Stiftung Rheinland-Pfalz für Innovation

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mix quality / mixing optimization of refractory castables; a rheological view (ball measuring system and slump flow)

Mischqualität/Mischoptimierung feuerfester Betone aus rheologischer Sicht
(Kugelmesszelle und Ausbreitmaß)

22. Workshop und Kolloquium
Rheologische Messungen an Baustoffen
Regensburg, 06.+ 07.03.2013

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refractory castable
definition

- refractory castables are defined in DIN EN ISO 1927-1 by following components:
  - refractory aggregate
  - binder
  - liquid
  - ultrafines and dispersing agent (for deflocculated castables)

- practical appearance of a dense, hydraulic bonded refractory castable:
  - particle size distribution by Andreasen or Dinger and Funk
    → optimization for a reduced porosity and water demand
  - raw material selection by application
    → evaluation of chemical attack and working temperature
  - hydraulic bonding with CAC (CalciumAluminatCement)
    → minimization due to elevated corrosion
  - Dispersing agent for enhanced rheological properties
    → further reduction of water demand

initial situation for mixing and measuring rheological properties

How **are** refractory castables mixed?

intensive mixer, pan mixer, planetary mixer

→ various mixing devices, variable mixing time and tool speed

no systematic investigations at present

How **are** rheological properties of refractory castables determined?

slump flow (DIN EN ISO 1927-4)

for variable shear intensity only few investigations in experimental stage are available

→ **insufficient knowledge for mixing and rheological properties of refractory castables**

→ **large diversity in nature and grain size of raw materials**

→ **small but fine refractory industry compared to the construction industry**

1. Under which condition **should** refractory castables be mixed?
2. How **should** rheological properties of refractory castables be determined?
3. How are rheological properties influenced by an optimized mixing process?
4. Which material properties arise from the optimized mixing process?

general experimental setup

mixing technology: intensive mixer (Eirich, type R05)
(variation of rotation speed, rotation direction, mixing tool, time)

rheology: slump flow according to DIN EN ISO 1927-4
Schleibinger Viskomat NT with ball measuring system 20mm
profil 0-5rpm/5rpm/5-0rpm bei <360°/<15s

material: self-flowing castable

component	percentage	
T60 3-6mm	25,0	aggregate tabular alumina
T60 1-3mm	15,0	
T60 0,5-1mm	11,0	
T60 0,2-0,6mm	6,0	
T60 0-0,2mm	20,0	
E-SY1000	18,0	fines reactive alumina
Secar 712	5,0	calcium aluminate cement
Summe	100,0	
PCE	0,2	dispersing agent
Wasser	4,3	

rheological characterization of refractory castables at present

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- rheological characterization of self-flowing castables

  DIN EN ISO 1927-4: determination of consistency of castables

- determination of slump flow (linear spread in % respectively in mm)

- cone of 70 / 100 mm x 80 mm as mould is recommended

$$F_V = \left(\frac{\overline{d} - d_0}{d_0} \right) x 100\%$$

- procedure for self-flowing castables:

  pour wet mix in mould / wait 15s / lift mould / 120 s flowing / measuring slump flow two times orthogonally

- reference value for a well densification is slump flow >150% (>250 mm)

→ **the subjective consistency for a slump flow of 150% deviates significantly**

rheological characterization of refractory castables at present

advantages	disadvantages
simple procedure (especially for industrial quality control)	no defiened shear velocity
minor technical complexity	no distinction of rheological values only an overall-value
reproducible measuring values	comparable slump flow may show variable visual properties
low cost	

consequence

testing method for the determination of shear rate dependening rheological properties of refractory castables will help to get deeper insights in material and process properties with special regard to R&D

rheometer selection

- rotational viscometer in general are not suitable for coarse grained materials

- common solution
  - → refusal of physical models (p~~arallel-plate-model~~)
  - → common practise: **several stirrer designs** (e.g. Schleibinger)
  - → results are comparable within a distinct system (device type specific values)
  - → no definite physical values are obtained

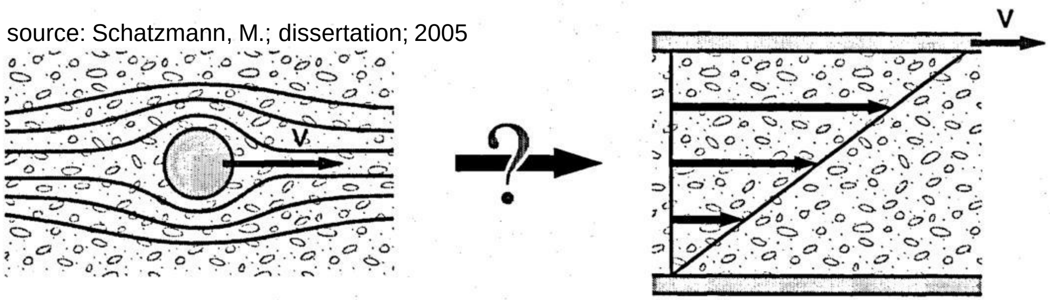
- alternative solution: e.g. ball measuring system
  - → flow profile around a ball is well known
  - → **Tyrach and Schatzmann** conclude:
    conversion of measuring values in rheological values is possible
    - → shear rate is small (self-flowing castables):
      linearity between
      moment and shear stress as well as
      rotation and shear rate

ball measuring system

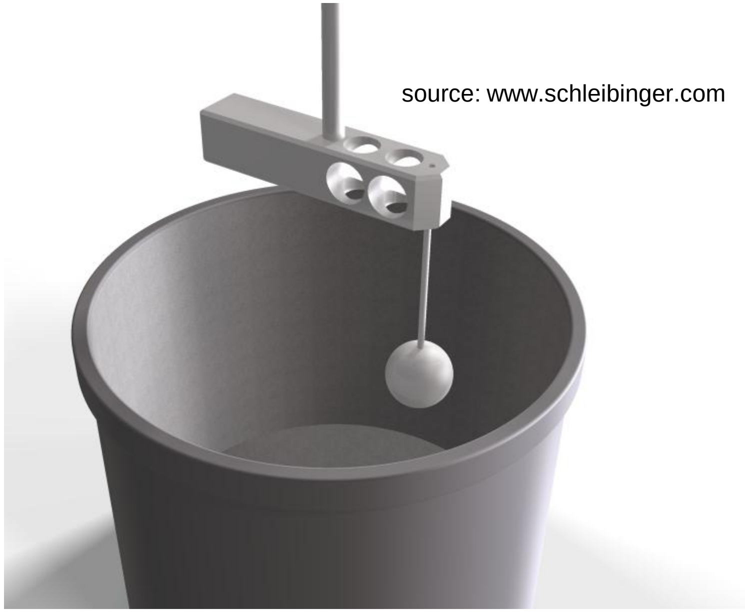
V relative viscosity
M torque
n rotation speed
η dynamic viscosity
τ shear stress
γ shear rate
K_γ linearity constant shear rate
K_τ linearity constant shear stress

measuring values → relative viscosity $V = \frac{M}{n}$

source: Schatzmann, M.; dissertation; 2005



source: www.schleibinger.com



converted values → dynamic viscosity $\eta = \frac{\tau}{\dot{\gamma}}$

- rotation speed n /rpm → shear rate γ /s^{-1}

$$\dot{\gamma} = K_{\dot{\gamma}} * n$$

- torque /Nmm → shear stress τ /Pa

$$\tau = K_\tau * M$$

experimental setup for rheological data acquisition

0	rpm	for	60 s	slow-down
0-5	rpm	in	6 s	accelerating
5	rpm	for	3 s	constant
5-0	rpm	in	6 s	decelerating

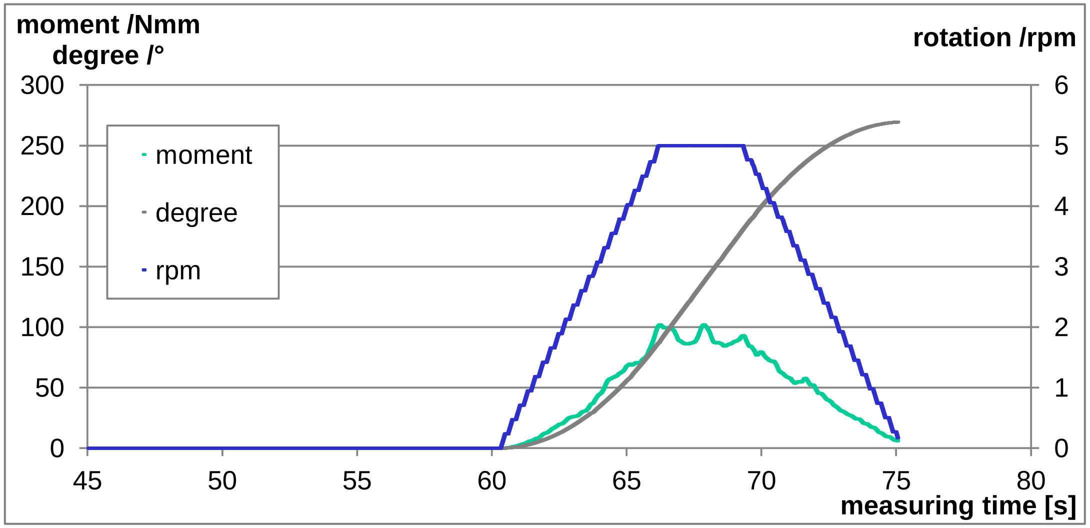
problem during acceleration:

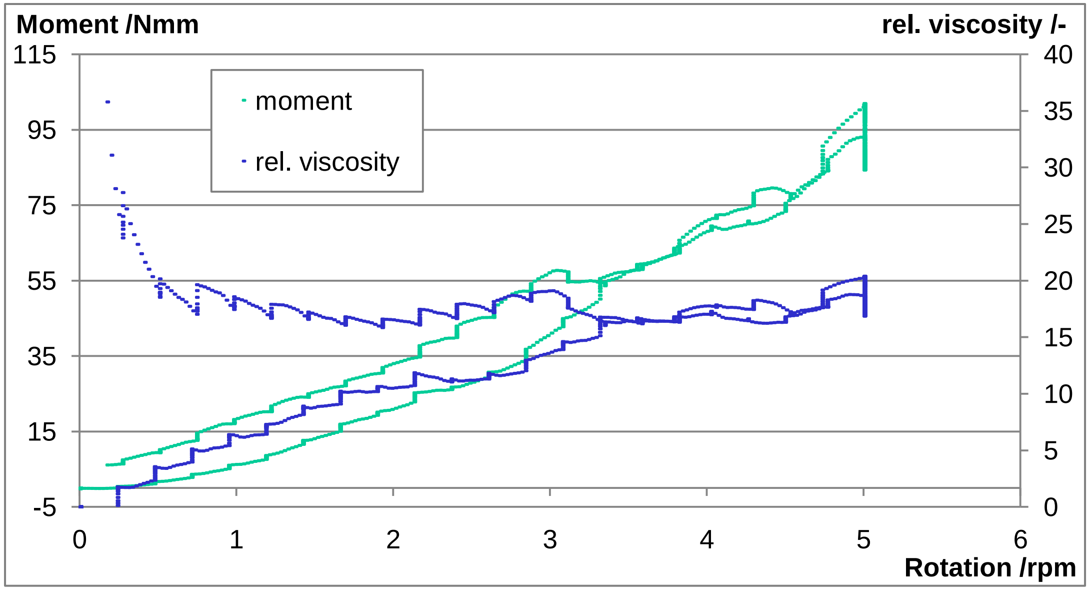
with proceeding measuring time fresh material mounts up in front of the ball retainer

solution:

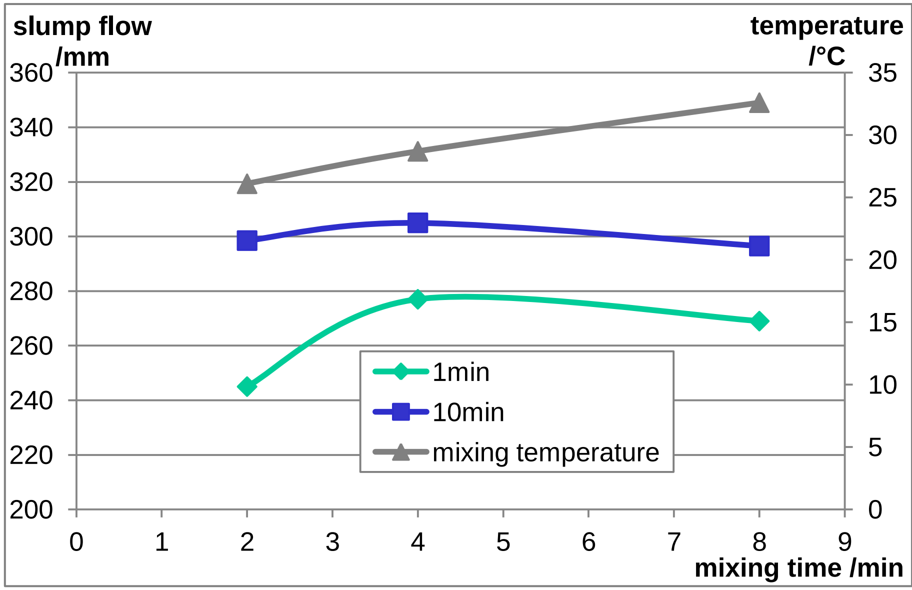
measuring during decelerating speed portion of mounting material is almost constant

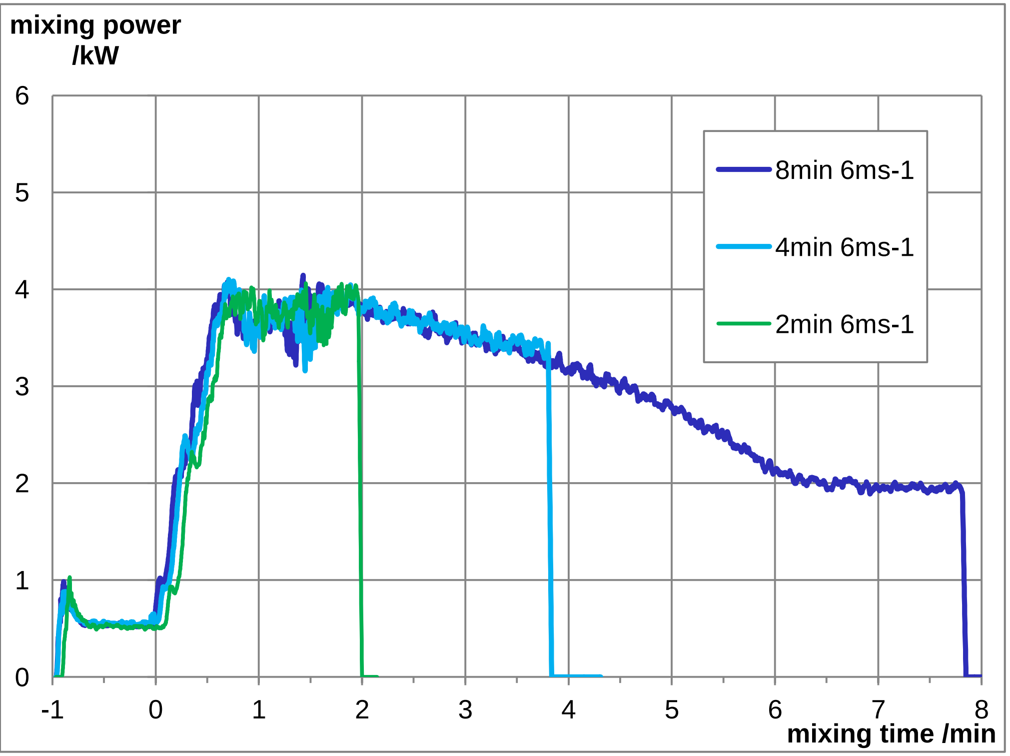
→ constant error

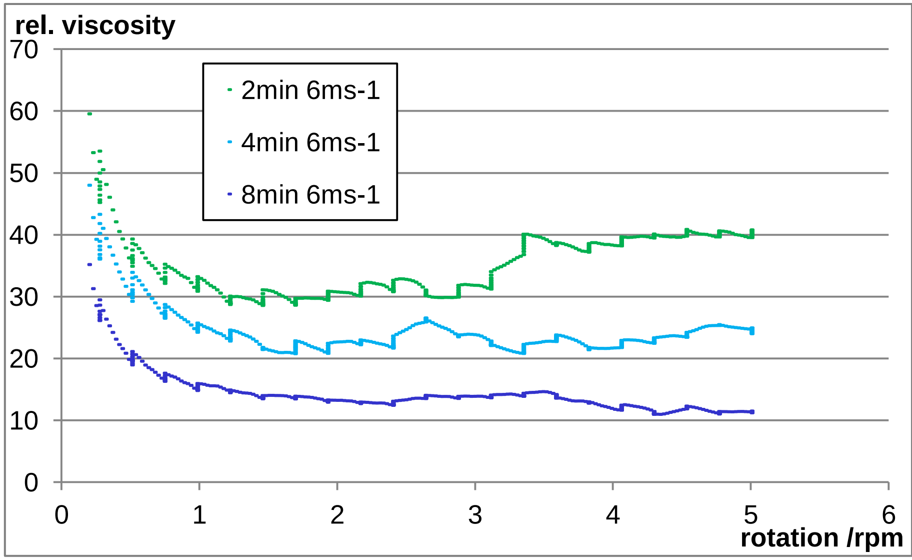




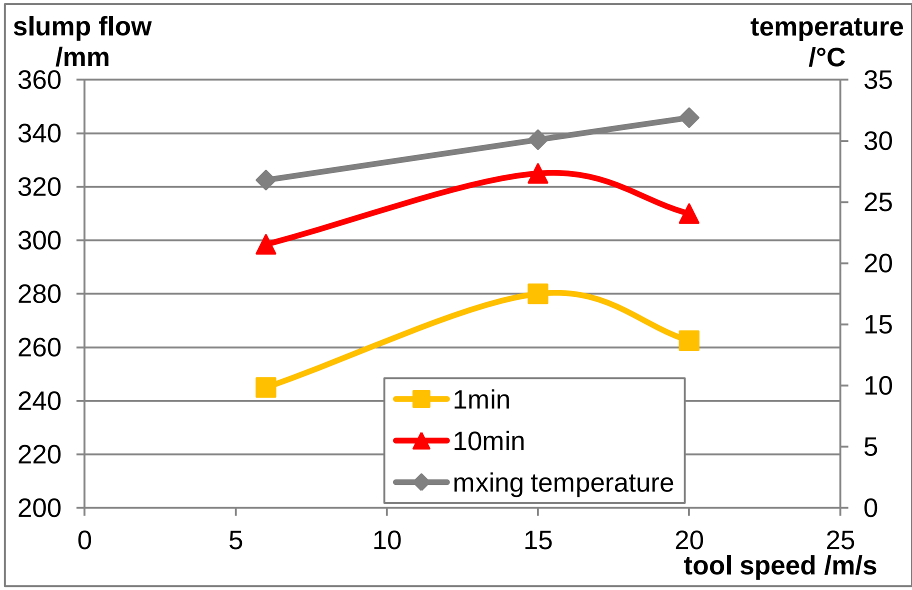
influence of mixing time

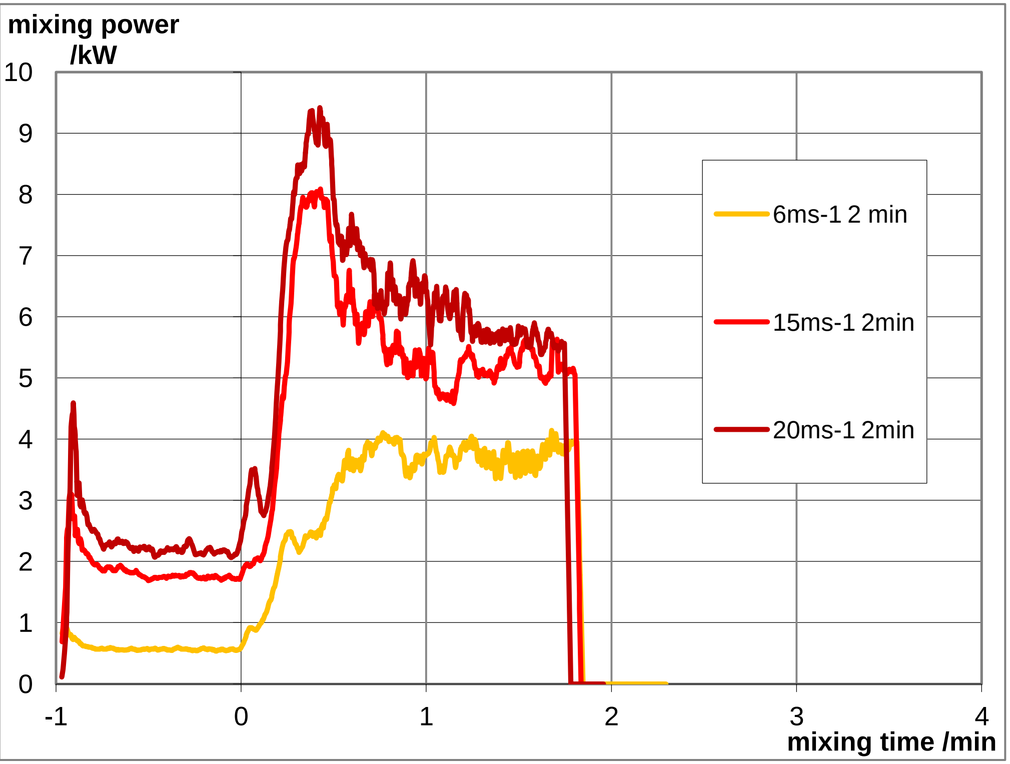


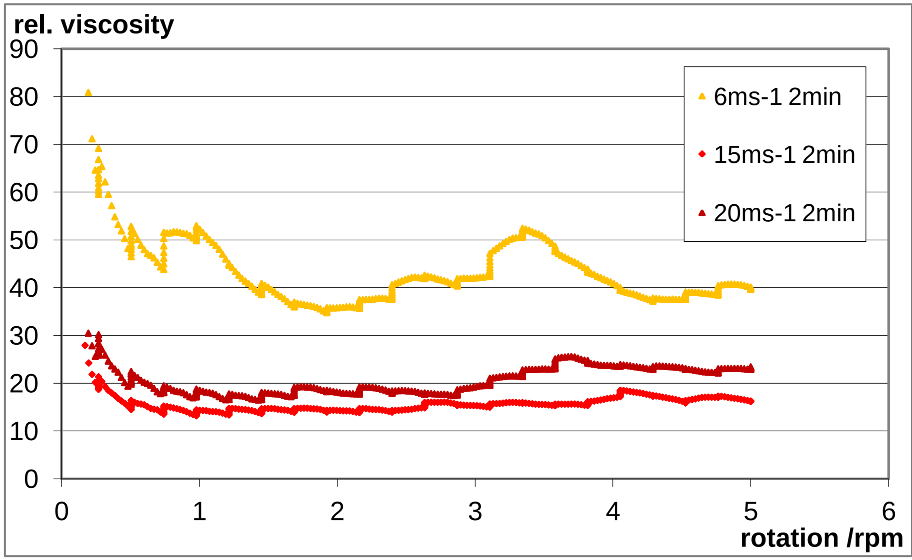




influence of mixing intensity







conclusion / outlook

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conclusions

- slump flow is only an overall value of all rheological properties
- slump flow is strongly influenced by surface drying due to high mixing temperature
- measurements with the ball measuring system are suitable for the rheological characterization of refractory castables
- the relative viscosity can be significantly reduced if mixing time is elongated and tool speed is elevated

outlook

- the conversion of relative viscosity to dynamic viscosity is still an outstanding issue (calibration measurements with bingham fluids are required)
- in the current research project further investigations are scheduled in which the dispersing agent and microfiller are systematically altered.

acknowledgement

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Thank you for your attention!