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F.A. Finger -Institut für Baustoffkunde

HÁSKÓLINN Í REYKJAVÍK
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Interaction of cement and admixtures – and its influence on rheological properties

S. Kubens

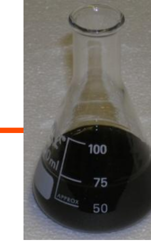
19. Kolloquium und Workshop
Rheologische Messungen an Baustoffen
März 2010 in Regensburg

ICI
Rheocenter

Content

- Objective and scope of project
  - Cement-admixture interaction, rheology and routine cement production
- State of scientific and technical knowledge
  - Polymers and their dispersing power
- Methods used
  - Standard tests, mineralogy, static and dynamic PSD, rheological measurements, calorimetry, polymer adsorption, pore solution analysis and microstructure
- Results
- Final remarks

Introduction



- Annual concrete production is 8 billion cubic metres and most of it contains dispersing admixtures
- Dispersing admixtures play an important role in today's concrete world

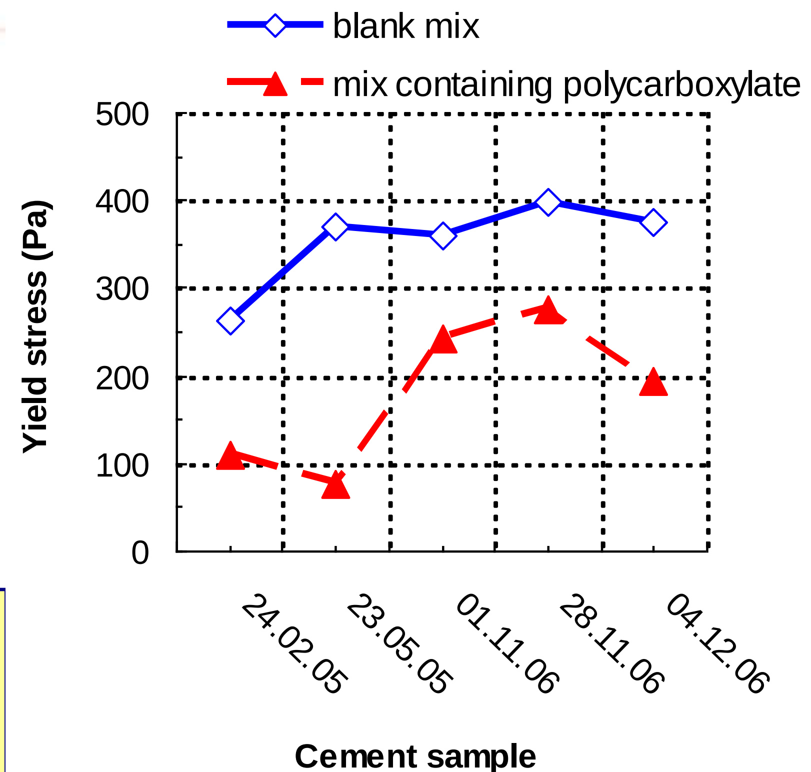
Interaction

Objective and scope of project

- Tests on mortar showed a strong effect of cement production on rheological properties

Blank mix w/c = 0,46
No admixture

PC mix w/c = 0,36
0,11% PC1



Each mortar mix repeated at least 3 times

Cement is **only** differing in production date

Slump flow of mortar

- Same mix design, only differing in production date of cement (from same plant and same production line)

Water to cement ratio: 0,40
Polymer content: 0,20% PC1

Figure 5.2.1.1.5: Mini slump of mortar produced with high yield cement

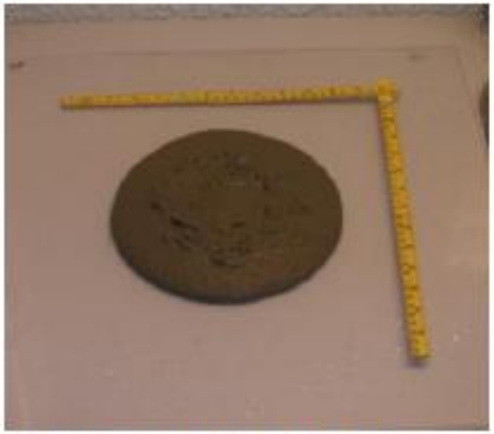


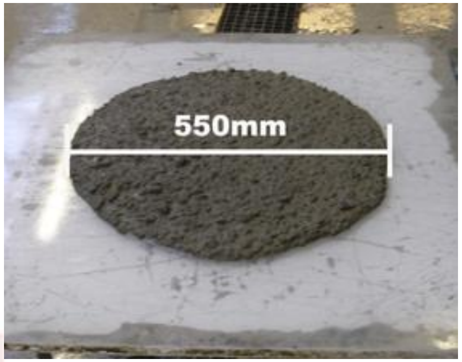
Figure 5.2.1.1.6: Mini slump of mortar produced with intermediate cement

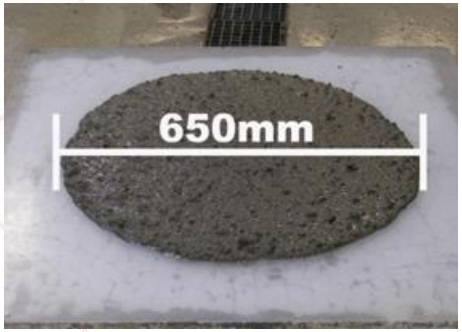
Figure 5.2.1.1.7: Mini slump of mortar produced with low yield cement

Cement-admixture interaction in practice

- Cement-admixture interaction can result in too liquid or too stiff concrete
- Too stiff concrete loses its self-levelling properties
- Too liquid concrete will segregate or increase form pressure

Eco-SCC
Water to cement ratio: 0,60
Polymer content: 0,30% PC1





Aim of study

- Measure and evaluate the effect of variations during routine cement production on the rheology of mortar and concrete
- Identify the constituents in cement which lead to fluctuations in rheology
- Quantify the effect of production-related variation on concrete

Essential for cement and concrete producers to guarantee a homogeneous concrete production plus deeper understanding of cem-admix interaction

State of scientific and technical knowledge

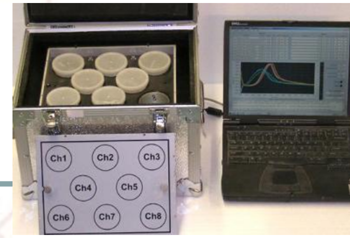
Brief overview

Admixture	Constituent	Interaction
Lignosulfonate Polynaphthalene sulfonate Sulfonated Melamine	•Clinker surface •Chemical composition •Molecular weight of admixture •Charge density •Counterion	•Sharp retardation •Hydrate growth inhibitor •Selective blocking of reactive surfaces •Competitive adsorption between admixture and sulfates
Polycarboxylate	•Alkali sulfates •Charge density •Side chain number and length	Shrinkage of side chains Organo-mineral phases

My contribution

1) Systematic study on factors affecting polycarboxylates
2) Quality control tool by rheological approach

Methods used



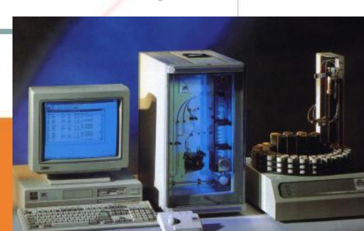
Calorimetry

Rheology

Big picture

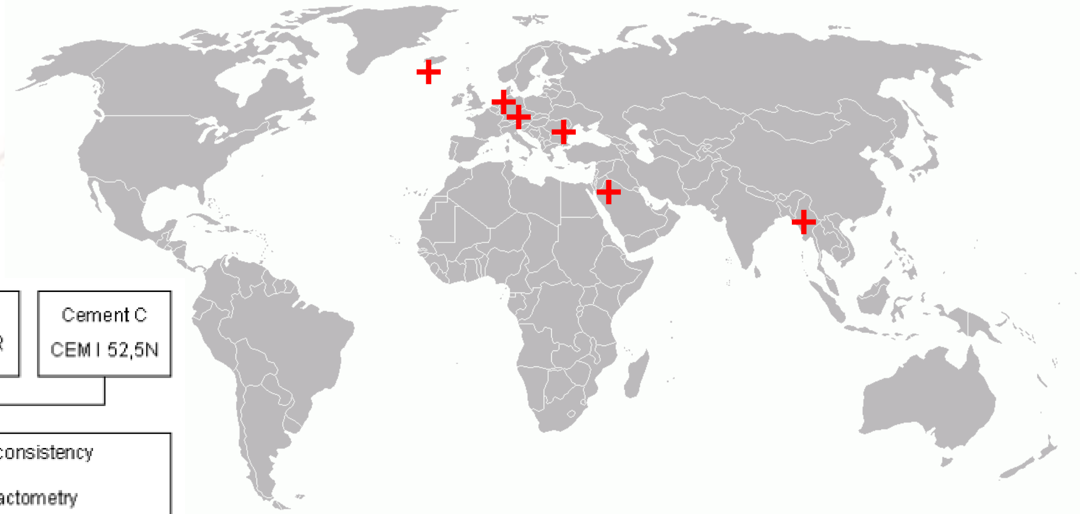
Scanning Electron Microscopy

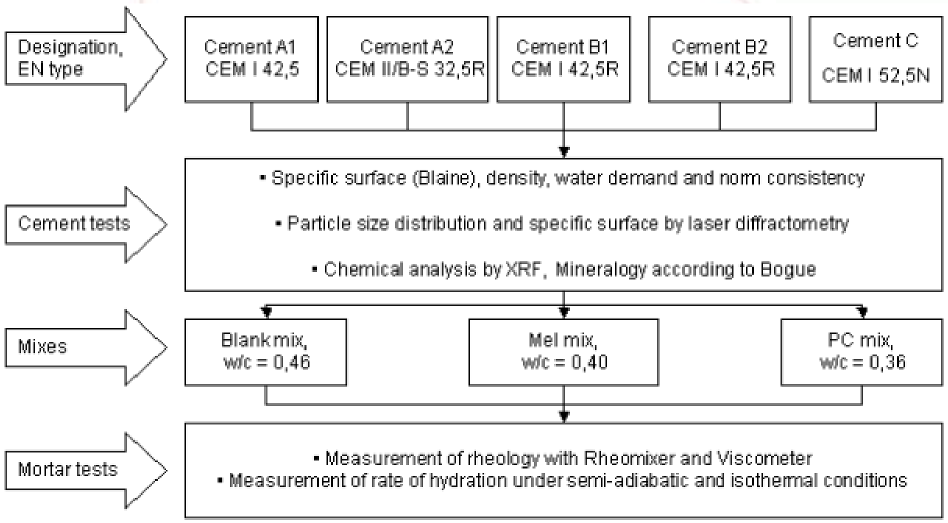
Chemistry,
Pore solution analysis,
adsorption data



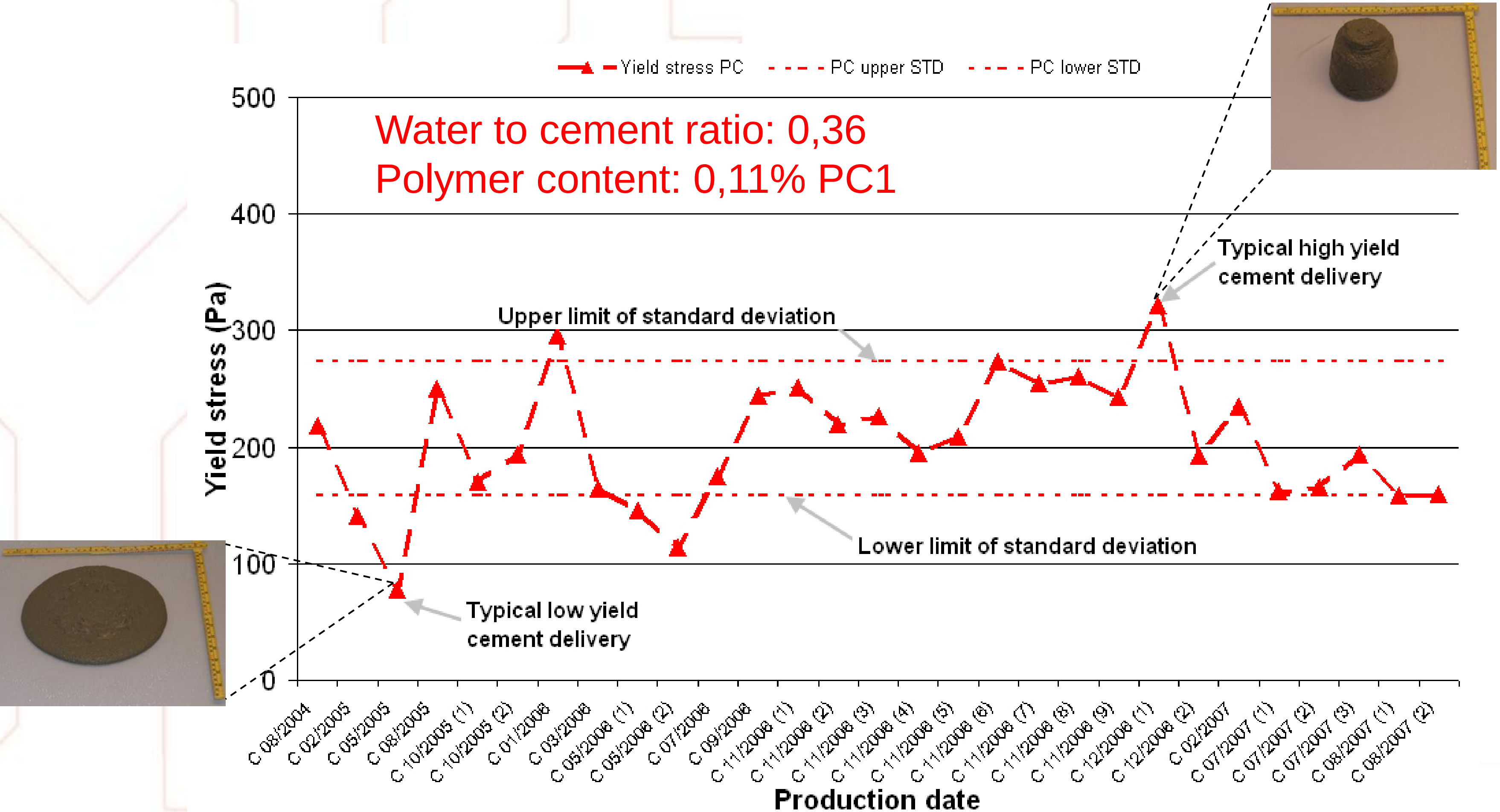
Experimental program

- Cement supplied by seven cement producers from six countries, located in Europe, the Middle East and Asia



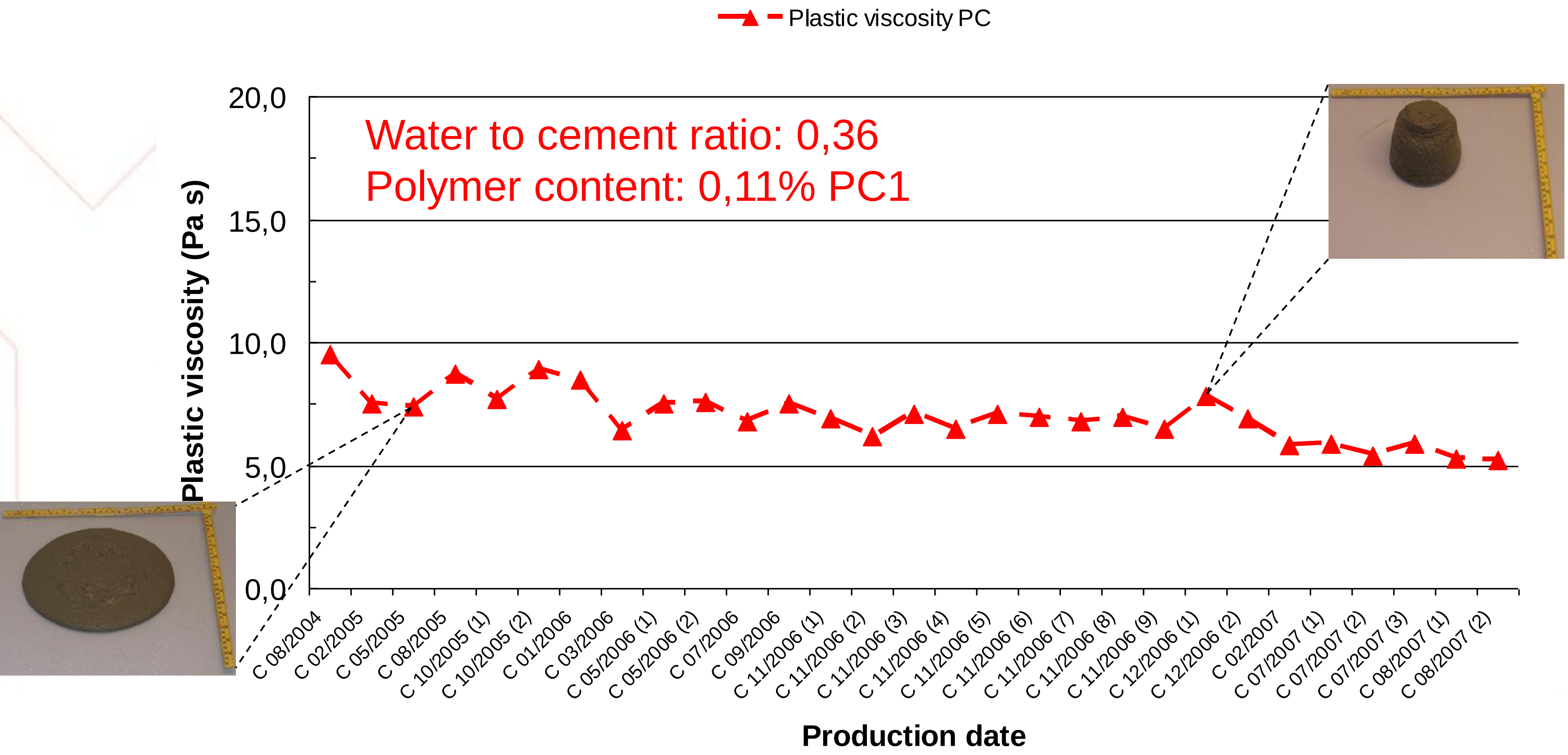


Results Yield stress of mortar



6 out of 29 deliveries showed a significant variation from the mean within the test series

Results Plastic viscosity of mortar



Only minor influence of cement delivery on plastic viscosity

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Results Correlation mortar versus concrete

- Production-related variations in SCC are around 10-12 cm in slump flow and ~50 Pa in yield value

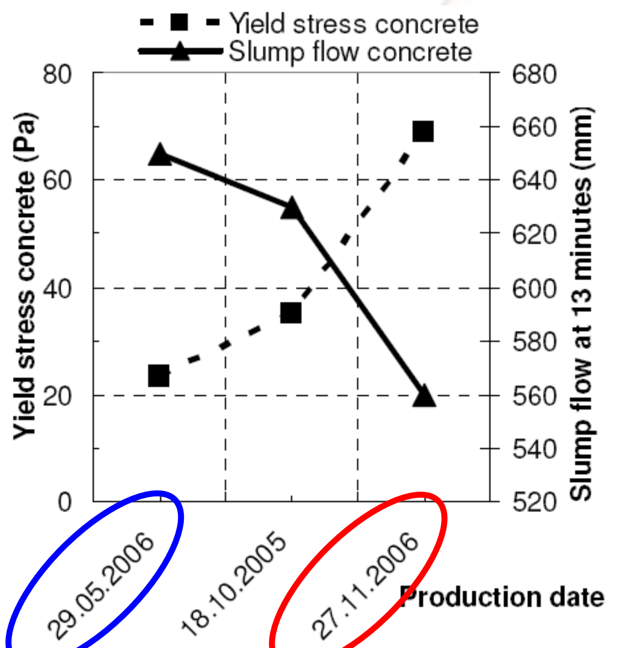


Figure 5: Effect of production date on yield stress and slump flow of concrete /Ku 2/

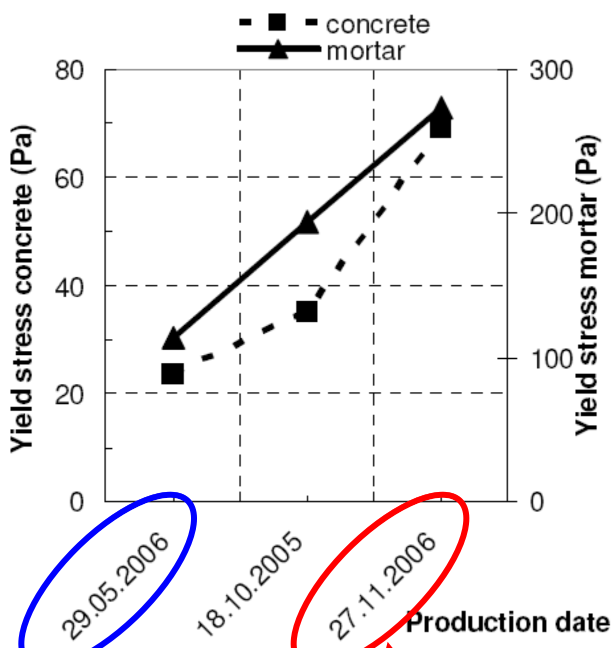


Figure 6: Effect of production date on rheology of concrete and mortar /Ku 2/

Low yield

High yield

Low yield

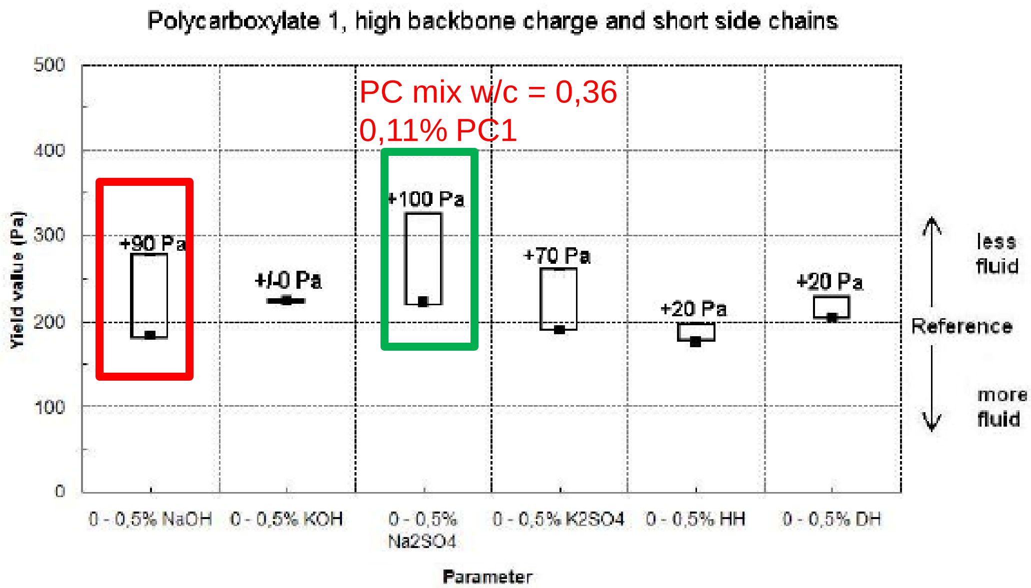
High yield

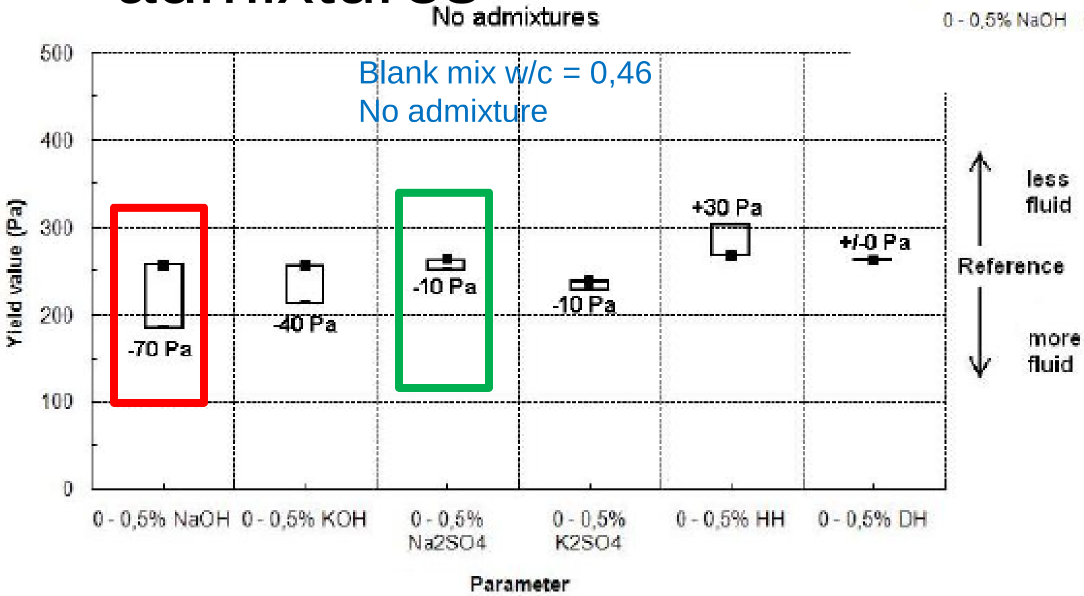
Results Correlation mortar versus concrete

- The devices used for rheological measurements showed a good ability to predict the rheological properties in concrete
- On the contrary, standard tests on cement (such as setting time and water demand) did not show any indication of cement-admixture interaction problem

Results constituents influencing rheology

- Constituents which influence blank mixes have only minor or sometimes opposite effect in mixes with admixtures





Results aluminate phase was stable in reference cement

- No influence of aluminate phase on yield value was observed. In the reference cement, the fluctuations in aluminate phase content were very low.

Cement production date	23.01.2008 (low yield)	15.04.2008 (2) (high yield)
Yield value in mortar with PC1 (Pa)	135	330
Plastic viscosity in mortar with PC1 (Pa·s)	7	8
C_3S (%)	58,6	58,6
C_2S (%)	19,8	19,4
C_3A (%)	4,1	4,0
$C_2(A,F)$ (%)	12,4	12,3
Calcite (%)	3,9	3,6
Hemihydrate (%)	1,3	1,6
Anhydrite (%)	---	0,4
Dihydrate (%)	---	---

---) below limit of detection

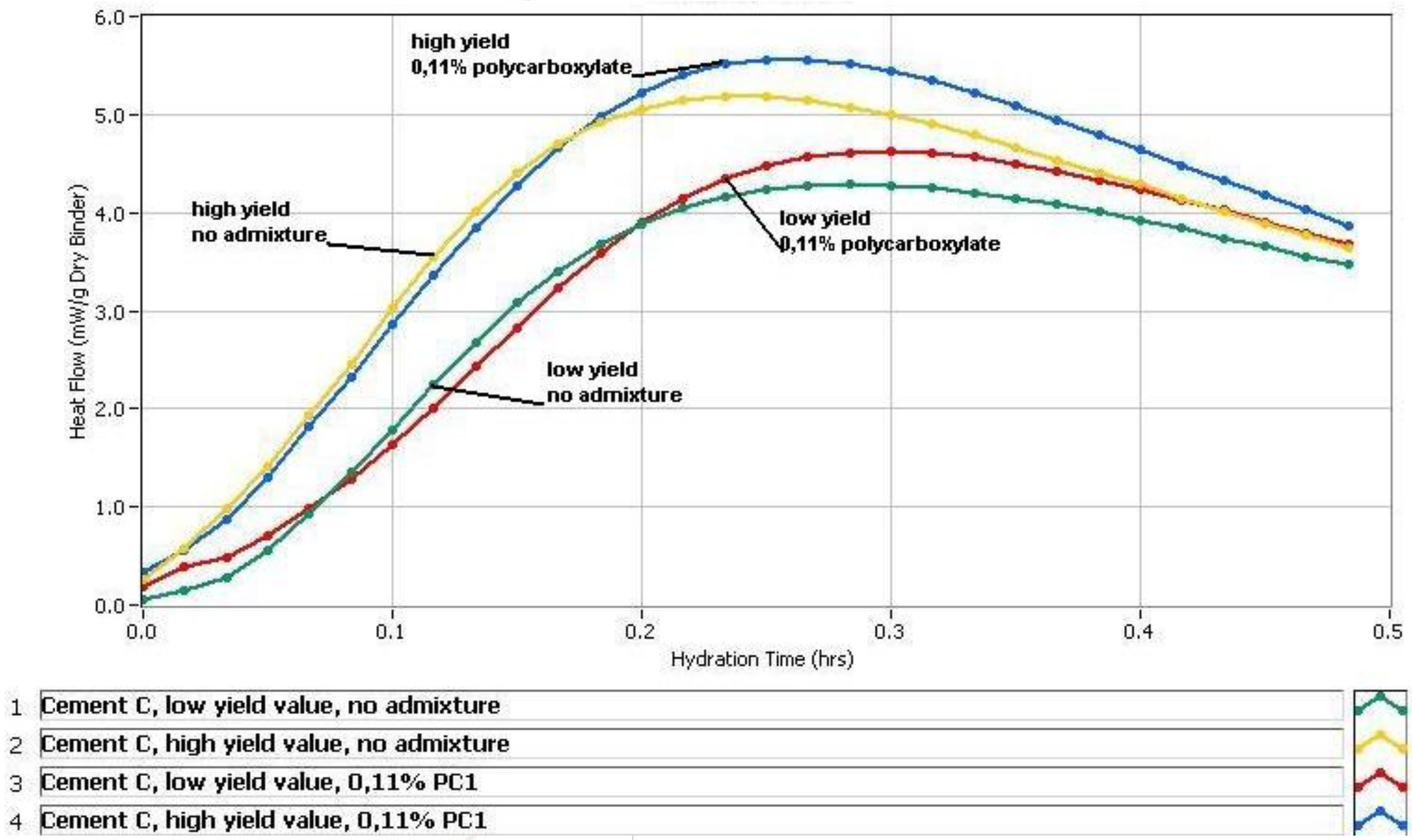
Similar composition, but lets look on the hydration curves...

Results initital hydration process (reactive aluminate phase)

- All ingredients were stored overnight in the calorimeter -> temperature equilibrium.

Blank mix w/c = 0,46
No admixture

PC mix w/c = 0,36
0,11% PC1



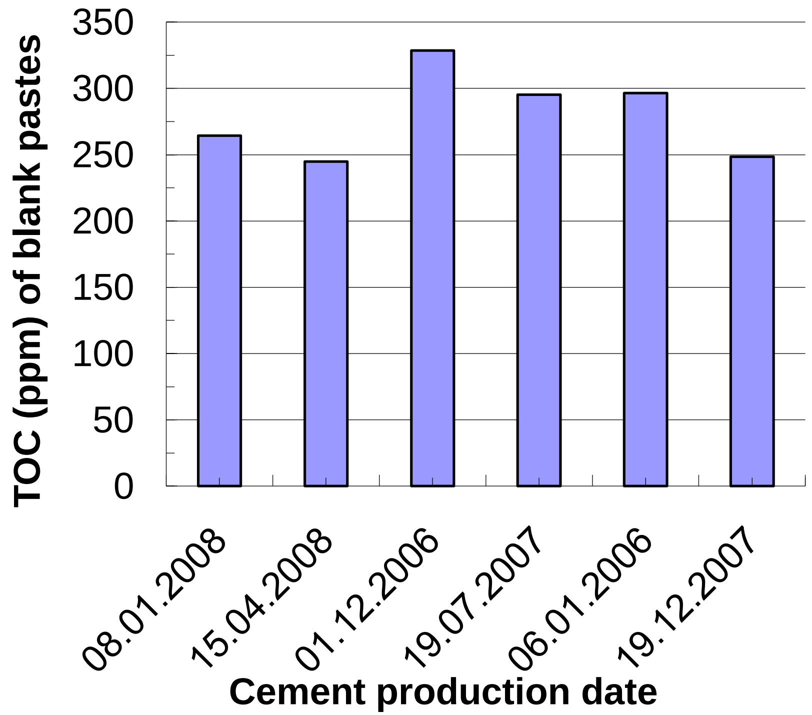
Results Adsorption measurements

- The total organic carbon (TOC) was measured to calculate the adsorption degree
  - Input: TOC of admixture, blank cement, water and cement plus admixture
  - Output: Available admixture in pore solution giving the polymer adsorption/consumption

Results TOC of blank cement

- The TOC of blank cement is differing with production date

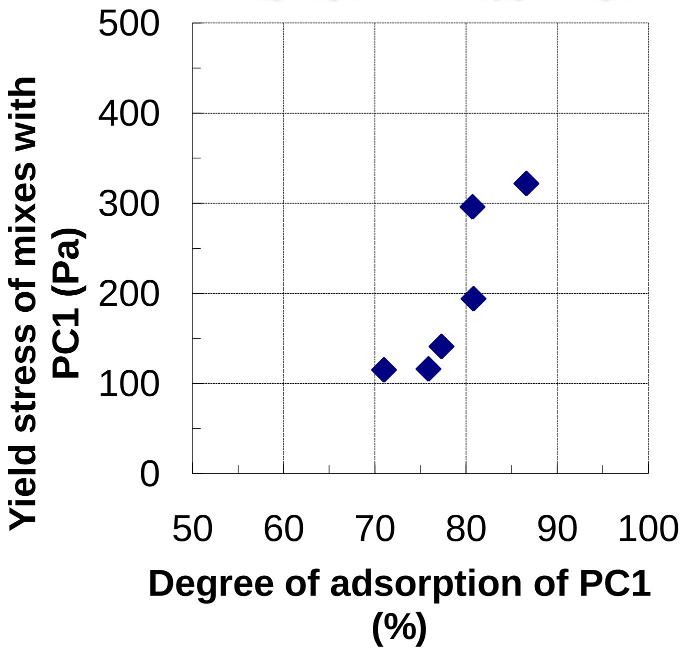
Blank mix w/c = 0,46
No admixture

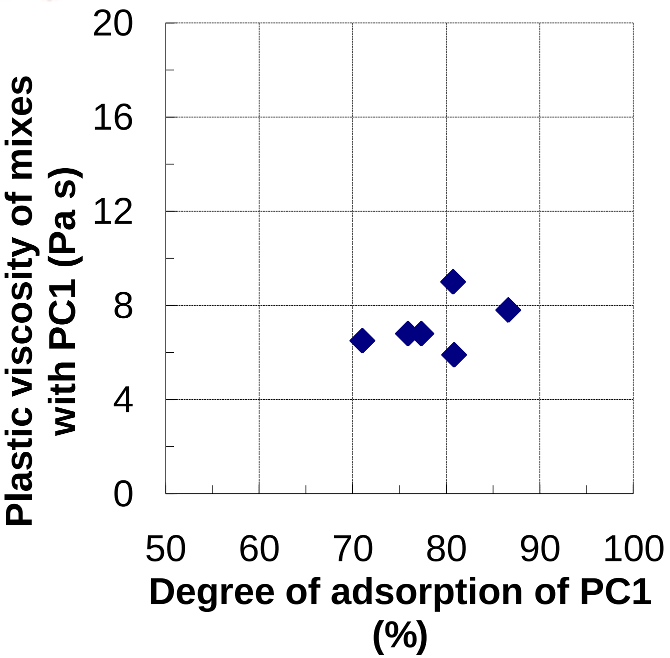


Results polymer adsorption on various cement

- Cement with a high yield value (sticky mix) adsorbed most polymers. Or in other words, cements with a high yield value consumed more polymers (intercalation?)

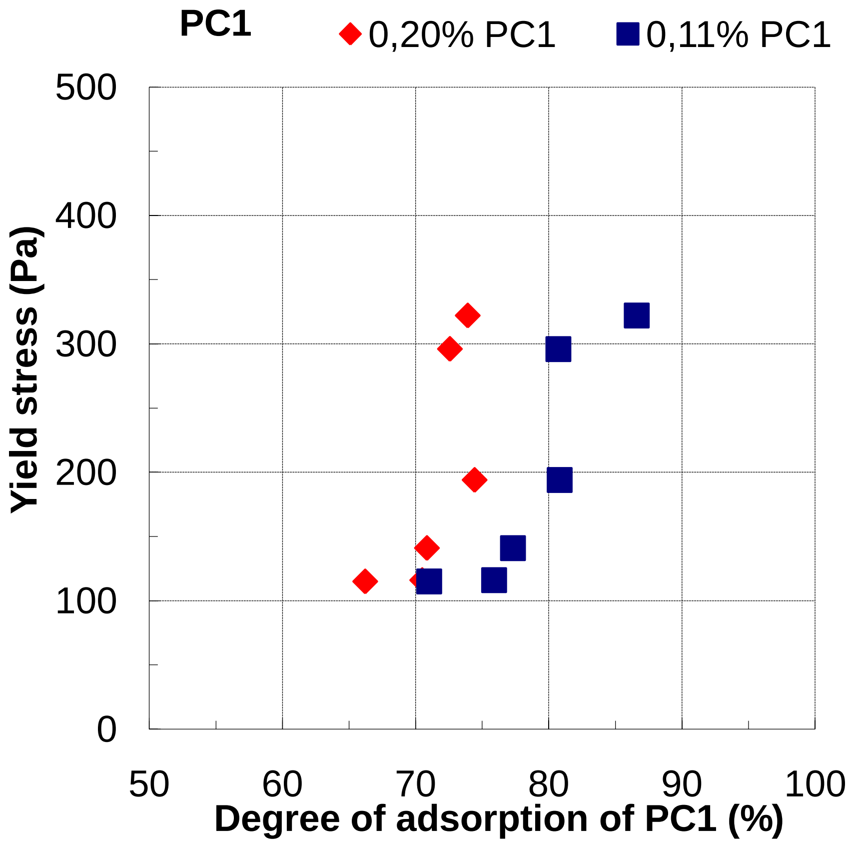
PC mix w/c = 0,36
0,11% PC1





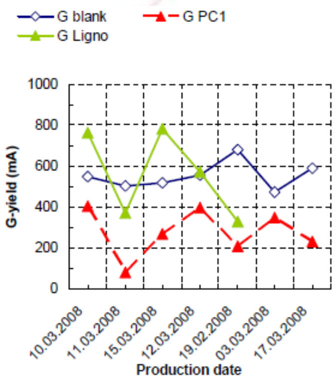
Results Adsorption measurements

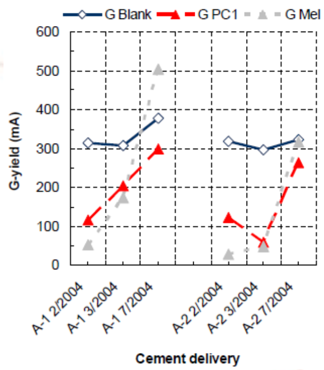
- Same trend at higher polycarboxylate dosage => shifting of adsorption degree to lower values

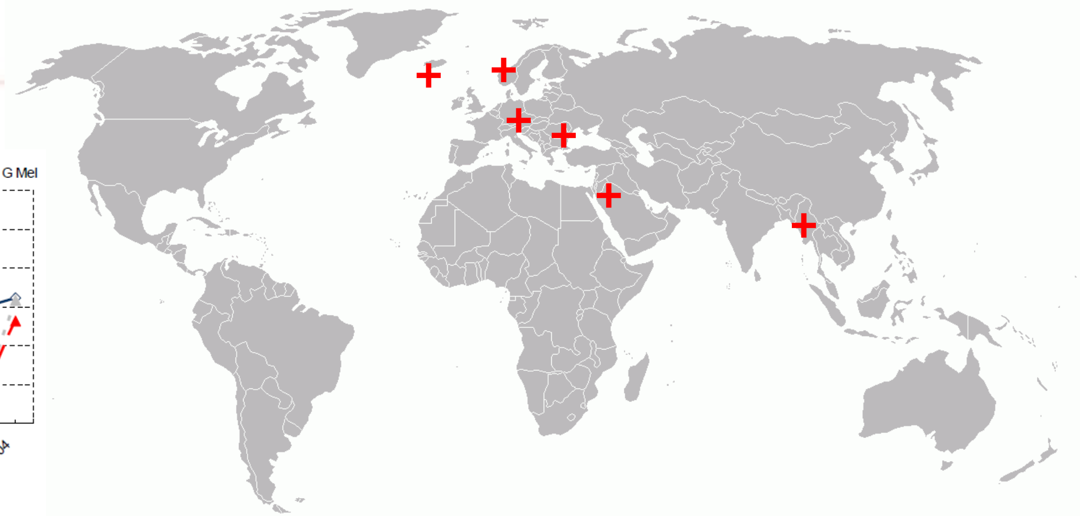


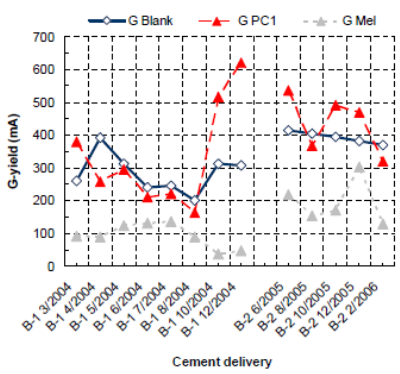
Production-related variation from 6 countries

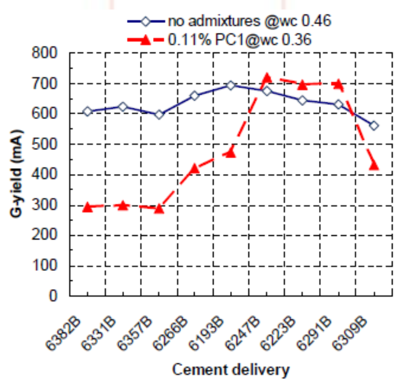
- The variations are independent from origin.

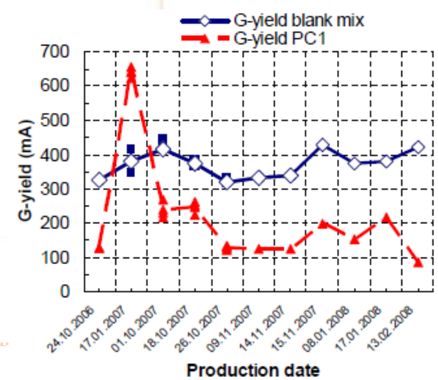












A schematic model to explain the variations

Yield stress

A property of the fluid at shear rate 0 (rest state).

It is hence mainly affected by attraction forces and mechanical entangelments.

Plastic Viscosity

A property of the fluid already in motion.

It is hence mainly determined by parameters like dynamic friction between the moving elements.

A schematic model to explain the variations

Yield stress

• Varying early hydration products influence the attraction forces and mechanical entangelments

• Adsorption behaviour of polymers on cement surface are most likely different from one delivery to another

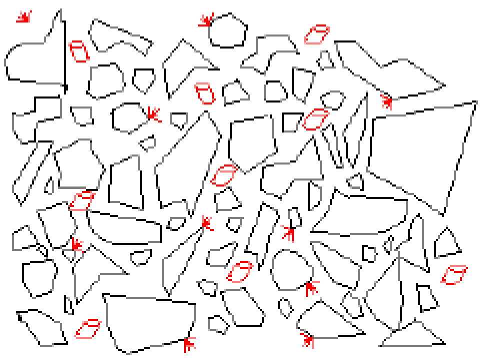
Plastic Viscosity

• However, the influence of primary hydration products and the lower adsorption degree on the dynamic friction seem to be minor

Two mechanisms to explain the variations

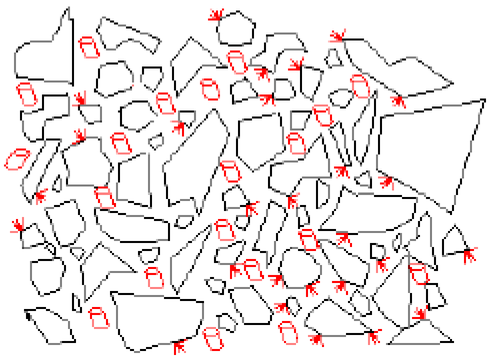
Polymer consumption due to excessive ettringite

Yield stress: 100 Pa
Plastic viscosity: 6 Pa s



low yield cement paste
w/c-ratio 0,36
0,11% polycarboxylate

Yield stress: 300 Pa
Plastic viscosity: 6 Pa s



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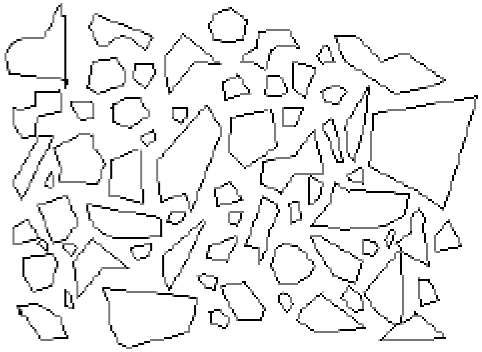

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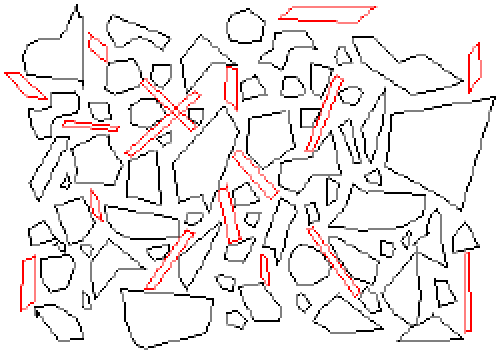

high yield cement paste
w/c-ratio 0,36
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Mechanical entanglements due to syngenite formation

Questions?