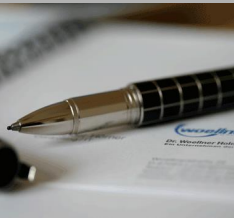




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Soda ash/polysilicates cogranules and Biodegradable chelates in Laundry powders



Serge Mathieu
SEPAWA Nordic 2013 May 6th.-7th.

Agenda

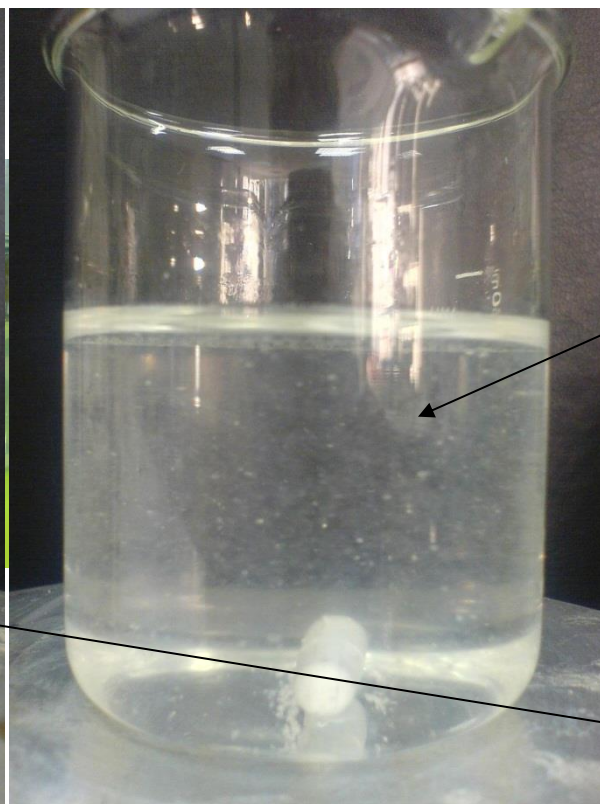
- Is soda ash –sodium silicate a precipitating builder?
- Protocol for calcium precipitation test.
- Performance of Nabion® on calcium precipitation reduction.
- Comments.
- Protocol for mineral incrustation & detergency tests.
- Performance of Nabion® on mineral incrustation & detergency tests.
- Conclusions

Is soda ash –Na silicate a precipitating builder?

Test has been performed with Ca²⁺ binding kinetic protocol together with turbidity measurements



Hydrated silicate



Nabion®

The solution is still clear during 7 minutes : no Ca precipitation, no insoluble matter due to the dispersing power of Nabion®26

Cloudy solution after 1 minute Ca-silicate/Ca carbonate complexes precipitation

Calcium binding Kinetic Test.

conditions with variable parameters

- Temperature: 25°C
- Water hardness: 30°THf
- pH: 9 - 9.5
- 1 g/l builder
- Volume in solution: 100 ml
- Mixing: 300 rpm
- ISE - Ca²⁺ electrode DX 240 Metrohm
- Reference electrode Epoxy Orion
- Calibration slope before the kinetic:
21 - 29 mV/log Ca²⁺



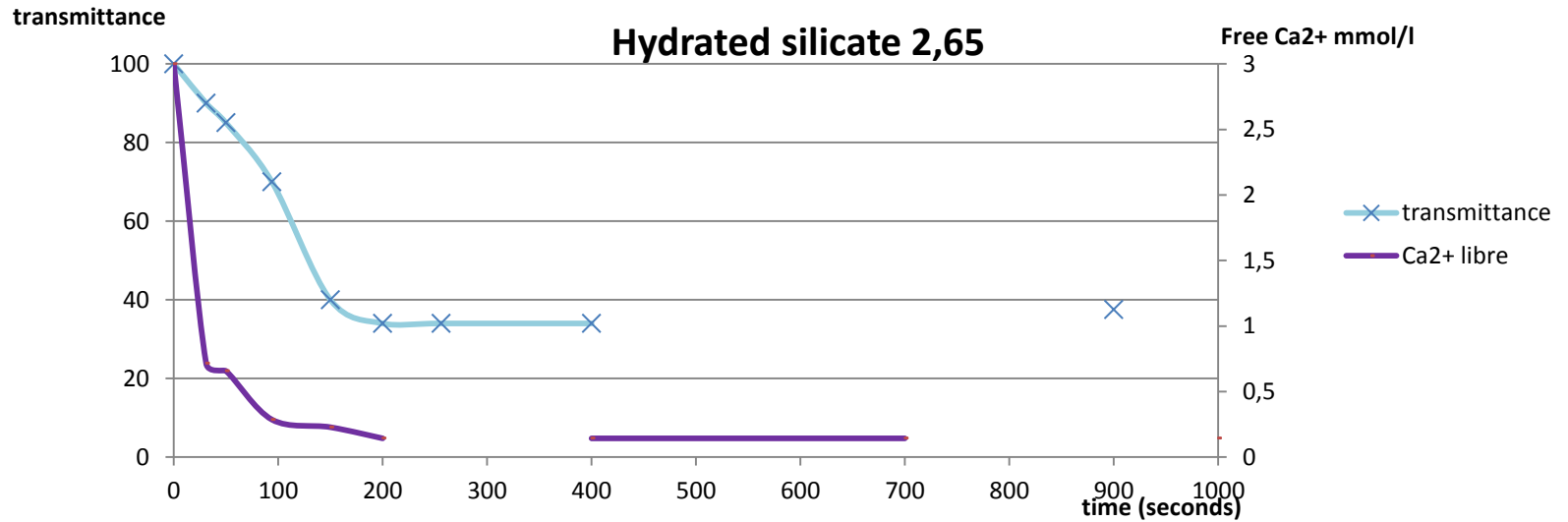
Results:

In according to the Nernst equation $\Delta E = + 30 \cdot \log [Ca^{2+}_{free}]$

The electrode measures the concentration of $[Ca^{2+}_{free}]$ during the kinetic

This amount is in relation with the % Ca²⁺_{bind}

Protocol for calcium binding kinetic and performances

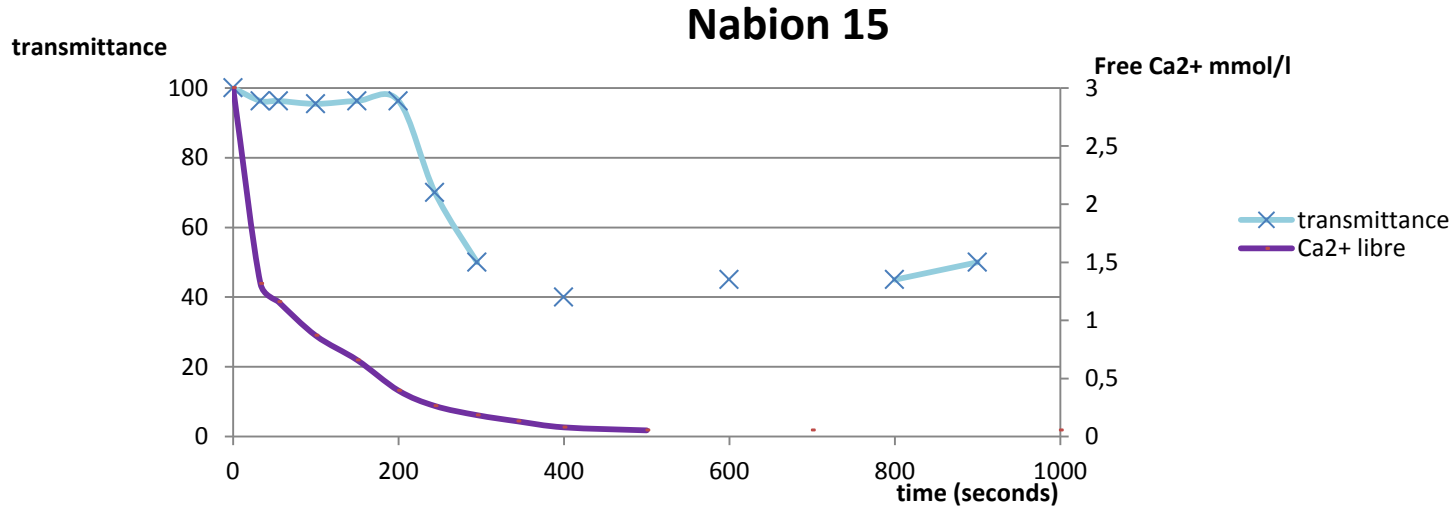


The Ca²⁺ curve shows that **90%** of free calcium ion is consumed by the soda ash within 100s.

The transmittance curve shows that the calcium ion is reacting with the soda ash to generates calcium carbonate complexes precipitation in **1mn**.

The dissolution time of the hydrated silicate granules do not support the calcium carbonate precipitation reduction.

Protocol for calcium binding kinetic and performances

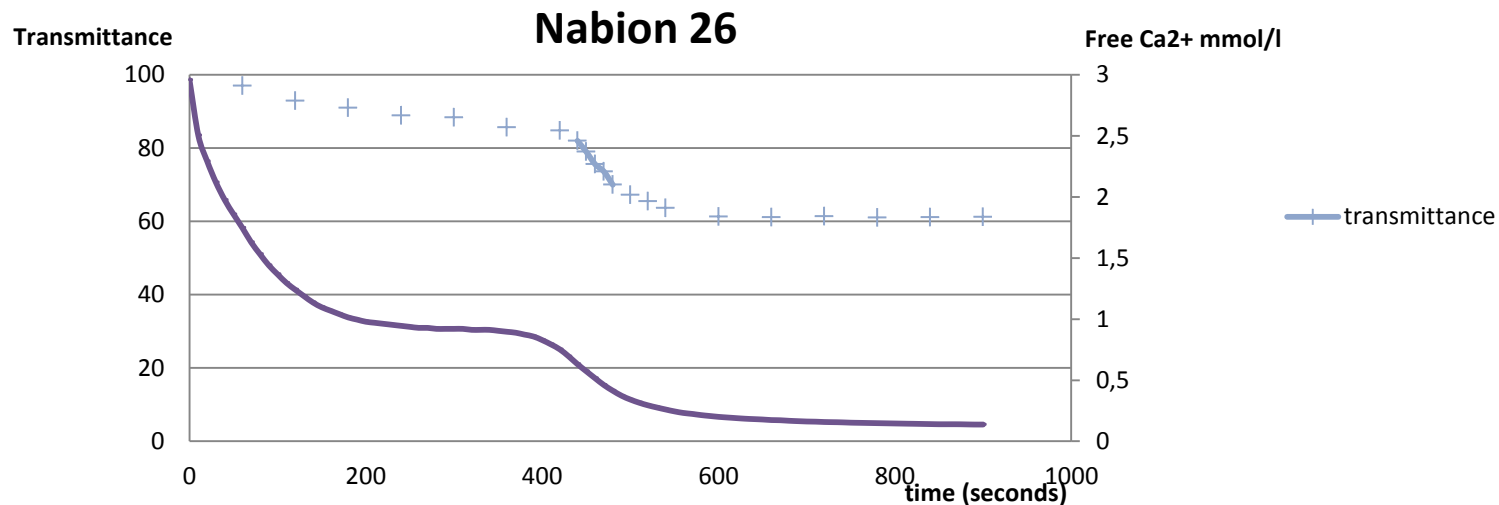


The Ca²⁺ curve shows that **75%** of free calcium ion is consumed by the soda ash within 100s.

The transmittance curve shows that the calcium ion is reacting with the soda ash to generates calcium carbonate complexes which remains dispersed for at least **4 mn** before precipitation.

The dissolution speed of the Nabion®15 granules release the silicate species for complexes calcium carbonate growth inhibition.

Protocol for calcium binding kinetic and performances

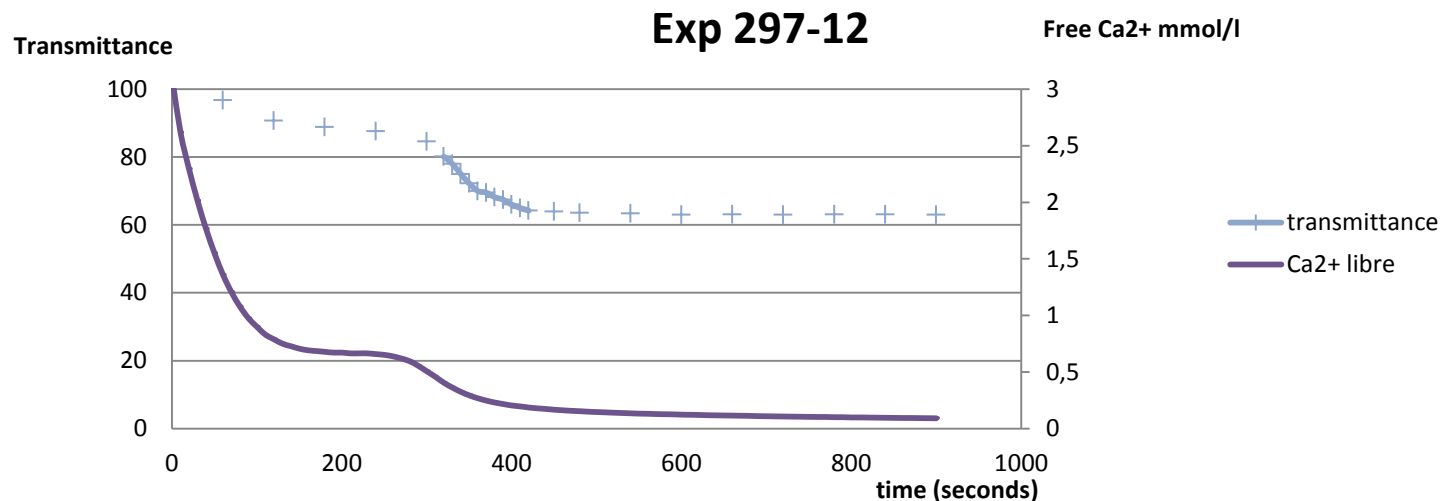


The Ca²⁺ curve shows that **60%** of free calcium ion is consumed by the soda ash within 100s.

The transmittance curve shows that the calcium ion is reacting with the soda ash to generate calcium carbonate complexes which remain dispersed for at least **7 mn** before precipitation.

The dissolution speed of the Nabion®26 granules releases the silicate species for calcium carbonate growth inhibition.

Protocol for calcium binding kinetic and performances



The Ca²⁺ curve shows that **75%** of free calcium ion is consumed by the soda ash within 100s.

The transmittance curve shows that the calcium ion is reacting with the soda ash to generates calcium carbonate complexes which remains dispersed for at least **6 mn** before precipitation.

The dissolution speed of the EXP 297-12 [Nabion26@90%-Glutamic chelate@10%] granules release the silicate species for complexes calcium carbonate growth inhibition. The part of the glutamic type chelate consume the free calcium ion.

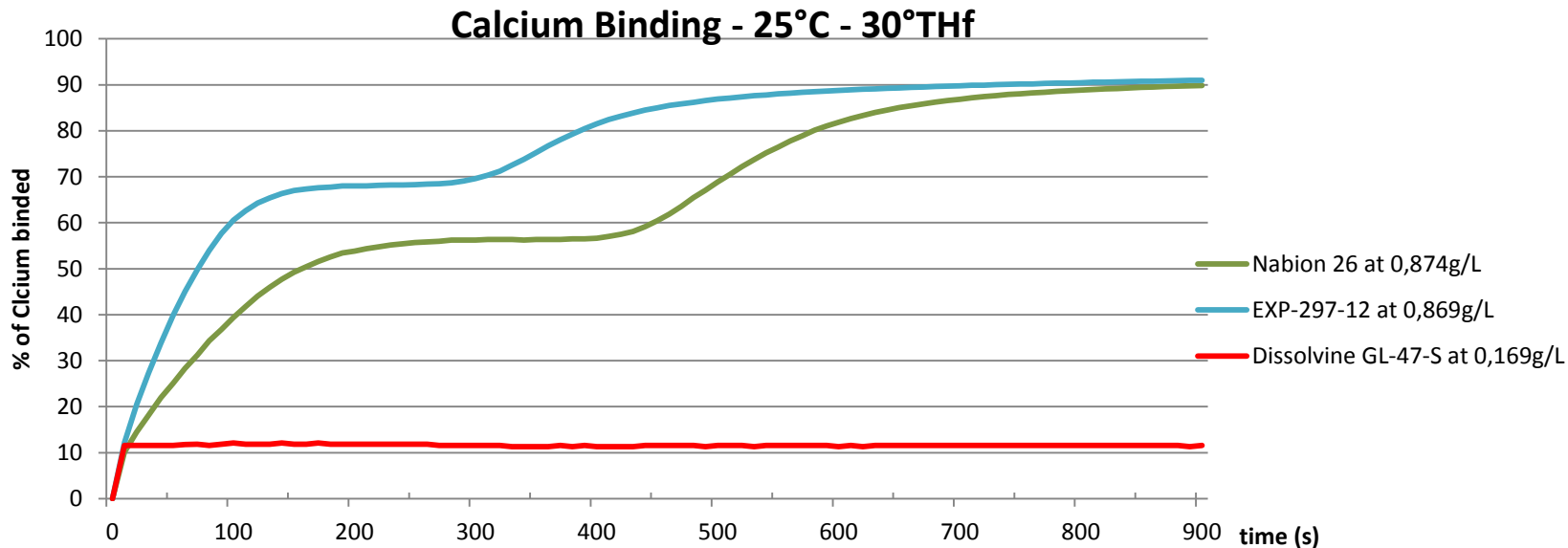
Comments

Soda ash and sodium silicate based builder leads to calcium carbonate precipitation complexes and thus generates mineral incrustation onto the washed fabrics.

Soda ash/sodium silicates granules tends to reduce the calcium carbonate complexes precipitation. Silicates ratio 2,65 further reduced the complexes precipitation.

Glutamic chelates when granulated with soda ash/sodium silicate further improved Calcium ion complexation even at low dosage.

Combined Nabion® and Glutamic chelate calcium binding kinetic



According Calcium binding performance, incrustation and detergency tests were carried out with 17% Nabion®/EXP 297-12 leaving glutamic chelate amount of 1,6% in the laundry powder formulations.

Mineral incrustation test of Nabion® granules

Mineral incrustation test.

conditions with variable parameters

- Temperature: 60°C - 90°C
- Water hardness: 200-400 ppm, as CaCO₃ (Ca/Mg: 3:1)
- 5 washing cycles of 20 min. in a Tergotometer
- 3 rinses after each washing cycles
- Detergent dosage: 5g/l

- 3 test fabrics 8x8cm of: white flat cotton CN 11 and white terry cotton W12A

Results:

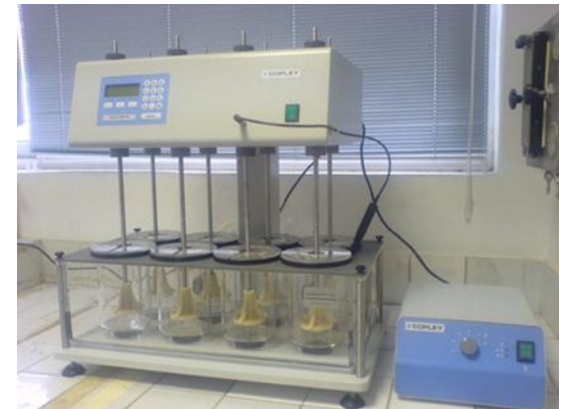
$$\text{Deposit (\%)} = ((m_2 - m_1) / m_0) \times 100$$

[m₀ = mass of the dry sample]

[m₁ = mass of the empty melting pot]

[m₂ = mass of the melting pot + mass of ashes]

- The fabrics are burned at 800°C for 2-3h to obtain ashes.



Mineral incrustation test of Nabion® granules

Products	type / trade name	Nabion 26 % in detergent	EXP 297-12 % in detergent	Soda ash + silicate + glutamic chelate % in detergent
Na-carbonate	Solvay	7	7	13,8
Na-sulfate		28,1	25,11	29,13
polymer	Sokalan CP5/BASF	1	1	1
EXP 297-12	Woellner		17,4	
Nabion 26	Woellner	14,4		
silicate Rp2,6	Woellner	4	4	
glutamic chelate @47%	Akzo			1,6
sikalon A	Woellner			9
		54,50	54,50	54,50

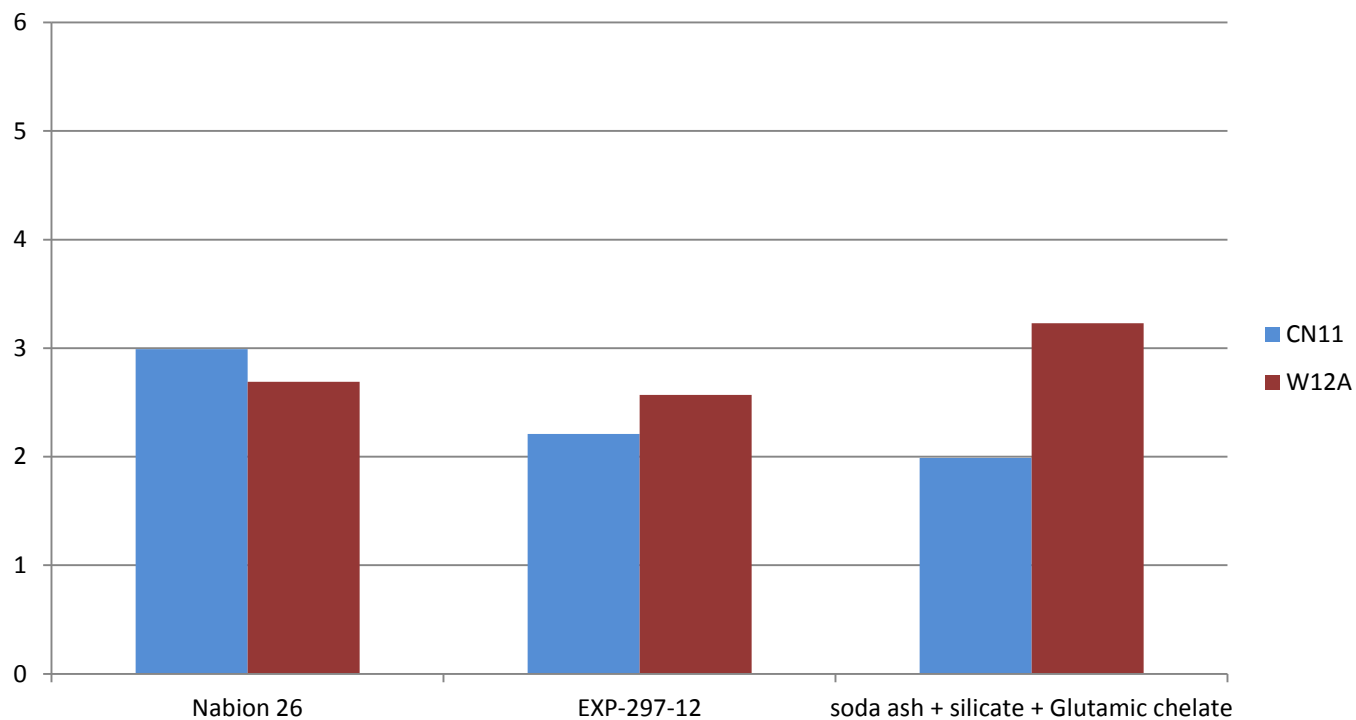
Water hardness (g/l)	30°F
Washing Temperature (°C)	30°C
Detergent dosage (g/l)	5

- 3 laundry powders based builder were evaluated to further evaluate the performance of
- Nabion 26 as reference @ 14,4%.
 - EXP 297 @ 17,4%.
 - Soda ash + Na-silicate + glutamic chelate @47%.

No phosphonate is added in the formula.

Mineral incrustation test of Nabion® granules

incrustation after 5 cycles = f(formula)



CN11: plain cotton

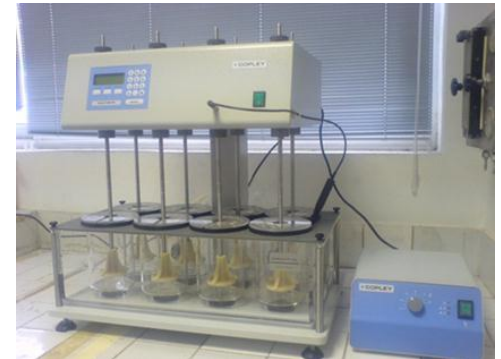
W12A: Terry towel

Detergency test of Nabion® granules

Detergency test conditions

with variable parameters

- Temperature: 20-40°C
- Water hardness: 200-400 ppm as CaCO₃ (Ca/Mg: 3:1)
- 1 cycle = 1 washing phasis of 20 min. + 3 x 5min. of rinsing phasis
- Detergent dosage: 5g/l
- pH: 10,5
- Immersion ratio : 1/32 (weight fabrics / water)
- Soiled fabrics



Results:

The total colorimetric difference $\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$

L, a, b are measured in the Hunter LAB system, before and after the cycles:

$$\Delta L = L_{\text{before}} - L_{\text{after}}$$

$$\Delta a = a_{\text{before}} - a_{\text{after}}$$

$$\Delta b = b_{\text{before}} - b_{\text{after}}$$

The measurements are made with a chromameter CR 310 from Konica Minolta

Detergency test of Nabion® granules

Products	type / trade name	Nabion 26 % in detergent	EXP 297-12 % in detergent	Soda ash + silicate + glutamic chelate % in detergent
Na-carbonate	Solvay	7	7	13,8
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silicate Rp2,6	Woellner	4	4	
glutamic chelate @47%	Akzo			1,6
sikalon A	Woellner			9
		54,50	54,50	54,50

Water hardness (g/l)	30°F
Washing Temperature (°C)	30°C
Detergent dosage (g/l)	5

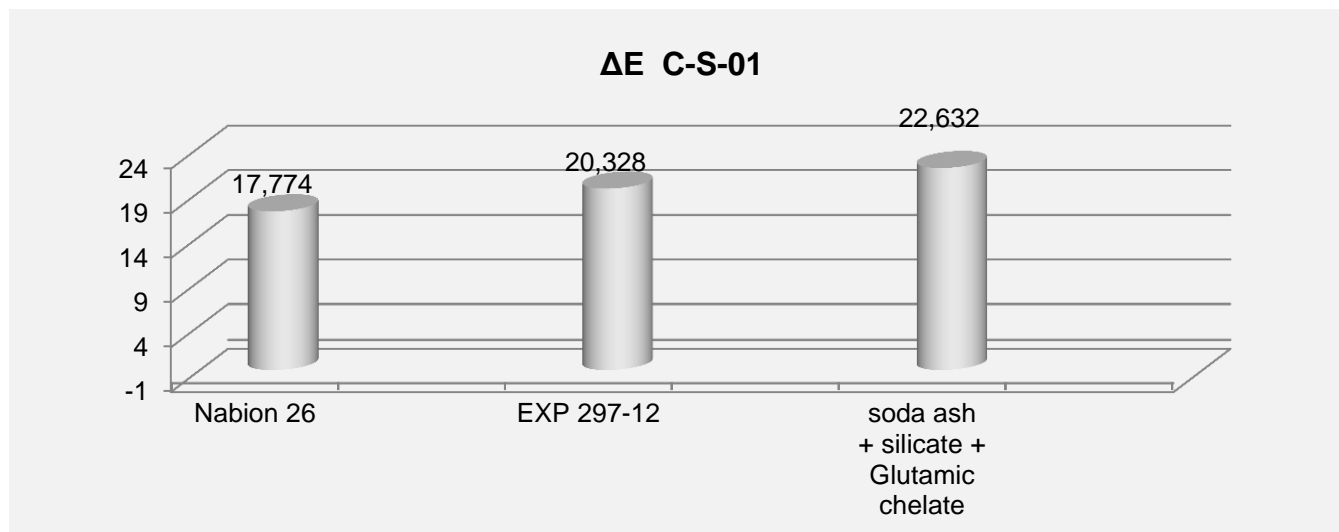
C-S-01 = Blood aged - Cotton (CFT)

W-10 D = Pigment/Sebum - Cotton (CFT-WFK)

E-104 = Carbon Black / olive oil polyester/cotton (CFT-EMPA)

C-10 = Pigment, oil, milk on cotton

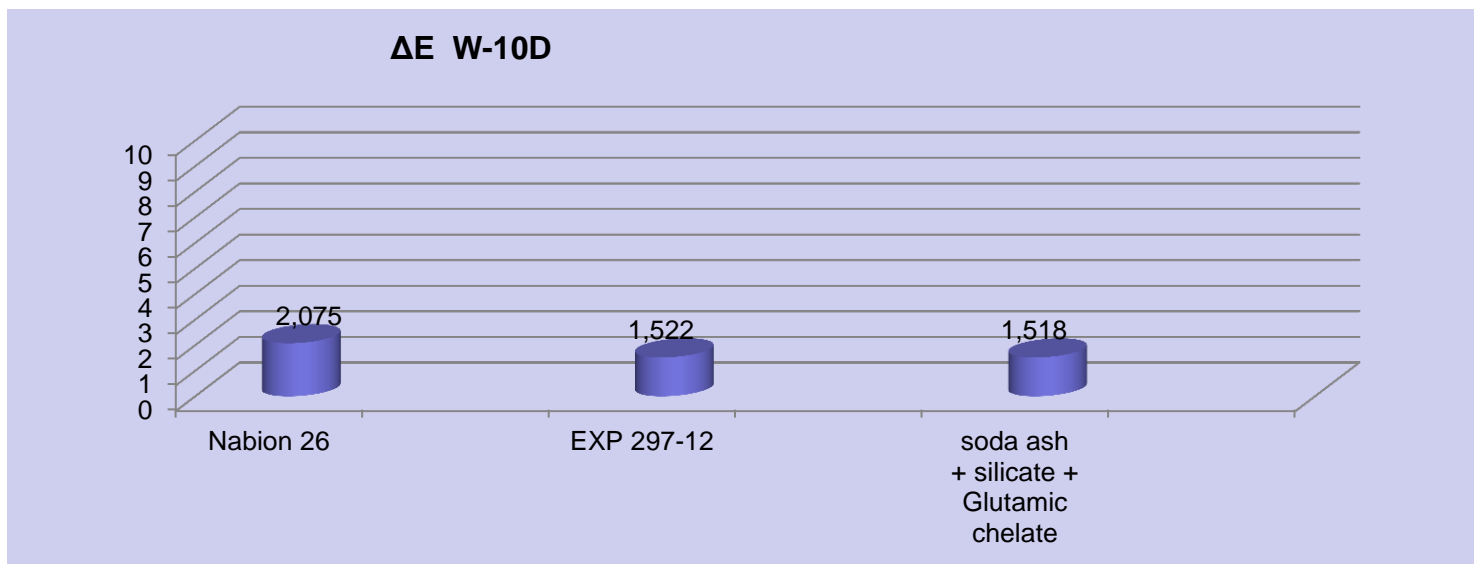
Detergency test of Nabion® granules C-S-01



level of significance :

- deviation ≤ 2 points in cumulative detergency = no difference
- deviation > 2 points and ≤ 5 points in cumulative detergency = small to moderate difference
- deviation > 5 points in cumulative detergency = significant difference

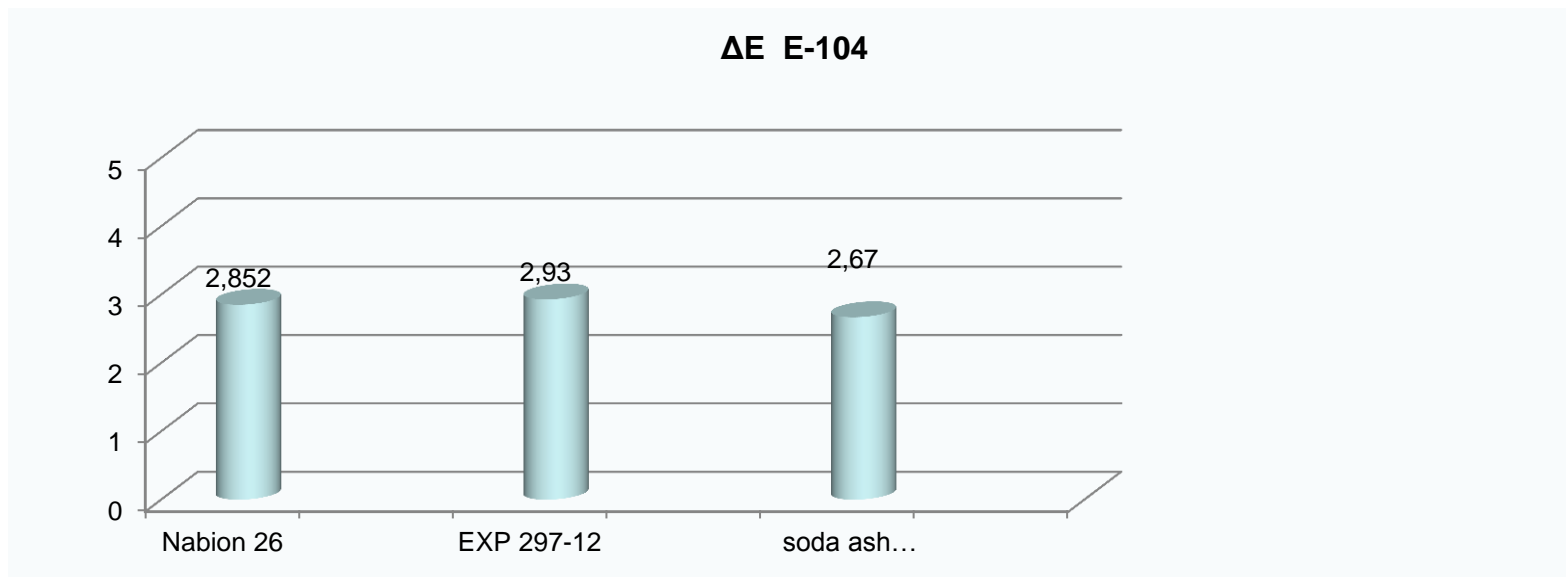
Detergency test of Nabion® granules W-10D



level of significance :

- deviation ≤ 2 points in cumulative detergency = no difference
- deviation > 2 points and ≤ 5 points in cumulative detergency = small to moderate difference
- deviation > 5 points in cumulative detergency = significant difference

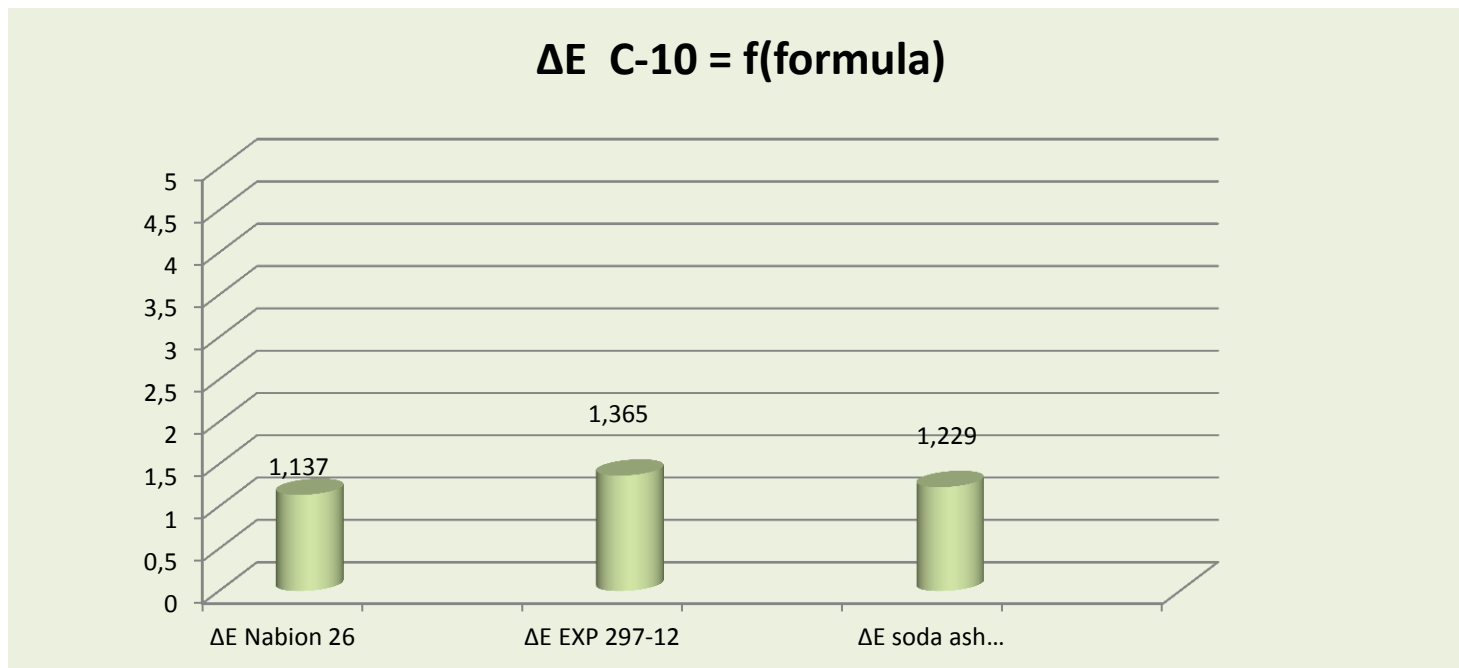
Detergency test of Nabion® granules E-104



level of significance :

- deviation ≤ 2 points in cumulative detergency = no difference
- deviation > 2 points and ≤ 5 points in cumulative detergency = small to moderate difference
- deviation > 5 points in cumulative detergency = significant difference

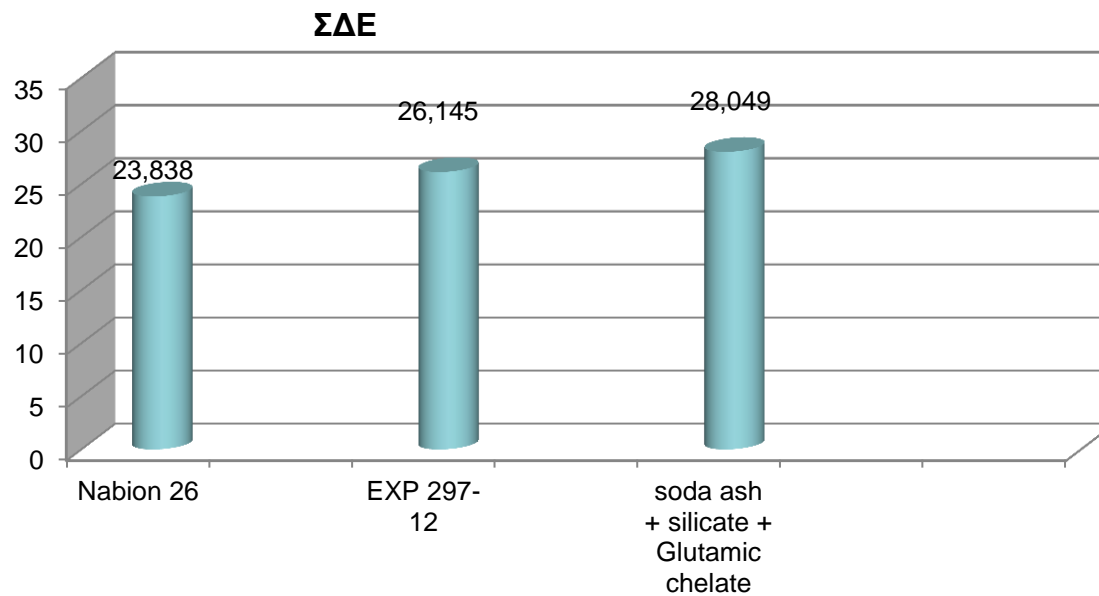
Detergency test of Nabion® granules C10



level of significance :

- deviation ≤ 2 points in cumulative detergency = no difference
- deviation > 2 points and ≤ 5 points in cumulative detergency = small to moderate difference
- deviation > 5 points in cumulative detergency = significant difference

Detergency test of Nabion® granules, overall performance



level of significance :

- deviation ≤ 2 points in cumulative detergency = no difference
- deviation > 2 points and ≤ 5 points in cumulative detergency = small to moderate difference
- deviation > 5 points in cumulative detergency = significant difference

Product characteristics:

- dust-free granules
- fast-dissolving silicate based builder
- fast and complete dissolution even at low temperatures
 - delivers soluble polysilicates
- minimum of insolubles
- resistant to attrition
- cost saving
- post addition for dry mixing processes

Washing performance:

- fast and efficient elimination of calcium and magnesium
- support on alkalinity and buffering effects
- reducing incrustation
- reduces loss of whiteness
- improvement of soils suspensions
- treats coloured fabrics with care
- no effects on the environment



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**Thank you
very much.....**

.....For your attention!

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