Effect of Particle Size Distribution of Metakaolin on Hydration
Kinetics of Tricalcium Silicate

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Introduction

- Metakaolin (MK) has been widely investigated and benefit the bulk properties and environmental costs of mortars and concretes due to MK’s filler and pozzolanic effects. [1, 2]
- **Filler effect**: increases formation C-S-H gel promoting heterogeneous nucleation.
- **Pozzolanic effect**: SCM reacts with Ca(OH)$_2$ (i.e. CH) to form C-S-H gel.
- **Motivation**: How does differences MK particle size distributions affect these mechanisms and C-S-H hydration.

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Material Preparation

- 99+% pure C-S was synthesized to a 3:1 molar ratio of CaCO$_3$ and SiO$_2$ at 1500°C.
- Commercially available MK (Imerys Metastar HP501 — 99.1% pure) were separated into three (3) particle size distributions (PSDs) via wet sieving process per ASTM C325-07 standard.
- The median particle size ($d_{50}$) was measured using a static light scattering analyzer (Microtrac S3500); the specific surface area (SSA) was calculating from the $d_{50}$.
- MK does not rehydrate during the wet sieving confirmed via thermal analysis and X-ray diffraction.

Experimental Procedure

- Hydration kinetics of [C$_3$S + MK] pastes were prepared with 1/s 0.45 for 72 hours in the TAM IV calorimeter at 20°C ± 0.1°C.
- Similar dissolution experiments were conducted with [CH + MK] pastes at a 1 : 1 molar ratio.
- Pastes were kept in isopropanol to arrest hydration before conducting thermogravimetric analysis.
- A modified phase boundary nucleation and growth model (pBNG) was implemented to extract product nuclei, allowing to evaluate effectiveness of MK.

Experimental Results

**C$_3$S + MK Calorimetry—Effect of MK Size**

A. and B. Regardless of the MK PSD, the both 8% and 30% MK additions broaden the heat flow curves and delays the hydration peak.
- The induction period duration increases with increasing MK dosage where the intermediate MK delays the peak the most.
- C. and D. The addition of MK increases the cumulative heat release more so than the C-S paste, approaching $= 300 \text{ J per g of C-S}.$

Experimental Results: C$_3$S + MK Calorimetry—Effect of MK Size

Extracted Calorimetry Parameters

E. and F. Low replacement level fine and coarse MK improves the slope acceleration and maximum heat flow rate indicating the filler effect. Larger additions of metakaolin reverse these benefits due to excess aluminate [Al(OH)$_4$]$^-$ ions. [4] Intermediate MK is more complicated and is explained below see below.

Simulation Results

I. and J. The addition of [C$_3$S + MK] reduces the amount of CH below the dilution line (shown as grey area) during early ages suggesting that MK is delaying the hydration and is not rapidly reacting with CH.
- However, the CH content in [C$_3$S + MK] pastes is much lower than 1 : 1 [MK + CH] paste and at later ages pozzolanic reactivity is expected to increase.

Conclusions

- Unlike fillers such as limestone and quartz, the correlation between the specific surface area (SSA) on hydration of C-S is nonlinear.
- MK is able to facilitate heterogeneous nucleation and growth of C-S-H, enhancing nucleation density (i.e. the amount of C-S-H) by filler effect.
- MK reacts significantly with CH but kinetics are slow in paste conditions.
- But the magnitude of these benefits is dependent on PSD and dosage level (MK<10%).
- Low replacement of coarse (i.e. 10µm-100µm) MK due to slight filler effects while being least susceptible to filler-effect and particle agglomeration.

References

1. Lapeyre J, Ma H, Kumar A. Effect of particle size distribution of metakaolin on hydration kinetics of tricalcium silicate. J Am Ceram Soc. 2019 (This poster is based on this work)

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