SILKO test programme of liquid membrane forming curing compounds of concrete 2011

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Summary

This is an unofficial translation of the Finnish research report. In case of interpretation disputes the Finnish report.

These test instructions are to be used in examining the suitability of liquid membrane forming curing compounds of concrete for outdoor concrete structures owned by the Finnish Transport Agency (Former Finnish Road Administration). On the basis of test results Finnish Transport Agency decides on the acceptability of liquid membrane forming curing compounds of concrete for their use. The approved materials are published in SILKO-directions which are available on the web pages of Finnish Transport Agency.

There are two types of curing compounds for concrete, evaporation reducers used at an early age and final curing agents applied after final set. The test programme and acceptance criteria for both types of curing compounds are given here.

Part of the SILKO-tests are obligatory and part of them are voluntary. The obligatory tests must be carried out on all protective agents for which the acceptance by Finnish Transport Agency is applied for. The voluntary tests are carried out to prove that a material has a special property suitable for a special case.

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Public

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1 Test programme of liquid membrane forming curing compounds of concrete

The test programmes and acceptance criteria of evaporation reducers used at an early age and final curing agents are given in Tables 1 and 2.

Table 1 shows the tests on the bases of which Finnish Transport Agency decides about the acceptance for their use. These tests are the so called obligatory tests.

Table 2 shows the so called voluntary tests on the bases of which the curing compound may be shown to have some extra properties beneficial for Finnish Transport Agency.

Concrete according to CEN/TC 14754-1 is used in all other tests where concrete is needed except in the test of the effect of curing compound on frost salt durability. In the frost salt test the concrete specimens are prepared of air-entrained concrete MC(0,40) according to the standard EN 1766.
Table 1. Test programmes and acceptance criteria of curing compounds of concrete. Obligatory tests.

<table>
<thead>
<tr>
<th>Property</th>
<th>Evaporation reducers used at an early age</th>
<th>Final curing agents</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test</td>
<td>Criterion</td>
</tr>
<tr>
<td>Identification</td>
<td>EN 1767</td>
<td>-</td>
</tr>
<tr>
<td>Spreadability</td>
<td>- Spraying. Test temperatures +5 °C and +30 °C. Spraying is carried out according to the instructions given by the manufacturer. If the manufacturer does not recommend any method, the size of the nozzle is ( \varnothing \leq 2 \text{mm} ) and spraying pressure is 1 MPa.</td>
<td>- Must be sprayable</td>
</tr>
<tr>
<td>Spraying</td>
<td>- The sprayer must not clog</td>
<td>-</td>
</tr>
<tr>
<td>Spreadability</td>
<td>- Must be applicable by roller or brush</td>
<td>-</td>
</tr>
<tr>
<td>Applicability by roller or brush</td>
<td>- The surface must not be softer that the water cured surface.</td>
<td>-</td>
</tr>
<tr>
<td>Compatibility with concrete</td>
<td>1. Scarping by knife of the surface of the specimen of water retention test CEN/TC 14754-1 or drying rate test ( \geq 72 \text{ h after casting} ) (The method is based on ASTM Designation C 309-3 and ASTM Designation C 1315-03)</td>
<td>1. Scarping by knife of the surface of the specimen of water retention test CEN/TC 14754-1 or drying rate test ( \geq 72 \text{ h after casting} ) (The method is based on ASTM Designation C 309-3 and ASTM Designation C 1315-03)</td>
</tr>
<tr>
<td>Content of water soluble chloride</td>
<td>EN 1744-1, chapter 9</td>
<td>( \leq 0.2 \text{ wt-%} )</td>
</tr>
</tbody>
</table>

continued
Table 1. continues

<table>
<thead>
<tr>
<th>Property</th>
<th>Evaporation reducers used at an early age</th>
<th>Final curing agents</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test</td>
<td>Criterion</td>
</tr>
<tr>
<td>Drying time</td>
<td>SILKO test</td>
<td>Concrete specimens are cast. The surface is finished with steel float. 1 d in moulds covered with plastic sheets.</td>
</tr>
<tr>
<td></td>
<td>Concrete specimens are cast. Curing compound is spread according to the instructions given by manufacturer.</td>
<td>Testing conditions +21 °C, RH 50 %, air velocity parallel to the surface ca. 3 m/s (The method is based on ASTM Designation C 309-3 and ASTM Designation C 1315-03)</td>
</tr>
<tr>
<td></td>
<td>Drying time must be ≤ 4 h</td>
<td></td>
</tr>
<tr>
<td></td>
<td>- Evaporation rate of water from concrete surface treated with evaporation reducer at early age ≤ 0,05 kg/m²/h,</td>
<td>Curing agent must reduce water evaporation 90 % by the times of 24 and 72 h after application of curing agent.</td>
</tr>
<tr>
<td>Water retention ability</td>
<td>CEN/TC 14754-1</td>
<td>- Total amount of evaporated water from concrete surface 6 h after application of evaporation reducer ≤ 0,23 kg/m²;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Evaporation reducer must reduce water evaporation 80 % by the time of 72 h after application of evaporation reducer.</td>
</tr>
</tbody>
</table>

continued
<table>
<thead>
<tr>
<th>Property</th>
<th>Evaporation reducers used at an early age</th>
<th>Final curing agents</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test</td>
<td>Criterion</td>
</tr>
</tbody>
</table>
| Removability of wax based curing compound | VTT TEST 375-93  
Concrete specimens are cast. The surface is finished with steel float. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer.  
At the earliest 7 d after casting the curing compound is removed by the method recommended by the manufacturer. If the manufacturer does not give a recommendation the curing agent is removed by grit blasting. Eater colour is brushed on the surface and immediately wiped by cloth. Visual inspection using magnifying glass. | There must not be non-coloured areas. |
| Applicability on vertical surfaces | SILKO test  
Concrete specimens are cast. The surface is finished with steel float. 1 d in moulds covered with plastic sheets At the age of 1 d curing compound is applied according to the instructions of the manufacturer.  
Testing conditions +21 °C, RH 50 %.  
The surface is inspected visually at the earliest 1 d after the application of curing compound. | There must not be traces of running of the curing compound. |
Table 2. Test programmes and acceptance criteria of curing compounds of concrete. Voluntary tests.

<table>
<thead>
<tr>
<th>Property</th>
<th>Evaporation reducers used at an early age</th>
<th>Siliko test</th>
<th>Final curing agents</th>
<th>Criterion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Effect on soiling</td>
<td>Test: Concrete specimens are cast. The surface is finished with steel float. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. At the earliest 10 d after casting:  - The colour is measured  - A mixture of water and soot is sprayed on the surface  - The surface is flushed with water  - The colour is measured</td>
<td>The result is compared with water cured surface. No criterion</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cleanability</td>
<td>Test: Concrete specimens are cast. The surface is finished with steel float. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. At the earliest 10 d after casting graffiti is made on the surface and the surface is cleaned. Visual inspection.</td>
<td>The result is compared with water cured surface. No criterion</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Performance on surface exposed to direct sunlight</td>
<td>Test: Concrete specimens are cast. The surface is finished with steel float. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. EN ISO 11507: 170 h UV At the earliest 10 d after casting: Visual inspection and scraping of the surface by knife</td>
<td>- The visual appearance must not change because of UV exposure  - The surface must not be softer than the water cured surface.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Effect on frost salt durability</td>
<td>Test: Concrete specimens are cast. The binder is CEM I 42,5 R. The surface is finished with steel float. 1 d in moulds covered with plastic sheets. At the age of 1 d curing compound is applied according to the instructions of the manufacturer. CEN/TC 12390-9 slab test, 3% NaCl solution</td>
<td>Scaling must not be higher than that of the water cured specimens.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
2 Evaporation reducers used at an early age

The properties required of the evaporation reducers used at an early age on bridges do not depend on their used. The required properties are:

- water retention ability
- spreadability,
- compatibility with concrete,
- content of water soluble chloride and
- drying time.

2.1 Test method and criteria

2.1.1 Water retention ability

Test method

Water retention ability is testing according to CEN/TC 14754-1.

Criterion

Evaporation rate of water from concrete surface treated with evaporation reducer at early age $\leq 0.05$ kg/m$^2$/h,

Total amount of evaporated water from concrete surface 6 h after application of evaporation reducer $\leq 0.23$ kg/m$^2$,

Evaporation reducer must reduce water evaporation 80 % by the time of 72 h after application of evaporation reducer.

2.1.2 Spreadability

Test method

The test temperatures are $+5 \pm 2 \, ^\circ C$ and $+30 \pm 2 \, ^\circ C$. The temperature of the compound must be $+5 \pm 2 \, ^\circ C$ and $+30 \pm 2 \, ^\circ C$ respectively. The compound is sprayed according to the manufacturers instructions 1 000 ml in 60 min. The spraying does not have to be continuous.

If the manufacturer does not recommend any other the pressure of 1 $\pm 0.1$ MPa and a nozzle with a diameter of not greater than 2 mm are used.

Criterion

The compound must be sprayable and The sprayer must not clog.

2.1.3 Compatibility with concrete

There are two alternate methods to test the compatibility with concrete.
Test method 1

The compatibility is tested by scraping by knife of the surface of the specimen of water retention test CEN/TC 14754-1 or drying time test described below at the earliest ≥72 h after casting.

Test method 2

The testing conditions are +21 ± 2 °C and RH 50 ± 10 %.

Four concrete specimens are cast. The minimum dimensions of the top surface of the specimens are 150 · 150 mm² and their thickness is 50 mm. Two specimens are cured using curing compound according to the instructions given by the manufacturer and they are removed to RH 50 ± 10 %. Two reference specimens are covered with plastic sheets after casting. 1 d after casting a water layer is put on the surface of the reference specimens. The water layer is removed 7 d after casting. At the age of 7 d the specimens are removed to RH 50 ± 10 %. A the earliest 3 d after removal of the water the surface of all specimens is scraped with knife.

Criterion

The surface cured with curing compound must not be softer than the water cured surface.

2.1.4 Content of water soluble chloride

Test method

The content of water soluble chloride is measured according to EN 1744-1.

Criterion

The content of water soluble chloride must not be higher than 0,2 wt-%.

2.1.5 Drying time

Test method

The testing conditions are +21 ± 2 °C, RH 50 ± 10 % and air velocity parallel to the surface ca. 3 m/s.

Two concrete specimens are cast the minimum dimensions of the upper surface of which are 150 · 150 mm² and the thickness of which are 50 mm. The curing compound is applied according to the instructions of the manufacturer. The surface is tested with a finger until soft tacky condition no longer exists and the film feels firm. Drying time is the time elapsed from curing compound application to the dry to touch condition.

Criterion

Drying time must not exceed 4 h.
3 Final curing agents

The properties required of the final curing agents depend on their use. The properties required in different uses on bridges are:

- water retention ability
- spreadability: applicability by spray, brush or roller
- compatibility with concrete
- content of water soluble chloride
- drying time
- applicability on vertical surfaces, when the compound is used on vertical surfaces
- removability of wax based curing compound
- performance on surface exposed to direct sunlight, when the compound is used on a surface exposed to direct sunlight during the hardening of concrete
- effect on frost salt durability, when the compound is used on a surface exposed to frost and salt and it is not removed
- effect on soiling, when the compound is used on a visible surface and it is not removed
- cleanability, puhdistettavuus, when the compound is used on a visible surface and it is not removed.

3.1 Test methods and criteria

3.1.1 Water retention ability

*Test method*

Water retention ability is testing according to CEN/TC 14754-1.

*Criterion*

Curing agent must reduce water evaporation 90 % by the times of 24 and 72 h after application of curing agent.

3.1.2 Spreadability

3.1.2.1 Sprayability

*Test method*

The test temperatures are \(+5 \pm 2 \, ^\circ C\) ja \(+30 \pm 2 \, ^\circ C\). The temperature of the compound must be \(+5 \pm 2 \, ^\circ C\) and \(+30 \pm 2 \, ^\circ C\) respectively. The compound is sprayed according to the manufacturers instructions 1 000 ml in 60 min. The spraying does not have to be continuous.

If the manufacturer does not recommend any other the pressure of ca. 1 MPa and a nozzle with a diameter of not greater than 2 mm are used.
Criterion

The compound must be sprayable and the sprayer must not clog.

3.1.2.2 Applicability by brush/roller

Test method

The test temperatures are +5 ±2 °C and +30 ±2 °C. The temperature of the compound must be +5 ±2 °C and +30 ±2 °C respectively.

Four concrete specimens are prepared/application method. The dimensions of the specimens are 150 × 150 × 50 mm³. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of 1 d, 2 specimens/application method are removed to the temperature of +5 ± 2 °C and two to the temperature of +30 ± 2 °C. After 2 h curing the plastic sheets are removed. When the surface is mat dry, the curing agent is applied on all four specimens according to the instructions given by the manufacturer either by brush or by roller.

Criterion

The curing compound must be applicable by roller or brush so that a layer thickness according to the instructions given by the manufacturer is obtained.

3.1.3 Compatibility with concrete

There are two alternate methods to test the compatibility with concrete.

Test method 1

The compatibility is tested by scraping by knife the surface of the specimens of water retention test CEN/TC 14754-1 or drying time test described below at the earliest ≥72 h after casting.

Test method 2

The testing conditions are + 21 ± 2 °C and RH 50 ± 10 %.

Four concrete specimens are cast. The minimum dimensions of the top surface of the specimens are 150 × 150 mm² and their thickness is 50 mm. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. The plastic sheets are removed at the age of 1 d followed by application of a water layer on two specimens. When the surfaces of the two remaining specimens are mat dry curing compound is applied according to the instructions given by the manufacturer after which they are removed to RH 50 ± 10 %. At the age of 7 d the water layer is removed and the specimens are removed to RH 50 ± 10 %. At the earliest 3 d after the removal of the water layer the surface of the specimens is scraped with knife.
Criterion

The surface cured with curing compound must not be softer than the water cured surface.

3.1.4 Content of water soluble chloride

Test method

The content of water soluble chloride is measured according to EN 1744-1 chapter 9.

Criterion

The content of water soluble chloride must not be higher than 0.2 wt-%.

3.1.5 Drying time

Test method

The testing conditions are +21 ± 2 °C, RH 50 ± 10 % and air velocity parallel to the surface ca. 3 m/s.

Two concrete specimens are cast the minimum dimensions of the upper surface of which are 150 · 150 mm² and the thickness of which are 50 mm. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of 1 d the plastic sheets are removed. Curing compound is applied on mat dry surface specimens according to the instructions given by the manufacturer. The surface is tested with a finger until soft tacky condition no longer exists and the film feels firm. Drying time is the time elapsed from curing compound application to the dry to touch condition.

Criterion

Drying time must not exceed 4 h.

3.1.6 Applicability on vertical surfaces

Test method

The testing temperature is +21 ± 2 °C.

A concrete specimen is cast the minimum dimensions vertical surface to be treated with curing compound are 150 · 150 mm². For the first day the specimen is cured in its mould covered with a plastic sheet. At the age of 1 d the plastic sheet is removed. The curing compound is applied according to the instructions given by the manufacturer on the vertical concrete surface. The surface is inspected visually not earlier than 24 h after curing compound application.

Criterion

There must not be traces of running of the curing compound.
3.1.7 Removability of wax based curing compound

*Test method*

The testing conditions are $+21 \pm 2 \, ^\circ\text{C}$, RH $50 \pm 10 \%$.

A concrete specimen is cast the minimum dimensions of the upper surface of which is $150 \cdot 150 \, \text{mm}^2$ and the thickness of which is $50 \, \text{mm}$. The upper surface is finished with steel float. For the first day the specimen is cured in its mould covered with a plastic sheet. At the age of $1 \, \text{d}$ the plastic sheet is removed. The curing compound is applied on mat dry surface of the specimen according to the instructions given by the manufacturer. At the earliest $7 \, \text{d}$ after casting the curing compound is removed following the instructions given by the manufacturer. If the manufacturer does not give instructions the curing compound is removed by grit blasting.

The remnants of the curing compound are inspected according to VTT TEST 375-93. Water colour is applied by brush on the surface. The brush must not be dripping wet. Immediately while the surface is still wet the surface is wiped with a dry cloth.

The surface is inspected visually using a magnifying glass. There are no remnants of curing compound where the surface is coloured by water colour. Where as the not coloured surfaces indicate curing compound remnants

*Criterion*

There must not be uncoloured areas on the surface treated with curing compound.

3.1.8 Performance on surface exposed to direct sunlight (Voluntary)

*Test method*

Two concrete specimens are cast the minimum dimensions of the upper surface of which are $150 \cdot 150 \, \text{mm}^2$ and the thickness of which are $50 \, \text{mm}$. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of $1 \, \text{d}$ the plastic sheets are removed. A water layer is applied on one specimen and curing compound is applied on mat dry surface of the other specimen according to the instructions given by the manufacturer. The water layer is maintained for $6 \, \text{d}$. At the age of $7 \, \text{d}$ the specimen is removed to RH $50 \pm 10 \%$. Immediately after the application of he curing compound the surface is exposed to UV for $170 \, \text{h}$ according to EN ISO 11507 after which the specimen is removed to RH $50 \pm 10 \%$.

At the earliest $3 \, \text{d}$ after the end of the exposure to UV the surfaces are inspected visually and they are scraped with knife.

*Criterion*

The surface cured with curing compound must not be softer than the water cured surface and the visual appearance must not change because of UV exposure.
3.1.9 Effect on frost salt durability (Voluntary)

Test method

Four concrete specimens are prepared of air-entrained concrete MC(0,40) according to the standard EN 1766. The dimensions of the specimens are 150 · 150 · 50 mm³. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. The plastic sheets are removed at the age of 1 d followed by application of a water layer on two specimens. When the surfaces of the two remaining specimens are mat dry curing compound is applied according to the instructions given by the manufacturer. At the age of 7 d the water layer is removed and all the specimens are removed to +21 ± 2 °C and RH 65 ± 10 %. At the age of 25 ± 1 d a rubber mat is glued on all other but test surfaces according to CEN/TS 12390-9. A frost salt test according to CEN/TS 12390-9 slab test using 3-% NaCl solution is carried out.

Criterion

Scaling must not be higher than that of the water cured specimens.

3.1.10 Effect on soiling (Voluntary)

Test method

The testing conditions are +21 ± 2 °C, RH 50 ± 10 %.

Four concrete specimens are cast the minimum dimensions of the surface to be treated with curing agent are 150 · 150 mm². The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. The plastic sheets are removed at the age of 1 d followed by application of a water layer on two specimens. When the surfaces of the two remaining specimens are mat dry curing compound is applied according to the instructions given by the manufacturer. At the age of 7 d the water layer is removed and the specimens are removed to RH 50 ± 10 %.

At the earliest 3 d after the removal of water layer the surfaces of the specimens are inspected visually and the colour of the surfaces is measured using spectrophotometer. At least three points of every surface is measured. The test surfaces are placed vertically and they are soiled by lightly spraying Degussa Printex 300 furnace black powder mixed in water on them. When the surfaces have dried they are inspected visually and their colour is measured. The colour change of the soiled surfaces compared to the colour of original clean surfaces of the with curing compound treated specimens is $\Delta E_{JHA-1}$ and that of the water cured reference specimens $\Delta E_{VERT-1}$. The absolute value of the remainder, $|\Delta E_{CUR-1} - \Delta E_{REF-1}|$, must not exceed 1,0. The test surfaces are carefully flushed with water. After drying they are visually inspected and their colour is measured.

The means of the change of colour at the end of the test compared to the colour of the original clean surfaces is calculated, $\Delta E_{CUR-2}$ by $\Delta E_{REF-2}$
Criterion

The result is compared with water cured surface. There is no criterion.

3.1.11 Cleanability (Voluntary)

Test method

The testing conditions are $+21 \pm 2 ^\circ\text{C}, \text{RH} \ 50 \pm 10 \%$.

Six concrete specimens are prepared. The minimum dimensions of the surface to be cured with curing agent are $150 \cdot 150 \text{ mm}^2$. The upper surface is finished with steel float. For the first day the specimens are cured in their moulds covered with plastic sheets. At the age of 1 day the plastic sheets are removed. A water layer is applied on three specimens and the curing agent is applied on three specimens according to the instructions given by the manufacturer when the surface is mat dry. At the age of 7 day the water layer is removed and the specimens are removed to RH $50 \pm 10 \%$.

At the earliest 3 days after the removal of water layer the specimens are inspected visually. The surface of two specimens cured with curing compound and two water cured specimens are divided in three sections. Graffiti are drawn on one section using paint from a spray can, on one section using alkyl paint and on one section using a water-proof felt pen. At the age of 35 day one specimen cured with curing compound and one water cured specimen are cleaned using (high-pressure) water cleaning (using hot water) and one using a cleaning agent. When the surfaces have dried they are inspected visually to evaluate how effectively the applied graffiti have been removed by comparing their appearance to that of the specimens with no graffiti.

The result evaluated numerically using scale from 0 to 5. 0 means that the graffiti does not remove at all and 5 means that the graffiti is totally removed. The cleanability is compared to that of the water cured reference specimens.

Criterion

The result is compared with water cured surface. There is no criterion.

References

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32 p.