P10: Cryoporometry characterization for cement based materials

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A native of China, Min Wu got his MS in Material Science at the Tongji University, China in 2008. He started to work at DTU April 1st 2011.

Project description

The freezing point of water or melting point of ice confined in pores is lower than that of bulk water or ice. The magnitude depending on the size of the pore where the freezing/melting takes place can be quantitatively defined through thermodynamic considerations together with important assumptions. By using the properties of the confined water/ice in the freezing and melting process of a saturated sample, the ice volume which is an indication of that of the pores can be calculated and the pore size distribution can be derived accordingly. This is the principal concept of cryoporometry. The major disadvantage shared by the conventional porosity characterization methods, i.e., the drying of the samples, can be avoided if cryoporometry is adopted. Low temperature (micro-) calorimetry (LTC), which is also called (DSC) thermoporometry, and nuclear magnetic resonance (NMR) cryoporometry are two commonly adopted methods for cryoporometry characterization.

Both LTC and NMRC are adopted in the project. The LTC and NMR cryoporometry are complemented with direct measurements on moisture fixation. The moisture fixation method includes sorption analysis at hygroscopic range (which will be measured by a dynamic water vapor sorption instrument) and suction analysis at over-hygroscopic range (pressure plate extractors will be used).

The project focuses on learning the existing measurement techniques and analysis methods and trying to refine these procedures. Attempts will be made to couple the measurement techniques of LTC, NMRC and moisture fixation method with the aim of gaining more accurate information about the C-S-H porosity.

Project results

Low temperature calorimetry (LTC)

The LTC results include the discussions about data analysis and experimental procedures. Some experimental results are also presented.

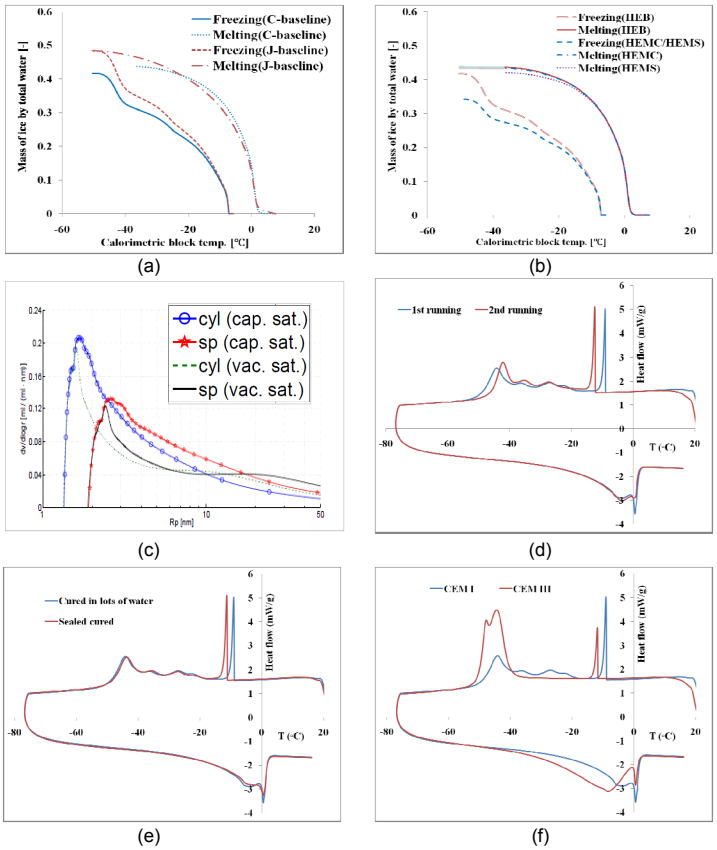

Figure 1. Factors influencing the calculated ice content: (a) baseline calculation and (b) the heat of fusion of the confined water; (c) sample saturation on the detected pore size distribution curves; (d) examination of possible frost damage of a cement paste during freezing; (e) preliminary study of the ions in pore solution on the freezing behavior; (f) comparison of the heat flow curves of two cement pastes (CEM I and CEM III of w/c=0.4).

Moisture fixation

The results of moisture fixation cover the measured sorption isotherms and the analyzed surface area and pore size distribution.

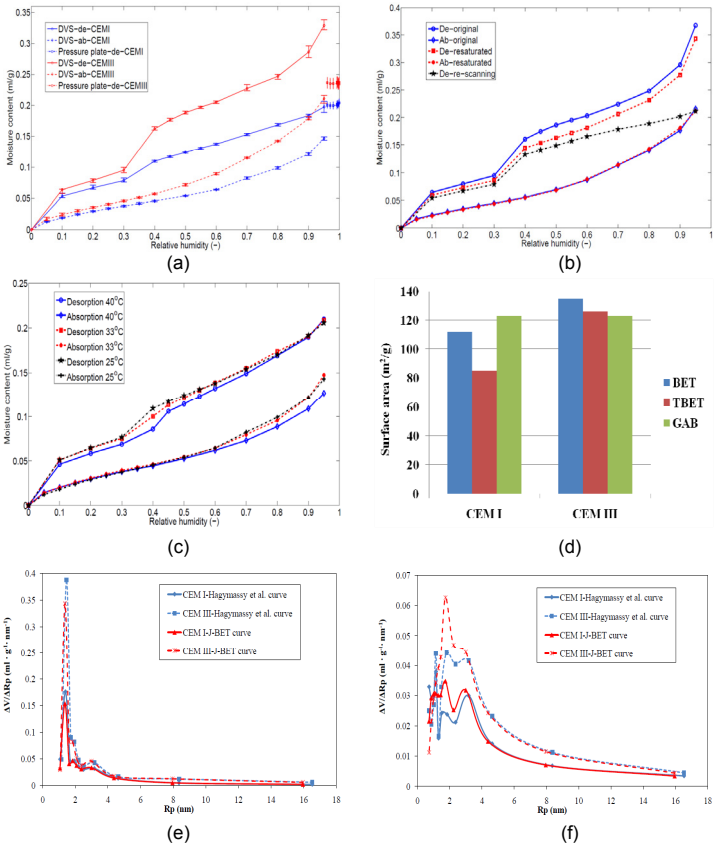

Figure 2. (a) sorption isotherms of cement pastes CEM I and CEM III (w/c=0.4); (b) sorption isotherms of resaturation study of paste CEM III; (c) temperature dependent isotherms of paste CEM I; (d) calculated surface area based on different adsorption equations; (e) pore size distribution derived from the desorption isotherms and (f) from the absorption isotherms using different 't-curves' as the thickness of the adsorbed layer.

What I am planning to do for the remaining time

- ❑ Complete low temperature calorimetry (LTC) measurements;
- ❑ Start and complete NMR cryoporometry measurements;

Figure 3. A NMR instrument (Yermakou 2012).

- ❑ Explore the ions in concrete pore solution and the freezing behavior;
- ❑ Data analysis and comparison study of the results by different methods.

Figure 4. Pore solution (Johannesson 2008).

Outstanding questions

- ❖ Data analysis and measurement protocol establishment of LTC;
- ❖ Pore solution and its freezing behavior;
- ❖ The connection between the pore connectivity and the hysteresis observed on sorption isotherms and the freezing-melting curves;
- ❖ Comparability between the results of the three methods.

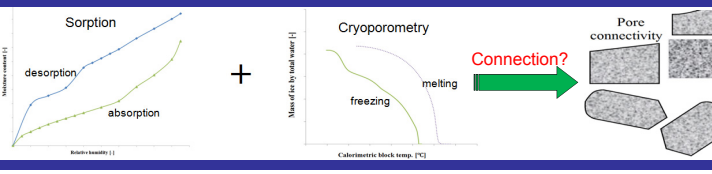

Figure 5. Characterize pore connectivity from hysteresis?

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