by field and laboratory testing. Increasing the set accelerator dose was found to increase the rate of early age strength development but reduce density, long term strength and increase suction porosity of hardened sprayed concrete.

Key words: Sprayed concrete, shotcrete, accelerator, porosity.

### 1 INTRODUCTION

#### 1.1. Background

Set accelerators are added to sprayed concrete at the nozzle immediately before spraying and increase the early age strength development of sprayed concrete. Studies [1–4] state that alkalifree sprayed concrete set accelerators increase the rate of the early-age strength development due to formation of hydrous calcium aluminium sulphates (mainly ettringite), which causes rapid increase in stiffness of the matrix (setting). The addition of set accelerator at the nozzle is essential for sprayed concrete used for tunnel linings as the rapid early age strength development allows concrete sprayed onto sidewalls and overhead to remain in place, rather than falling or flowing from the substrate. The set accelerator reduces the dormant period after the initial reaction during hydration [1].

Several authors [1,5,6,7,8] have reported that modern alkali-free set accelerators are mainly aluminium sulphate water solutions or slurries. Myrdal [5] reviewed well-known commercial alkali-free sprayed concrete accelerators on the market in Europe and North America, based on their Material Safety Data Sheets (MSDS), and demonstrated that modern, commercial, alkali-free set accelerators contain aluminium salts and almost all (probably all) contain aluminium sulphate. Although Myrdal [5] stated that the actual chemical composition of the commercial alkali-free accelerators are closely guarded trade secrets and therefore the precise chemical compositions are not published. Wang [8] reported that alkali-free accelerators can also contain fluoride salts, such as hydrogen or sodium fluoride, which increase the solubility of aluminium sulphate, as well as accelerate the setting of Portland cement. Furthermore Wang [8] reported that almost all alkali-free liquid accelerators contain alkanolamines, especially diethanolamine and triethanolamine, which both accelerate the hydration of Portland cement and increase the solubility of aluminium sulphate.

Many of the latest studies [1-4] on the effect of alkali-free accelerators on the hardening and hardened cementitious matrixes used accelerators that were composed of aluminium hydrosulphate solutions (aluminium sulphate solutions with different additions of aluminium hydroxide) with an addition of inorganic silicate or organic acid as the stabilisers. These admixtures had a pH of around 3.0 and an  $Al_2O_3/SO_4^{2-}$  molar ratio of 0.33–0.74.

Higher alkali-free shotcrete accelerator doses are in the range of 6–8 % of the mass of cement or equivalent binder (further simply stated as dose %), or even exceeding 10 % [5,7]. The actual dose is varied depending on the concrete and binder composition, ambient environment [7] and geometry of the substrate. The dose can be varied to compensate for the changes in any of these conditions. However, the actual effect of the accelerator dose on the hardening and hardened properties of concrete has not been thoroughly explored and only a few papers on this subject have been published over the last few years. Furthermore, almost no publications are available that have also attempted to consider the actual full-scale spraying mechanisms in combination with the set accelerator dose.

Previous studies [1,2,3,4,6,8,9] have shown that the use of the alkali-free accelerators increases the rate of the early-age strength development due to formation of hydrous calcium aluminium sulphates (mainly ettringite), which causes rapid increase in stiffness of the matrix (setting). Each mole of ettringite contains 32 moles of water [10]. Ettringite formation and chemical binding of water thus increase the solid/liquid ratio and the viscosity of the cement paste, leading to setting of the cement. Wang [8] reported that the needle-like ettringite crystals connect to form a reticular

structure, which also causes setting. Tan [11] mixed C<sub>3</sub>S pastes with aluminium sulphate and found that CSH and ettringite coexist at ages 2 hours to 28 days.

It has also been demonstrated [1,2,3,4,6] that the compatibility between the cement type and the alkali-free accelerator plays a major role in the hydration kinetics and mechanical strength evolution. This is mainly with respect to the final C<sub>3</sub>A/SO<sub>3</sub> molar ratio that is calculated considering the sulphate remaining after the accelerator reaction and the initial C<sub>3</sub>A content of a cement. The initial strength and setting determined by needle penetration resistance testing (up to 3-4 hours of sprayed concrete age) will mainly depend on the accelerator reactivity. A high  $Al^{3+}$ content incorporated into cement matrices will result in exothermic ettringite formation, providing fast setting and elevated early strength gain. The strength gain after 3-4 hours (typically measured by the stud driving or pin penetration testing) will not depend solely on the porosity of the obtained matrix, but also on the final C<sub>3</sub>A/SO<sub>3</sub> molar ratio. This is because when the system is undersulphated (high final C<sub>3</sub>A/SO<sub>3</sub> molar ratio), accelerated C<sub>3</sub>A reactions occur before or during the acceleration period of the silicate reactions, filling the pores of the matrix and reducing solubility of the main cement phases. Consequently the accelerator decreases the extent and rate of alite hydration and leads to lower degrees of hydration and strength gain at lower ages. This explains a lower strength development after the initial 4 hours when high (about 8-10 %) alkali-free accelerator doses are used.

A low dose (typically at around 2 %) is known from practice to cause retardation of the sprayed concrete [7]. This has been explained experimentally by [9], who showed that a low dose, a small extra amount of  $Al^{3+}$  and  $SO^{2-}_{4}$  are supplied to the pore solution, leading to a further thin layer of ettringite forming on the surface of the cement particles. This thin layer can slow the hydration of cement pastes, causing a retarded setting compared to samples without accelerator.

The above mechanisms also suggest that the availability of the sulphate ions in the solution of the fresh cementitious matrix is important. This has been verified by [1,9], who demonstrated that these mechanisms of alkali-free accelerator effect in a cementitious matrix depend on the type of set regulator in a cement. It has been found [1,9] that calcium sulphate hemihydrate saturates the mixing water faster with calcium and sulphate ions (as it has a higher solubility than gypsum) and thus leads to a faster sulphate ion reactivity and faster precipitation of the hydrous aluminium sulphates.

The addition of set accelerators changes the microstructure of the concrete [8]. When large ettringite amounts are formed, the matrix stiffens quickly due to increase in solid/liquid ratio leading to improper consolidating and air entrapment [4]. Salvador [4] reported that the "water accessible porosity", which we interpret as the suction porosity, increases in accelerated matrices and the compressive strength reduces. Han [12] reported that a 6 % dose of aluminium sulphate decreased the volume of 5–30 nm pores but increased the volume of 30–100 nm pores at 24 hour age, indicating a coarser capillary pore structure, with a similar conclusion at 28 days.

It has been demonstrated by [4] that from a certain time the accelerated mix compressive strength is always lower than that of a reference mix without alkali-free set accelerator. The faster the setting time (more reactive accelerator or higher dose), the lower the compressive strength from 1 day on.

Salvador [3] investigated the influence of the spraying process on the hydration of cement pastes with alkali-free accelerators and demonstrated that the mixing procedure significantly influences the microstructure of the matrix. They determined that the aluminate hydrates are more evenly distributed throughout the whole matrix in sprayed pastes. Furthermore, ettringite is found in a disarranged microstructure and is composed by shorter, irregular, and dispersed needle-like crystals. Salvador [3] accordingly recommended preparing pastes by spraying in contrast to simple hand-mixing in order to obtain a more representative microstructure of the resulting hardened cementitious matrix.

The suction porosity of hardened wet sprayed concrete in tunnel linings was investigated by Holter [13] by systematic measurements on 234 slices sawn from drilled cores taken from four different tunnels after 150–1100 days. Suction porosity measurements on the concretes with w/b = 0.45, 0.44, 0.46 and 0.47 were compared to theoretical values calculated with Power's model [14]. Holter [13] stated that mix designs, including w/b ratio, were corrected for water content of the set accelerator. The comparison to Power's model indicated degrees of hydration of 65 %.

Myren & Bjøntegaard [15] measured the suction porosity of sprayed concrete samples with w/b ratio of 0.42 and set accelerator dose of 6 % of binder mass. The sprayed panels were cured in air then cores were taken, which were cured in water and tested at 400 days age. Again, Power's model [14] was used to calculate theoretical values of suction porosity, and an assumed 80 % degree of hydration gave similar suction porosity values to those measured by capillary suction and PF (pore fraction) tests.

## 1.2 Scope

This paper attempts to connect the previous detailed laboratory studies, which have been mainly done on paste and mortar samples with the actual sprayed concrete process by describing two full scale wet spraying experiments and presenting results from these experiments. The set accelerator dose added at the nozzle was varied in both spraying experiments. The properties of the hardening and hardened sprayed concrete from full scale wet spraying experiments that are investigated are early age strength development, density, compressive strength, porosity and capillary absorption.

## 2 EXPERIMENTAL WORK

## 2.1 Spraying experiments

Two full scale wet spraying experiments were carried out at separate locations with different equipment, teams and ready mixed concrete suppliers. The details of the two full scale wet spraying experiments are given in Table 1.

Location	AMV, Flekkefjord	NTNU, Trondheim
Dates	18 <sup>th</sup> February 2020	16 <sup>th</sup> June 2020
Spraying machine	AMV 7450 shotcrete robot	Normet Spraymec NorRunner 140
		DVC shotcrete robot
Concrete pump	Olin pump	Normet pump
Hose diameter	75 mm	75 mm
Nozzle diameter	40 mm	40 mm
Concrete flow rate	10 m <sup>3</sup> /hr	14 m <sup>3</sup> /hr
Accelerator type	Master Builders Masterroc SA 188	Master Builders Masterroc SA 168
Accelerator dose	Varied	Varied
	0, 3, 6 and 10% of binder mass	3.5 and 7% of binder mass
Distance nozzle to substrate	2.0 m	1.5 m
and angle	Perpendicular to the panel	Perpendicular to the panel
Curing and coring	Wrapped in plastic before coring.	Wrapped in plastic before coring.
	Cored 13 <sup>th</sup> March 2020	$1^{st} - 2^{nd}$ July 2020
	Ø 75 mm, h 150 mm for strength	Ø 75 mm, h 150 mm for strength
	and density tests. Cured in water	and density tests. Cured in water
	until compressive strength testing.	until compressive strength testing.
	Ø 100 mm for capillary suction, PF	Ø 100 mm for capillary suction, PF
	and image analysis tests. Cured in	and image analysis tests. Cured in
	air before oven drying.	water before oven drying.

Table 1 – Details of two full scale wet spraying experiments

Concrete constituents, proportioning and placed concrete composition

The concrete proportioning for the Flekkefjord spraying experiment is shown in Table 2. This mix represents a typical sprayed concrete mix currently used for sprayed concrete tunnel linings in Norway. The slump test immediately after batching was 220 mm. The placed concrete composition was corrected for the addition of set accelerator (at the nozzle) and equating compaction voids, measured by image analysis, to air content following [16].

Phase	Constituent	Mass [kg/m <sup>3</sup> ]	Volume [l/m³]	Volume [l/m³]	Placed concrete composition* [kg per m <sup>3</sup> concrete after spraying] for 10 % set accelerator dose
	Standard fly ash cement	462	156		444
	Water	206	206		221
	Silica fume	19.2	8,7		18.5
Matuin	Super plasticiser	4.8	4,6		4.6
watrix	Air entrainment agent	0.45	0,45	424	0.43
pnase	Set accelerator (dry)	-	-		25.0
	0-8  mm aggregate (< $0.125  mm$ )	21.8	8,3		21.0
	Air		40*		Volume = 55.5 litres **
Doutiala	0-8  mm aggregate (> 0.125	1505,8	570,4		1448
rarucie	mm)			576	

Table 2 – Fresh concrete composition and placed concrete composition (for sample with 10% accelerator added) from set accelerator spraying experiment in Flekkefjord

\* Assumed air content for batching.

Steel fibres

phase

SUM

\*\* Typical measured value for macro porosity of hardened specimens, see Table 6.

45

2304.8

The binder was standard fly ash cement, with density 2990 kg/m<sup>3</sup>, and microsilica with density 2200 kg/m<sup>3</sup>. The aggregate was granitic sand (crushed) with a particle density of 2645 kg/m<sup>3</sup> and a water absorption of 0.46 % by mass. The effective water/binder ratio of the mix was 0.42. But with set accelerator added to the mix at the nozzle the water/binder ratio of the placed concrete

5,6

43.3

2225.1

changed. The additional water content  $\Delta w$  was calculated for the concrete proportioned in Table 2 after addition of the varying doses of set accelerator. The set accelerator used had a water content of 0.48 and solids content of 0.52 by mass [17]. A placed concrete composition, which is calculated for the 10% set accelerator and corrected for compaction porosity, is shown in the column on the right of Table 2.

Table 3 –	Particle	size dis	stributic	on and	density	of agg	regate d	of the Fl	ekkeford	mix	
Sieve size	32	16	11.2	8	4	2	1	0.5	0.25	0.125	0.063
(mm)											
% passing	100	100	100	98	82	72	53	25	8	2	1

 $\Delta w$  = accelerator dose × accelerator water content × ( $m_c + k m_s$ ) New  $m_w/(m_c+k.m_s) = (206 + \Delta w)/500$ 

0% acceleration	tor_		6 % accelerator		
$\overline{m_{\rm w}}/(m_{\rm c}+k.m_{\rm s}) = 206/500 = 0.419$			$\Delta w = 0.06 \times 0.48 \times (500) = 14.4 \text{ kg}$ m <sub>w</sub> /(m <sub>c</sub> +k.m <sub>s</sub> ) = (206 + $\Delta w$ )/500 = 0.448		
3 % accelerat	<u>tor</u>				
$\Delta w = 0.03 \times$	$0.48 \times (500) =$	= 7.2 kg	10 % accelerator		
$m_{\rm w}/(m_{\rm c}+k.m_{\rm s})$	$=(206+\Delta w)$	(2)/500 = 0.434	$\Delta w = 0.10 \times 0.48 \times (500) = 24.0 \text{ kg}$ m <sub>w</sub> /(m <sub>c</sub> +k.m <sub>s</sub> ) = (206 + $\Delta w$ )/500 = 0.467		
where	m <sub>c</sub>	mass of cement			
	ms	mass of silica fume			
	W	mass of water			
	k	k factor for equivalent w	$p/m_{\rm c}$ ratio		

The concrete proportioning for the Trondheim set accelerator experiment is detailed in Table 4. Again this mix was intended to represent a typical mix for sprayed concrete, but in this case steel fibres were omitted from the mix. The slump test before spraying was 220 mm. The placed concrete composition for the addition of 7 % set accelerator by binder mass and corrected for compaction porosity is shown in the right column of Table 4.

Phase	Constituent	Mass [kg/m <sup>3</sup> ]	Volume [l/m³]	Volume [l/m <sup>3</sup> ]	Placed co composit concrete	oncrete ion* [kg per m <sup>3</sup> after spraying]
	Standard fly ash cement	433	145			424
	Water	214	214	ŀ		226
	Microsilica	43	19.5	5		42.0
	Retarder	0.4	0.36	Ď		0.4
	Super plasticiser	3.7	3.5	5		3.6
Matrix	Air entrainment agent	0.6	0.6	5	177	0.6
phase	Set accelerator (dry)	-			4//	21.5
-	0 - 4  mm aggregate (<0.125 mm)	22	7.2	2		21.2
	0-8  mm aggregate (<0.125 mm)	129	47.3			126
	Air		40*	:	Volui	me = 45.5 litres **
D. (* 1	0-4  mm aggregate	153	50.5	;		150
Particle	0-8  mm aggregate	1284	472	2	523	1256
pnase	Steel fibres	-				-
SUM		2282.7				2268.9

Table 4 - Fresh concrete composition and placed concrete composition (with 7 % accelerator added) from set accelerator spraying experiment in Trondheim

\* Assumed air content for batching.

\*\* Typical measured value for macro porosity of hardened specimens, see Table 6.

Table 5 – Particle size distribution of aggregate of the Tronanelm mix											
Sieve size, mm	32	16	11.2	8	4	2	1	0.5	0.25	0.125	0.063
0/8 aggregate % passing	100	100	100	98.2	84.5	70.6	55.2	39.2	22.3	9.1	2.3
0/4 aggregate % passing	100	100	100	99.8	89.2	58.9	38.4	26.2	18.0	12.4	8.1

Table 5	Particle	siza dis	tribution	of	aggragata	ofthe	, Trondhain	n miv
Table 5	– Pariicie	size ais	iriouiion	010	aggregale	<i>oj ine</i>	e ironanein	i mix

The binder was standard fly ash cement, with density 2990 kg/m<sup>3</sup>, and microsilica with density 2200 kg/m<sup>3</sup>. The sand 0/4 mm was crushed gabbro rock with a particle density of 3030 kg/m<sup>3</sup> and a water absorption of 0.5 % by mass. The 0/8 mm sand was natural fluvial glacial deposits with mainly granitic gneiss and some sandstone and mafic rock with a particle density of 2720 kg/m<sup>3</sup> and 0.7 % water absorption by mass. The effective water/binder ratio of this mix was 0.42. The additional water content  $\Delta w$  was calculated for the concrete proportioned in Table 4 after additional of the varying doses of set accelerator. The accelerator used had a water content of 0.425 and solids content of 0.575 [18].

 $\Delta w$  = accelerator dose × accelerator water content × ( $m_c + k.m_s$ ) New  $m_w/(m_c+k.m_s) = (214 + \Delta w)/519.1$ 

<u>0 % accelerator</u>	$m_{\rm w}/(m_{\rm c}+k.m_{\rm s}) = (214 + \Delta w)/519.1 = 0.433$
$m_{\rm w}/(m_{\rm c}+k.m_{\rm s}) = 214/519.1 = 0.418$	
	7 % accelerator
	$\Delta w = 0.07 \text{ x } 0.48 \text{ x } (519.1) = 15.44 \text{ kg}$
3.5 % accelerator	$m_{\rm w}/(m_{\rm c}+k.m_{\rm s}) = (214 + \Delta w)/500 = 0.448$
$\Delta w = 0.035 \times 0.48 \times (519.1) = 7.72 \text{ kg}$	

One batch of concrete was used for each of the Flekkefjord and the Trondheim experiments.

#### Set accelerator

In the Flekkefjord spraying experiments the accelerator dose by effective binder mass was varied, and the different doses were 0, 3, 6 and 10 %. Concrete mixes with all four doses were sprayed. However the concrete intended to be sprayed with 6% accelerator did not behave as expected and likely contained zero accelerator – a blockage was discovered in the accelerator line immediately after spraying. This panel showed early strength development equal to the panel sprayed with 0 % accelerator and this result is marked with \* in each case.

In the Trondheim spraying experiments the accelerator dose by effective binder mass was varied, and the different doses were 0, 3.5 and 7 %. Concrete was sprayed with 3.5 % and 7 % set accelerator, whilst the concrete with zero accelerator was cast by pumping the concrete through the nozzle, adding neither compressed air nor set accelerator.

#### Spray application

For the experiments in Flekkefjord concrete was sprayed into 600 mm diameter and 100 mm deep moulds with a plywood base and steel sides. The moulds were placed horizontally on the floor so that the unaccelerated sprayed concrete would not flow during/after spraying. The nozzle was perpendicular to the mould and 2 m away when spraying, as shown in Figure 1.

For the experiments in Trondheim concrete was sprayed in  $1 \text{ m} \times 1 \text{ m}$  wooden panels of 150 mm depth and slanted sides in accordance with EN 14488-1 [19]. The panels were placed at 60° from

the horizontal. The concrete was sprayed with the nozzle at 1.5 m distance and perpendicular to the mould, as shown in Figure 2.



*Figure 1 – Spraying set up for Flekkefjord spraying experiments.* 



Figure 2 – Spraying set up for Trondheim spraying experiments.

## 2.2 Laboratory testing

## Strength

The early age strength development of the sprayed concrete was tested in accordance with EN 14487-1 [20] and EN 14488-2 [21]. The penetration needle was used up to a compressive strength of 1 MPa and above that the stud driving method was used until 24 hours after spraying. The

penetrometer needle method measures the force required to drive a needle of 3 mm diameter into the concrete to a depth of 15 mm [21]. For the stud driving method a stud is percussively fired into the concrete and the penetration depth measured. The stud is then extracted and the pull-out force measured. The ratio of pull-out force to penetration depth is used to determine the compressive strength [21].

Three parallel cores of 75 mm diameter and height 150 mm were drilled through the full thickness of the panels and tested for compressive strength in accordance with EN 12390-3 [22] at 28 days after spraying. The panels were wrapped in plastic before coring. The cores for density and compressive strength were cured in water before testing. The Flekkefjord cores for capillary suction and PF test were cured in air before oven drying, while the Trondheim cores for these tests were cured in water before oven drying.

#### Density

Cores were weighed in air and then in water to determine the density immediately before compression strength testing. Furthermore the PF test (Section 2.2.4) also gives density results for individual discs.

#### Capillary suction

The capillary suction test was undertaken on the sprayed concrete samples following [23] and [24]. Discs of 100 mm diameter and 25 mm thickness were cut and dried at 105 °C for 48 hours before testing. The mass of each sample after drying was recorded as  $w_1$ .

The discs were then placed on a perforated metal tray with a depth of 1 mm of the disc immersed in the water. The capillary suction causes water to be drawn into the concrete and there is an increase of mass from this absorbed water. The relationship between mass increase and square route of time is normally linear [24] and the capillary number of the concrete is determined by the gradient of the mass increase against square route of time. After immersion in 1 mm of water the samples were weighed at regular intervals over the first four days. The experiment determines the capillary and resistance numbers.

$$K_{\rm cap} = \frac{G(t)}{\sqrt{t}} \tag{1}$$

where  $K_{cap}$  is the capillary number, and G(t) is absorption (kg/m<sup>2</sup>) at time t (seconds)

The resistance number *m* is given by  $t_{cap}$ , which is the time for the water front rising in the specimen to reach the top of the specimen of thickness *h*.  $t_{cap}$  is the point of inflection on the absorption vs square route of time plot.

$$m = \frac{t_{\rm cap}}{h^2} \tag{2}$$

A lid was placed over the samples to reduce evaporation. The mass after four days of capillary suction and before submersion is recorded as  $w_{1.5}$ . The sum of gel and capillary porosity, equal to the suction porosity, is calculated as:

$$\varepsilon_{\text{suction}} = \frac{w_{1.5} - w_1}{v} \tag{3}$$

where v is volume measured by weighing in air and in water.  $\varepsilon_{\text{suction}}$  can then be used for comparison with total porosity (equal to capillary plus gel porosity) in Power's model after correction for paste volume fraction in the concrete [14].

#### *PF* (pore fraction) test

Following the capillary suction test the same samples were submerged completely under water at atmospheric pressure for four days. The additional uptake from unilateral to complete submersion is considered to fill the open macro pores. The mass after atmospheric (unpressurised) submersion is recorded as  $w_2$ . The samples were then submerged under a pressure of 5 MPa for 48 hours to fill the closed macro pores. The mass after pressurised submersion is recorded as  $w_3$  [14].

The total porosity is calculated by:

$$\varepsilon_{\text{total}} = \frac{w_3 - w_1}{v} \tag{4}$$

The open macro porosity is calculated by:

$$\varepsilon_{\text{open macro}} = \frac{w_2 - w_{1.5}}{v} \tag{5}$$

And the closed macro porosity, or air voids, is calculated by:

$$\varepsilon_{\text{closed macro}} = \frac{w_3 - w_2}{v} \tag{6}$$

#### Image analysis

The macro porosity was measured by black and white image analysis [25]. A core from each sprayed panel was cut in half lengthways to make two samples and the flat surface of each sample was polished. A Tegramin 30 apparatus was used and the samples were polished using resin bonded, diamond surfaced discs of:

- 1. Vickers hardness 220, grain size 75 µm,
- 2. Vickers hardness 600, grain size 30 µm,
- 3. Vickers hardness 1200, grain size 15 µm.

The polished surface was then coloured black using marker pen, and then the macro pores were filled with white sulphate nitrate powder of particle size  $1-4 \mu m$ . An example is shown in Figure 3(a). The samples were then scanned at a resolution of 2400 pixels per inch. A Matlab script was used to calculate the ratio of white and hence the macro porosity of each specimen.



Figure 3 – Scanned black and white image of sample sprayed with: (a) 7 % set accelerator (Trondheim series); (b) 10 % set accelerator (Flekkefjord series), with major lamination was considered not representative of the bulk material and was removed by cropping.

Major imperfections were cropped out of the image analysis. For example the concrete sprayed with 10 % set accelerator in Flekkefjord contained a major lamination normal to the spraying direction, several tens of millimetres wide, due to pulsation of the flow at the nozzle where accelerator was added at a steady flow, and this is omitted from the image analysis, as shown in Figure 3(b).

## **3 RESULTS AND DISCUSSION**

## 3.1 Early age strength development

The early age strength development of the Trondheim set accelerator experiments is shown in Figure 4, which shows the higher set accelerator dose with a faster strength development. The J1, J2 and J3 curves in Figure 4 are standard strength development curves that are defined in [20] and these define the strength class of the sprayed concrete.

Furthermore Figure 4 also gives a comparison of three different measurement methods of early age strength: penetrometer, stud driving and compressive testing of drilled cores. Both the penetrometer and stud driving method measurements were taken at 2.5 hours for the concrete sprayed with 7 % set accelerator, with a difference of 0.5 MPa between the readings. For the same specimen the stud driving method was used at 9 hours, with a result of 9.3 MPa, and a core was tested in compression at 8.5 hours after spraying, with the strength measured at 12 MPa. Furthermore a core was tested in compression at 30.5 hours after spraying with the strength at 26 hours after spraying with the stud driving method. The penetrometer and stud driving method at 26 hours after spraying with the first 6 hours and then the stud driving method and the compressive strength of cores indicate same strength class after 9 hours.



Figure 4 – Early age strength development of samples spayed in set accelerator experiment in Trondheim.

#### **3.2** Density and strength

The relationship between density and compressive strength for individual specimens from both the Flekkefjord and Trondheim series is plotted in Figure 5. This graph shows that there is a clear relationship between bulk density and compressive strength – higher density causes higher compressive strength. This is as we would expect since bulk density,  $\rho_{\text{bulk}}$ , scales linearly to total porosity  $\varepsilon_{\text{tot}}$  as  $\varepsilon_{\text{tot}} = 1 - (\rho_{\text{bulk}} / \rho_{\text{solid}})$  where  $\rho_{\text{solid}}$  is the density of pore free concrete material and porosity is the key to concrete strength. Furthermore Figure 5 shows that higher doses of set accelerator give a lower density of sprayed concrete, and hence a lower compressive strength.

The measured density values for the cast Trondheim samples are in fairly good agreement with the sum of mass calculated in Table 4 (2283 kg/m<sup>3</sup>) and after correction for the addition of 7 % accelerator given that the moisture content of the cylinders for strength measurements had first dropped during air curing of the panels and then increased after coring and 3 days water storage before testing.



Figure 5 – Combined plot of compressive strength at 28 days against density results from Flekkefjord and Trondheim (individual cylindrical specimens).

### 3.3 Porosity

The porosity of the samples measured by the PF test are shown in Table 6. There is a higher suction porosity values of the samples with higher doses of set accelerator. The capillary numbers in Table 6 are calculated as the gradient capillary absorption graphs during the initial phase of absorption [24], as defined by Eq. 1, and these are calculated from the capillary suction graphs included in Figures 7 and 8. The results show increasing capillary numbers and higher total volume of water absorption for the higher accelerator doses.

The capillary number is sensitive to the paste volume fraction of the concrete, which can vary in sprayed concrete due to rebound of the particle phase [26], whereas the resistance number, defined by Eq. 2, does not depend on the paste volume.

A graph of compressive strength against total porosity for samples from all the spraying experiments is presented in Figure 6(a). This shows a notable relationship, with decreasing compressive strength with increasing total porosity. Figure 6(b) shows a graph of compressive strength against macro porosity, but no relationship is evident. Figure 6(c) shows a graph of compressive strength against suction porosity, which shows the trend of decreasing compressive strength with increasing suction porosity, although with slightly weaker correlation than for total porosity vs strength. A graph of compressive strength against capillary number is shown in Figure 6(d), and no relationship is evident.

The relationship of decreasing strength with increasing total porosity is as we would expect – Neville [27] reported that a 2 % increase in void ratio (i.e. macro porosity) can decrease the compressive strength by 10 %. It is interesting that the relationship of decreasing strength is with the suction porosity, and even stronger with total porosity, rather than the macro porosity of the sprayed concrete, and this warrants further studies of the suction and microporosity of sprayed concrete. Perhaps the nature of the macro pores and the suction pores in sprayed concrete are

different from those of conventionally cast concrete in terms of the size and type of pores that fill under suction and under pressure saturation in the PF test.

	1	Capil	lary suction a	and PF test			Image
		I	v				analysis
Sample	$K_{\text{cap}}$ Eq.(1)	<i>m</i> Eq.(2)	Total	Suction	Open	Closed	Macro
-	Capillary	Resistance	porosity	porosity	macro	macro	porosity
	number	number			porosity	porosity	
	$(kg/m^2.s^{0.5})$	$(s/m^2)$	Volume	Volume	Volume	Volume	Volume %
			%	%	%	%	
Flekkefjord	0.012	$1.30 \ge 10^8$	18.4	14.6	0.4	3.7	4.4
0%	0.012	1.21 x 10 <sup>8</sup>	18.3	14.5	0.4	3.7	5.3
Flekkefjord	0.018	7.74 x 10 <sup>7</sup>	20.5	16.4	0.7	4.1	4.7
3%	0.018	$7.06 \ge 10^7$	20.4	15.8	0.5	4.7	4.5
Flekkefjord	0.015	$10.8 \ge 10^7$	19.2	15.4	0.4	3.8	4.4
6% *	0.015	9.60 x 10 <sup>7</sup>	18.1	14.7	0.3	3.4	4.0
Flekkefiord	0.055	2.88 x 10 <sup>7</sup>	26.5	22.7	0.6	3.8	5.3
10%	0.046	3.46 x 10 <sup>7</sup>	25.8	22.1	0.7	3.7	5.8
Trondheim	0.011	2.76 x 10 <sup>7</sup>	22.3	17.9	0.3	4.3	6.3
zero	0.013		22.7	18.2	0.3	4.5	6.1
accelerator –							
cast		2.76 x 10 <sup>7</sup>					
Trondheim	0.013	2.76 x 10 <sup>7</sup>	21.8	18.1	0.5	3.7	3.1
3.5%	0.009		22.5	17.7	0.6	4.9	4.7
accelerator		4.15 x 10 <sup>7</sup>					
Trondheim	0.017	$2.76 \ge 10^7$	24.5	20.5	0.5	4.0	4.4
7%	0.016		23.8	20.1	0.6	3.8	4.7
accelerator		2.76 x 10 <sup>7</sup>					

*Table 6 – Capillary numbers, resistance numbers and porosity results from capillary suction and PF tests (individual specimens) and macro porosity from image analysis* 

\* actually zero percent accelerator.

Furthermore, with the increasing suction porosity with accelerator dose (Table 6) there is a trend of decreasing strength with increasing set accelerator dose. This is apparent in the density against strength plot Figure 5, with higher porosity (and hence lower density) from higher set accelerator dose resulting in lower strength. This is as we would expect, for instance Opsahl [28] reported strength loss up to 50 % with a 12 % dose of (alkaline) accelerator. The mechanisms could perhaps be a mixture of increased w/c, different hydration products and how the macro-pores are formed during the spraying process.

As discussed with Figure 4, a higher dose of set accelerator increases the rate of early strength development of sprayed concrete. This agrees with the early strength gain found by [1,2,3,4,6,9] discussed in Section 1. But an excessive dose of set accelerator can have a detrimental effect on long term strength. Figure 4 shows that the early age strength development of the concrete with 3.5% set accelerator dose overtakes that with 7% dose at around 10–12 hours after spraying.



Figure 6 – Compressive strength (mean value for cores from each panel) against: (a) total porosity (mean value for cores from each panel); (b) macro porosity (mean value for cores from each panel); (c) suction porosity (mean value for cores from each panel); (d) capillary number (mean value for cores from each panel).

The samples of higher accelerator doses exhibited a higher total porosity than the zero and lower accelerator doses, as shown in Table 6. The values for macro porosity are similar, albeit with a degree of scatter. The difference between the higher accelerator doses and the lower and zero doses is the effect on suction porosity. The difference in suction porosity could be for two reasons:

- The set accelerator has a water content of 0.4–0.5 so adding set accelerator at the nozzle increases the water/binder ratio of the placed concrete as discussed in Section 2.1.1.
- The effect on hydration of the set accelerator is "freezing" the hydration at a low degree and/or altering the hydration products, in line with Salvador [1–4].



Figure 7 – Capillary suction curves for samples from Flekkefjord spraying experiments.



Figure 8 – Capillary suction curves for samples from Trondheim spraying experiments.

We can use Power's equation linking suction porosity, w/b ratio and degree of hydration to calculate the expected increase of suction porosity due to the increased w/b ratio only. Eq. 7 is a modified Power's equation [14,29].

$$\varepsilon_{\text{suction}} = \frac{\frac{w}{c} - 0.172\alpha + 0.116\frac{s}{c}\alpha_{\text{s}}}{\frac{w}{c} + 0.333 + \frac{1}{2.2c}} V_{\text{p}}$$
(7)

where

$\epsilon_{suction}$	suction porosity
w/c	mass of water / mass of cement
α	degree of hydration of cement
s/c	mass of microsilica / mass of cement
$\alpha_{\rm s}$	degree of hydration of microsilica
$V_{p}$	volume fraction of paste.

Calculating  $\Delta \varepsilon_{\text{suction}}$  using the *w/b* ratios of the sprayed concrete with 7 % set accelerator by equivalent binder mass versus that with zero set accelerator, the theoretical  $\Delta \varepsilon_{\text{suction}}$  is 1 %. Whereas there is a measured difference of 2.3 % between suction porosity of the Trondheim samples with 7 % set accelerator and those with zero set accelerator. Likewise there is a measured difference of 7.8 % between suction porosity of the Flekkefjord samples with 10 % set accelerator and those where is a theoretical  $\Delta \varepsilon_{\text{suction}}$  of 1.2 % between these samples.

Therefore the increased suction porosity is not only due the increased water content from the set accelerator added at the nozzle. There must be a "freezing effect" of the set accelerator on the hydration as well. This is consistent with [5], who used the notion that the water accessible porosity of the mortars increases in accelerated matrices and compressive strength reduces. This could be due to the matrix stiffening quickly due to the increase in solid/liquid ratio when large amounts of ettringite form, leading to improper consolidating and air entrapment [5].

Eq. 7 can be rearranged to solve for degree of cement hydration, giving Eq. 8. Calculated degree of hydration values are shown in Table 7, using calculated w/c from the concrete compositions in Section 2.1.1. The degree of hydration of the microsilica,  $\alpha_s$ , is assumed to be 1.0.

$$\alpha = \frac{1}{0.172} \frac{w}{c} + \frac{0.116\alpha_{\rm s}}{0.172} \frac{s}{c} - \frac{\varepsilon_{\rm suction}}{0.172} \left( 0.333 + \frac{w}{c} + \frac{1}{2.2} \frac{s}{c} \right) \tag{8}$$

The calculated degrees of hydration shown in Table 7 for the Flekkefjord samples with zero or low accelerator doses (remember the concrete supposedly sprayed with 6 % set accelerator actually contained zero accelerator) are from 0.681 to 0.984. These seem reasonable degrees of hydration given the age of these samples was 125 days at the start of the capillary suction test. However, calculated degrees of hydration for the samples sprayed with the accelerator dose of 10 % are 0.285 to 0.326, which are low. It appears that addition of accelerator has affected the usefulness of the equation and it appears that the modified Power's equation is not applicable for concrete with set accelerator added, presumably because the hydration products no longer have the usual fractions of CSH.

Sample	Suction	w/c	s/c	Paste volume	Degree of
•	porosity			$V_{\rm p}$	hydration
	Esuc			Volume of cement + silica	α
				fume + water + water	
	Volume %			from accelerator	Calculated
	from Table 6				with
					Equation 8
Flekkefjord 0%	14.6	0.446	0.0416	0.367	0.775
Flekkefjord 0%	14.5	0.446	0.0416	0.367	0.788
Flekkefjord 3%	16.4	0.478	0.0416	0.372	0.681
Flekkefjord 3%	15.8	0.478	0.0416	0.372	0.759
Flekkefjord 6% *	15.4	0.511	0.0416	0.376	0.944
Flekkefjord 6% *	15.1	0.511	0.0416	0.376	0.984
Flekkefjord 10%	21.5	0.554	0.0416	0.382	0.285
Flekkefjord 10%	21.2	0.554	0.0416	0.382	0.326
Trondheim zero	17.9	0.494	0.099	0.377	0.532
accelerator - cast					
Trondheim zero	18.2	0.494	0.099	0.377	0.492
accelerator - cast					
Trondheim 3.5%	18.1	0.536	0.099	0.382	0.666
accelerator					
Trondheim 3.5%	17.7	0.536	0.099	0.382	0.721
accelerator					
Trondheim 7%	20.5	0.578	0.099	0.387	0.483
accelerator					
Trondheim 7%	20.1	0.578	0.099	0.387	0.541
accelerator					

Table 7 – Suction porosity and solving for degree of hydration using modified Powers equation

\* Actually zero set accelerator.

All the concrete mixes for these experiments contained an air entrainment agent, which forms spherical air bubbles in the fresh concrete. Beaupré [30] reported that, for mixes with a high air content due to inclusion of an air entrainment agent, most of the air content can be lost during spraying and/or impact with the substrate, leaving an air content of, for example, 5 %. Table 6 shows that while this effect has expelled most of the entrained air from the sprayed samples, for the cast samples the high air content of the fresh concrete was retained. This high air content accounts for the higher suction porosity values recorded in Table 6 for the unaccelerated cast samples (Trondheim series), and hence lower calculated average degree of hydration values (Table 7), compared with the unaccelerated sprayed samples (Flekkefjord series). For the Trondheim samples with 3.5 % set accelerator the results are comparable to the Flekkefjord samples with 3 % set accelerator, while the samples with 7 % set accelerator show a reduction in calculated degree of hydration, but less than the samples with 10 % set accelerator from Flekkefjord.

Table 7 shows that when calculating cement paste volume fraction and then using it to calculate degree of hydration based on the modified Power's equation (Eq. 8), the accelerator reduced the degree of hydration for the Flekkefjord series, and possibly also for the Trondheim series. Though Eq. 8 is sensitive to the volume fraction of paste.

Concerning the low calculated degree of hydration values, it is likely that a high dose of set accelerator has led to an excess of aluminium-based ions and sulphate ions and that these have altered the hydration products – firstly due to the needle-like structure of the excess ettringite formed, which is likely to give a higher suction porosity. Secondly the composition of the CSH gel is likely to be altered. Gartner [31] reported that sulphates, alumina and other impurities are

found in the CSH, and that up to 50 % of the total sulphates and alumina are found in the CSH gel, rather than in the ettringite or monosulphate phases that are generally considered to contain these species. Therefore it is likely that such a high dose of set accelerator has led to an excess of aluminium based ions and sulphate ions and that these have altered the composition of the CSH gel, giving different CSH gel porosity in the samples with lower or zero set accelerator doses. The altered hydration products renders the modified Power's equation inapplicable given that it is based primarily on the hydration products of the standard Portland cement reactions. The latter reaction has its chemical shrinkage, gel and capillary formation from mainly CSH and calcium hydroxide- and not so much from aluminate-sulphate phase reactions.

Due to the extra water added with the accelerator the capillary number is possibly affected by both accelerator and variations in w/c. Smeplass [23] indicated that for silica fume concrete with w/c increasing from 0.41 to 0.46, the capillary number could increase from 0.010 to almost 0.015. And that for concrete with w/c variation in the range 0.41–0.46, resistance number could vary in the range  $9 - 11 \times 10^7$  (s/m<sup>2</sup>). Comparing these findings from [23] with the observed effects of accelerator on capillary and resistance numbers in Table 6, the variations for Flekkefjord 3 % mixes are within this scatter. However there is a probable effect of accelerator when comparing the 3 % to the 0 % mix, and there is a clear effect on capillary and resistance numbers for 10 % accelerator. For Trondheim there is a probable/clear effect on capillary and resistance numbers from the 0 % to the 7 % mix.

To definitively determine whether the increase in suction porosity in sprayed concrete with higher set accelerator doses, further work is planned: laboratory casting of concrete with varying doses of set accelerator, but with water also added to samples to give a constant w/b ratio in the samples. Capillary suction and PF test would then be carried out to measure suction porosity. Given constant w/b, any change in the suction porosity would be solely due to the effect of the accelerator on the hydration products.

## 4 CONCLUSIONS

- A higher set accelerator dose gives a higher early age strength development in sprayed concrete but reduces density and long-term strength of the sprayed concrete.
- There is a clear trend between the density of the hardened sprayed concrete and its compressive strength samples with a higher density have a higher compressive strength.
- The methods of measuring early age strength the penetrometer, the stud driving method and compression testing of cores give relatively close results.
- A higher set accelerator dose causes a lower density and a higher suction porosity in the hardened sprayed concrete. The higher suction porosity also causes an increased rate of capillary absorption in the hardened sprayed concrete.
- The capillary porosity increases with higher doses of set accelerator. This is due to the matrix stiffening quickly when large amounts of needle-like ettringite are formed in the early stages of hydration.
- With increasing doses of set accelerator the hydration products are altered and renders the modified Power's suction porosity equation inapplicable.
- The macro pore content of sprayed concrete is different from that of conventional air entrained concrete and image analysis seems to give the largest measurement of macro pore content.

• A further experiment to cast samples with varying set accelerator doses, with water added to give a constant *w/b* ratio in the samples, is proposed. Given constant *w/b*, any change in the suction porosity would be solely due to the effect of the accelerator on the hydration products.

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