

## **D-2-2 Water repellent wet sprayed concrete for tunnel applications**

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*ABSTRACT: Sweden consists mostly of stable crystalline bedrock of metamorphic or magmatic origin. Tunneling work for infrastructure project or mining is therefore often performed by securing less stable tunnel sections by rock anchors and lining the tunnel wall with sprayed concrete only. The application of sprayed concrete for tunnel linings, in particular for infrastructure tunnels, demands therefore high standards for durability. Tunnel operators often apply hydrophobic impregnations (e.g. for road tunnels) in order to reduce the ingress of chloride into the concrete and increase frost resistance. The presented paper investigates the idea of using a bulk hydrophobic agent which can be applied in the concrete mix and thus rendering the entire sprayed concrete layer hydrophobic and by this avoiding a later treatment. Two bulk hydrophobic agents were studied. Important was their impact on the hydration of cement with and without accelerator. This was done by studying cement hydration by in-situ XRD and isothermal calorimetry. Further investigations concerned the strength development, the performance of the agents in the concrete (water absorption, contact angles) and adhesion strength of hydrophobic sprayed concrete on a substrate. The latter was performed in the lab by casting and by an actual spraying experiment. The results showed that both bulk hydrophobic agents delayed hydration and strength development, but showed surprisingly had a lesser effect on the practical spraying as well as on adhesion strength. It seems that by adjusting the dosing of the set accelerator at the spraying nozzle, some of the negative effects of the delay in hydration by the hydrophobic agents can be counteracted. An application of bulk hydrophobic agents seems therefore to be an interesting possibility to increase the durability of sprayed concrete.*

*KEY-WORDS: Sprayed concrete, water repellent agent, tunnel, wet spraying, durability*

### **INTRODUCTION AND GOALS**

Sprayed concrete has a broad field of application for underground constructions. In particular in Sweden due to stable crystalline bedrock, sprayed concrete is often used as only tunnel lining material (in conjunction with rock bolts, anchorage and injections). In Scandinavia ca. 90 % of sprayed tunnel linings consist of wet sprayed concrete. The dry spray technique is almost exclusively used in conjunction with concrete repair. Wet sprayed concrete is due to its slightly more porous structure and its exposure environment more severely subjected to environmental influences than standard concrete. In particular in road tunnels, sprayed concrete is exposed to higher CO<sub>2</sub> levels increasing the risk for carbonation, frost attack and deicing salts, and in some cases, by soft rock water which leaches calcium out of the concrete.

Damages on sprayed concrete in road or train tunnels cause considerable costs to the society. This includes not only condition assessment, damage analysis and repair measures but also delays in traffic flow during the interventions and increased costs for performing repairs under high traffic loads. Any measure or strategy for

reducing the repair cycles in road or train tunnels can help to greatly safe costs for the society. Furthermore, it maintains the function of the tunnels for accommodating the present and future traffic loads on our infrastructure. The strategy to increase the durability of sprayed concrete is to focus in making it water repellent. Water repellency will reduce the amount of moisture and moisture flow within and through the concrete [1]. Consequently sprayed concrete will be more frost resistant, with lower penetration of chlorides [2] and reduced carbonation of the concrete [3]. By this maintenance costs can considerably be reduced as it was shown on case studies [4]. Water repellency of concrete can be achieved by an impregnation treatment with a hydrophobic agent. Most hydrophobic agents to impregnate concrete surfaces are based on alkyltrialkoxo silanes, e.g. triethoxy silane with an n-butyl or n-octyl group [5]. Silanes are monomeric and show therefore a better penetration as the larger di- or polymers (e.g. siloxanes or silicon oils). However, post treatment of concrete surfaces by impregnating contributes considerably to increased construction costs. A better approach is to blend the hydrophobic agent into the concrete as bulk during the mixing process. By this bulk treatment the entire concrete becomes hydrophobic. For sprayed concrete, where only a fairly thin concrete layer is applied, e.g. to a tunnel wall, this can be economically more favorable than the impregnation treatment.

Depending on the dosage and type, bulk hydrophobic agents can have a significant effect on the hydration of Portland or Portland limestone cement [6,7]. The effect can sometimes be mitigated by depositing the agent on carrier powders usually consisting of amorphous SiO<sub>2</sub> or calcium carbonate. The solid bulk hydrophobic agents, however, have the drawback that they are not as effective as agents in form of liquid emulsions. For the application of bulk hydrophobic agents for sprayed concrete there is therefore an uncertainty concerning their performance. Furthermore it is not exactly known how bulk hydrophobic agents may influence the very early setting of accelerated sprayed concrete. The practical use of bulk hydrophobic agents in sprayed concrete is therefore not as straightforward as it should be since the water repellent effect and the immediate application of sprayed concrete cannot necessary always be guaranteed without time consuming and costly test sprayings. Another complicating factor in Sweden is the more and more frequent use of low alkali Portland composite cements with fly ash and slag (usually CEM II/A-V or CEM/A-S) instead of pure low alkali CEM I. It has been shown in earlier studies that the performance of certain silanes may vary over time depending on the type of cement used [8,9]. However, the hydration behavior of low alkali Portland composite cements with fly ash or slag in conjunction with bulk hydrophobic agents, without or with acceleration, is not well known and understood.

The overall goal of the project was to increase expertise and experience in how to apply bulk hydrophobic agents in sprayed concrete with low alkali Portland composite cements (fly ash) in order to increase the concrete's durability and serviceability. Important was to identify effects on early setting and hydration as well as longer term performance behavior. This included also effects on modern alkali free set accelerators used for the wet spray technique.

## METHODOLOGY

### Materials

The study focused on two types of cements (manufactured by Cementa): A Portland cement CEM I 42,5 N - SR 3 MH/LA and a Portland composite cement CEM II/A-V 42,5 N – LA, both manufactured with an low alkali clinker. The former is normally used for many infrastructure project. The latter is a more sustainable low alkali cement alternative by Cementa (not yet put on the market) with a fly ash content of around 15 %. Two hydrophobic agents were used: Sitren P750 by Evonic Industries (E) and Silres BS 1001 from Wacker (W). Table 1 gives an overview of their characteristics. Additionally the alkali free set accelerator Sigunit L 2712 AS by SIKA (A) was used for all the experiments.

Table 1. Used hydrophobic agents and their characteristics.

	<b>Sitren P750</b>	<b>Silres BS 1001</b>
Abbreviation	E	W
Type	Powder (silica)	Emulsion
Active content (%)	Ca. 50	Ca. 50
Components	Silanes/siloxanes	Silanes/siloxanes
Applications	Bulk, mortars, tile grouts, plasters	Impregnations of porous substrates

For the different tests different sample types were used:

1. Pastes and mortars for hydration experiments and mortars for strength and performance tests

## 2. Concrete for bond strength, spraying experiments and performance tests

Table 2 shows the matrix for the paste and mortar samples. The mortar samples were blended with standard sand according to EN 196-1 [10], with the difference that the w/c stated in Table 2 was used. The hydrophobic agents were added under blending the pastes, mortars and concrete, respectively. Table 3 shows the composition of the concretes for the pull-out tests and the spraying experiments. For the concretes natural aggregate 0/8 mm was used.

Table 2. Matrix for paste and mortar samples.

Mix	w/c	Cement	Sitren P750 (E) (mass- % <sub>cement</sub> )	Silres BS 1001 (W) (mass- % <sub>cement</sub> )	Sigunit L-2712 AF (A) (mass- % <sub>cement</sub> )
CA	0.45	CEM I	-	-	-
CA-E			3	-	-
CA-W			-	3	-
CA-A			-	-	5
CA-A-E			3	-	5
CA-A-W			-	3	5
VA		CEM II/A-V	-	-	-
VA-E			3	-	-
VA-W			-	3	-
VA-A			-	-	5
VA-A-E			3	-	5
VA-A-W			-	3	5

Table 3. Matrix for concrete samples.

Material	Type	BCA-A	BCA-A-E	BCA-A-W	SBCA-A	SBCA-A-W
		For pull-out tests			For spraying experiments	
Cement (kg/m <sup>3</sup> )	CA	470			480	
Silica fume (kg/m <sup>3</sup> )	U972	23,5			-	
Fly ash (kg/m <sup>3</sup> )	V	-			25	
Aggregate 0/8 (kg/m <sup>3</sup> )		1550			1612	
Super plasticizer (mass- % <sub>cement</sub> )	SP	2			0.7	
Accelerator (Sigunit) (mass- % <sub>cement</sub> )	A	4			Added at nozzle: ca. 5 to 7 %	
w/c		0.470			0.405	
w/b		0.450			0.345	
Hydrophobic agent (mass- % <sub>cement</sub> )	E	-	3	-	-	-
	W	-	-	3		3

### Testing procedures

Early hydration was studied by in-situ x-ray diffraction on cement paste samples. For this an air tight sample cell was used which had a Kapton foil window. The measurements were started 30 min after mixing the pastes and then in hourly periods until 24 h of hydration. Measurements were performed on a Rigaku Miniflex 600 with a 1d solid-state detector. Isothermal calorimetry analyzes the heat of hydration. Measurements were performed on cement paste samples with an instrument from TAMAir over 7 days of hydration. Initial and final setting, as well as early shrinkage, was measured but results are not shown here.

The strength development was monitored from 3 h to 28 d. The tests were performed on standard mortar bars according to EN 196-1 [10]. Tests at early hydration ages (3h to 24 h) were performed with a load cell of 10 kN on an Instron universal testing machine, tests on later ages with a load cell of 250 kN on a hydraulic press from Testing GmbH. Pull-out tests for establishing the bond strength between treated concrete and a standardized concrete substrate were performed according to EN 1542 [11] with a pull-out tester after 28 d of hardening.

From each series 2 panels were prepared and tested after 28 d with 5 pull-out tests per panel. For the tests, only the series with Portland cement CA including accelerator and treatment was used. Pull-out tests were also performed from test specimens from the spraying tests. In the spraying mould a standard substrate panel consisting

of concrete was placed and fixed. The concrete mixes SBCA-A and SBCA-W-A were then sprayed into the mould on the substrate panel. 135 d after spraying, 70 mm drill cores were extracted from the sprayed concrete with the substrate and tested in tensile mode on a universal testing machine with a 150 kN load cell.

The performance of the hydrophobic treatment was tested on laboratory mortar specimen as well as on drill cores from the spraying tests. The test methods consisted on contact angle measurements (mortar samples), water absorption test according to EN ISO 15148 [12] and on chloride migration tests according to the NT Build method (drill cores from spraying tests only) [13]. Contact angle measurements were done with the sessile drop method (drop volume 3  $\mu$ l) on samples with the formwork side of lab mortar specimen. Tests were done after > 28 days of curing.

## RESULTS

### Early age hydration – Heat of hydration and phase development

The heat of hydration gives quantitative data about the reactivity of a binder system. It indicates how admixtures change the hydration of a given binder. The reaction heat can be given as cumulative total heat over the observed time period or its 1<sup>st</sup> derivative, which is called heat flow. Heat flow curves for the first 72 h are given in Fig. 1 for both cement paste series CA and VA.

The effect of the accelerator (A) can clearly be seen. In the initial period (Fig. 1) pastes with accelerator caused a much larger heat release. The accelerator initiated a strong heat release within the very first minutes of hydration by the consumption of gypsum and formation of ettringite (see below). In the induction to decelerating period the pure pastes CA and VA without admixtures had different peak positions of heat release: Portland cement CA showed its peak at 11 h, Portland composite cement VA at 12 h. The accelerator delayed the dormant period by ca 1 h for both cements but then shifted the acceleration period 2 to 3 hours ahead.

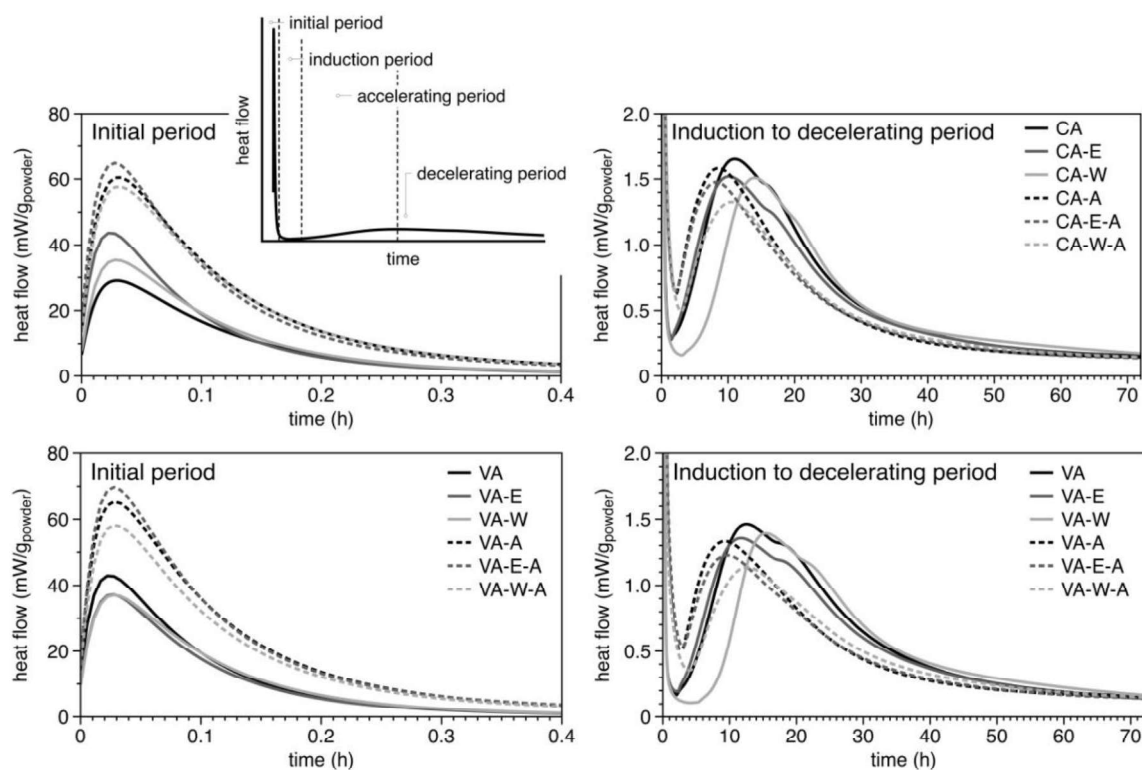


Fig.1. Heat flow curves of the cement paste series CA and VA. Note that the left graphs magnifies the first 24 minutes of the right graph.

Pastes with hydrophobic agents behave differently. In the initial period of the heat flow curves with accelerator, agent E increased the maximum heat release and in the accelerating period accelerated hydration for both cements. Agent W showed lower heat release in the initial period and from the induction to the accelerating period a delay of the maximum for ca. 3 h for both, CA and VA. That means that the silica powder of agent E reacted within the paste without delaying the maximum heat release. The agent W in emulsion form had the opposite effect: The

hydration was clearly delayed by several hours. This was also reflected by measuring initial and final setting on mortar pastes (results not shown here). On the other side, the effects of the accelerator alleviated the hydration delay of agent W at very early setting times.

The phase development measured by in-situ XRD was focussing on the consumption of gypsum and the formation of ettringite. Fig. 2 illustrates the development of ettringite and depletion of gypsum for the series CA and VA at 30 minutes and 24 h of hydration. The series CA-W with the agent from Wacker showed clearly lower amounts of ettringite at 0.5 h and 24 h, confirming the results from isothermal calorimetry of the delay in hydration with this series. For the fly ash containing series VA, both pastes with the hydrophobic agents (VA-E and VA-W) showed similar low amounts of ettringite at 0.5 h and 24 h.

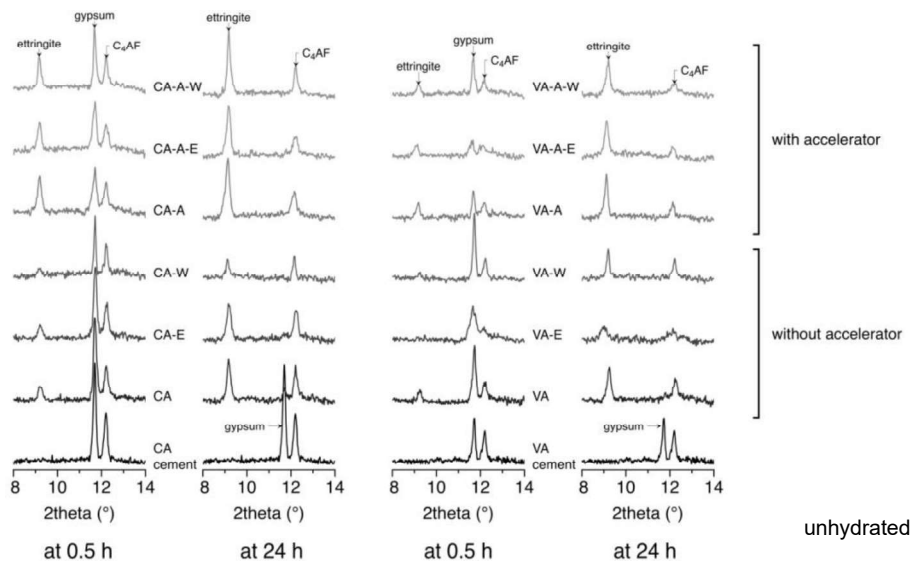


Fig.2. Results from in-situ XRD measurements at 0.5 h and 24 h of hydration. Shown is the 2theta range where gypsum and ettringite are visible.

Differently behaved pastes with accelerator. Here there was almost no qualitative difference in the amounts of ettringite formed between pastes without and with hydrophobic agents. The only difference was a lower amount of ettringite formed in the VA series. The XRD results confirm also here the data gained by isothermal calorimetry that early setting induced by the accelerator is not so much influenced by the hydrophobic agents.

### Strength development

The early strength development between 3 h and 24 h was only determined for the CA series with accelerator (mortar specimen) and the results are illustrated in Fig. 3. The influence of the hydrophobic agents is clearly visible. Both agents lead to a strength reduction of about 30 %. This is also in agreement with isothermal calorimetry, where a lower heat development was observed in the series with accelerator and hydrophobic agents (Fig. 1). The later strength development is shown in Fig. 4. Here the impact of the hydrophobic agents is clearly evident in reducing the compressive strength by 25 to 35 %. Generally the CA series gained higher compressive strength than the series VA, which is in agreement with other studies [14]. Interestingly, by using an accelerator some of the strength is recovered in case of the agent W with both binders and with binder CA also in case of agent E.

The bond strength between the concretes with the admixtures and a standard concrete substrate was determined with the cement CA. Sprayed specimens consisted of the reference mix SB-CA-A and a mix with the Wacker agent (SB-CA-A-W). The casted sample specimen showed a generally higher bond strength compared to the sprayed specimen. Important is that the hydrophobic agents showed no major visible impact on the bond strength. The specimens of BCA-A-W showed a high spread of the results due to only four values with a high variation.

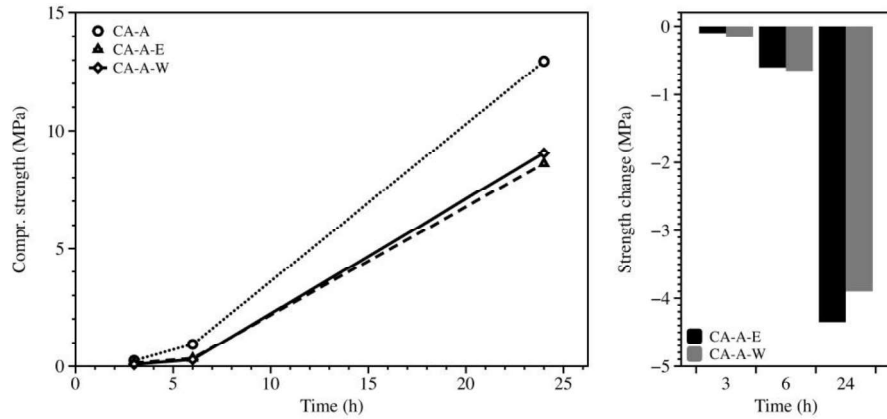


Fig.3. Early strength development measured on the series CA with accelerator (left). Change in strength compared to the series without hydrophobic agents (right).

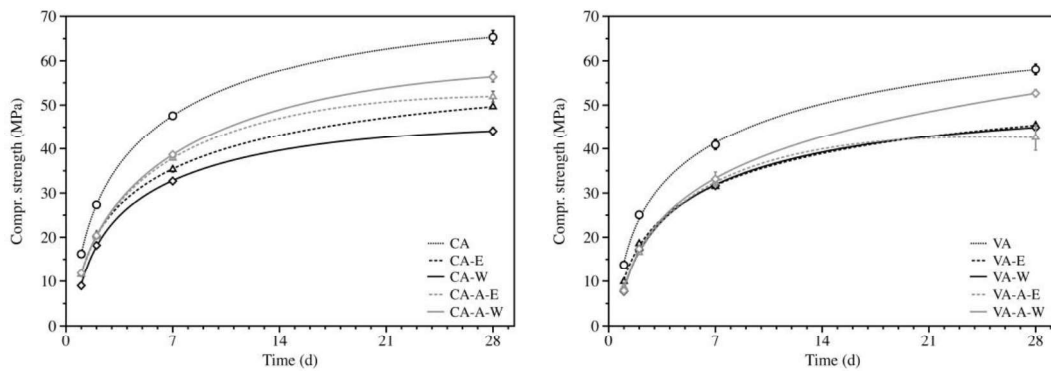


Fig.4. Strength development of the series CA and VA from 1 d to 28 d (mean values with standard dev.).

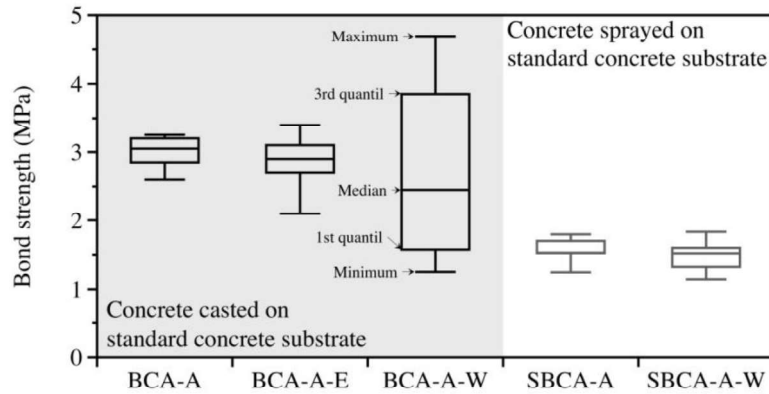


Fig.5. Bond strength results in form of a whisker boxes.

### Performance criteria

The performance of the hydrophobic treatment was established by contact angle and water absorption measurements on mortars as well as on drill cores from the spraying experiment. Additionally, chloride migration tests were performed on specimen from the spraying tests. Contact angle measurements on mortars were done only on the sample formwork surfaces with 5 measurements for each sample.

Fig. 6 shows the results. The untreated reference CA showed a high contact angle value  $> 90^\circ$ , which is probably due to the contamination of the surface with demolding oil (even though demolding oil has only been applied to the side of the formwork, not to the bottom). Paste samples with hydrophobic agents showed contact angles well above  $90^\circ$ , usually in the range of  $130$  to  $150^\circ$ . Both hydrophobic agents performed similarly well, with the agents E having slightly higher contact angles for most of the sample series. The water absorption tests yielded one magnitude lower absorption coefficients  $C$  for the treated specimen. The differences in  $C$  are low for the two agents but the agent W yielded slightly lower values. However, the results are within the error margin.

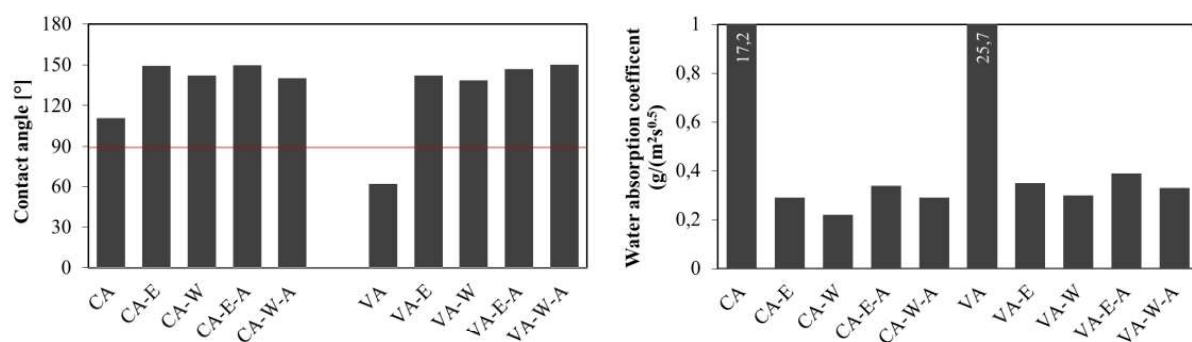


Fig.6. Results from contact angle and water absorption measurements on mortar specimen.

The spraying tests were performed on a spraying stand with a conventional spraying pump (at Vattenfall AB, Älvkarleby, Sweden). The fresh concrete was pumped to the nozzle, where the accelerator was added. The dosing of the accelerator was done by experience and was between 5 and 7 % on the cement mass. Prior to spraying into the mould of the test specimens, a certain amount of concrete was sprayed and discarded for fine adjusting the amount of accelerator. Both concrete mixes performed well under blending and spraying. The mix containing the agent W showed the same consistency and spraying behaviour as the mix without W.

After the spraying tests the two test specimen were wrapped in plastic foil for ca. 10 days and shipped to CBI. By arrival at CBI the concrete specimen were stored indoors until testing. Tested samples consisted of drill cores from the larger sprayed specimen. The results of the tests are listed in Table 4 and for bond strength additionally in Fig. 5. The results indicate that the performance of the hydrophobic agent W was less pronounced in the sprayed specimen as in the laboratory test samples. Both, absorption and chloride migration coefficient were reduced but the reductions were much lower than on the laboratory specimen.



Fig. 7. During and after the spraying test. The uncovered mould contains the sprayed concrete with the agent.

Table 4. Results from specimen from the spraying tests. Values in parenthesis are standard deviations.

Mix	Water absorption coefficient (g/(m <sup>2</sup> ·s <sup>0.5</sup> ))	Chloride migration coefficient (·10 <sup>-12</sup> m <sup>2</sup> /s)	Bond strength (MPa)
SBCA-A	0.74 (0.04)	9.8 (0.8)	1.5 (0.2)
SBCA-A-W	0.35 (0.02)	6.9 (0.8)	1.5 (0.2)

## DISCUSSION AND CONCLUSIONS

The results showed that the hydrophobic agents delay the hydration of both, Portland cement and Portland composite cement with fly ash. Though the agent E in powder form can compensate this to a certain degree by early reaction of the silica carrier with the cement, it cannot prevent the loss in strength until 28 days. However, by changing the setting process utilizing an alkali free set accelerator, some of the delays are alleviated. In particular agent W showed a strength development towards higher values compared to the specimen without accelerator. Surprisingly, the effects of the agents on bond strength were minimal for both, laboratory and sprayed specimen. For the laboratory samples the agents performed well having high contact angles and a strong reduction in water absorption coefficients. The performance of the sprayed specimen, however, was less pronounced. Crucial were the environmental conditions for the hydrophobic agents to condensate in order to make them fully functional.

Smaller samples dry out quicker and help the condensation process of the silane/siloxanes, which requires a moderate relative humidity within the specimen. Larger samples of a denser concrete take much longer to reach these moderate humidity levels. It might be that the active compounds in agent W were not fully condensed and therefore not entirely functional. However, this raises also a critical question for the application of silane/siloxane systems in tunnels, where generally higher humidity levels, in particular within the concrete, can be expected. In unfavourable conditions the moisture levels in the sprayed concrete might never reach a level, where the silanes/siloxanes can fully condensate.

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