P10: Cryoporometry characterization for cement based materials

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MARIE CURIE ACTIONS

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.

Background

Porosity is one of the most important characteristics of cement based materials. An accurate characterization of the porosity is a necessity for a better understanding of the performance of cement based materials.

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, which is schematically shown in Figure 1. 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.

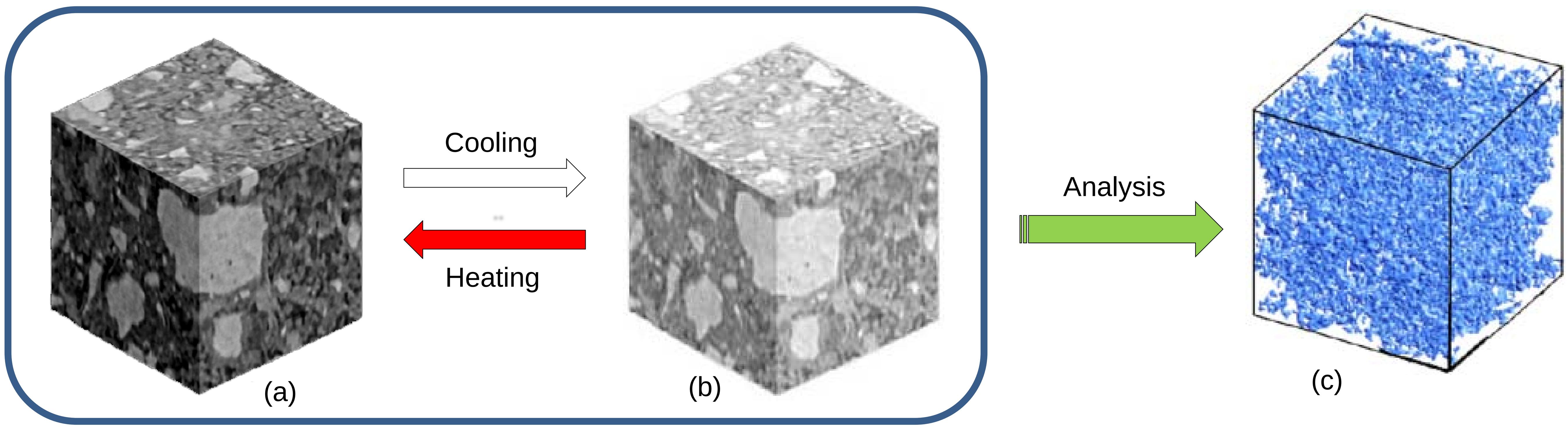

Figure 1. Schematic illustration of the principle of cryoporometry: (a) a water saturated sample of a porous material; (b) all the freezable water in the sample has been frozen; (c) by studying and analyzing the freezing process (from (a) to (b)) and/or the melting process (from (b) to (a)), the porosity information can be derived (pictures based on [1]).

Low temperature calorimetry (LTC)

The basic concept of LTC is that the freezing process of water is an exothermic process and the melting process of ice is endothermic. By studying the heat behavior during the freezing and the melting process of a sample, the porosity information can be derived.

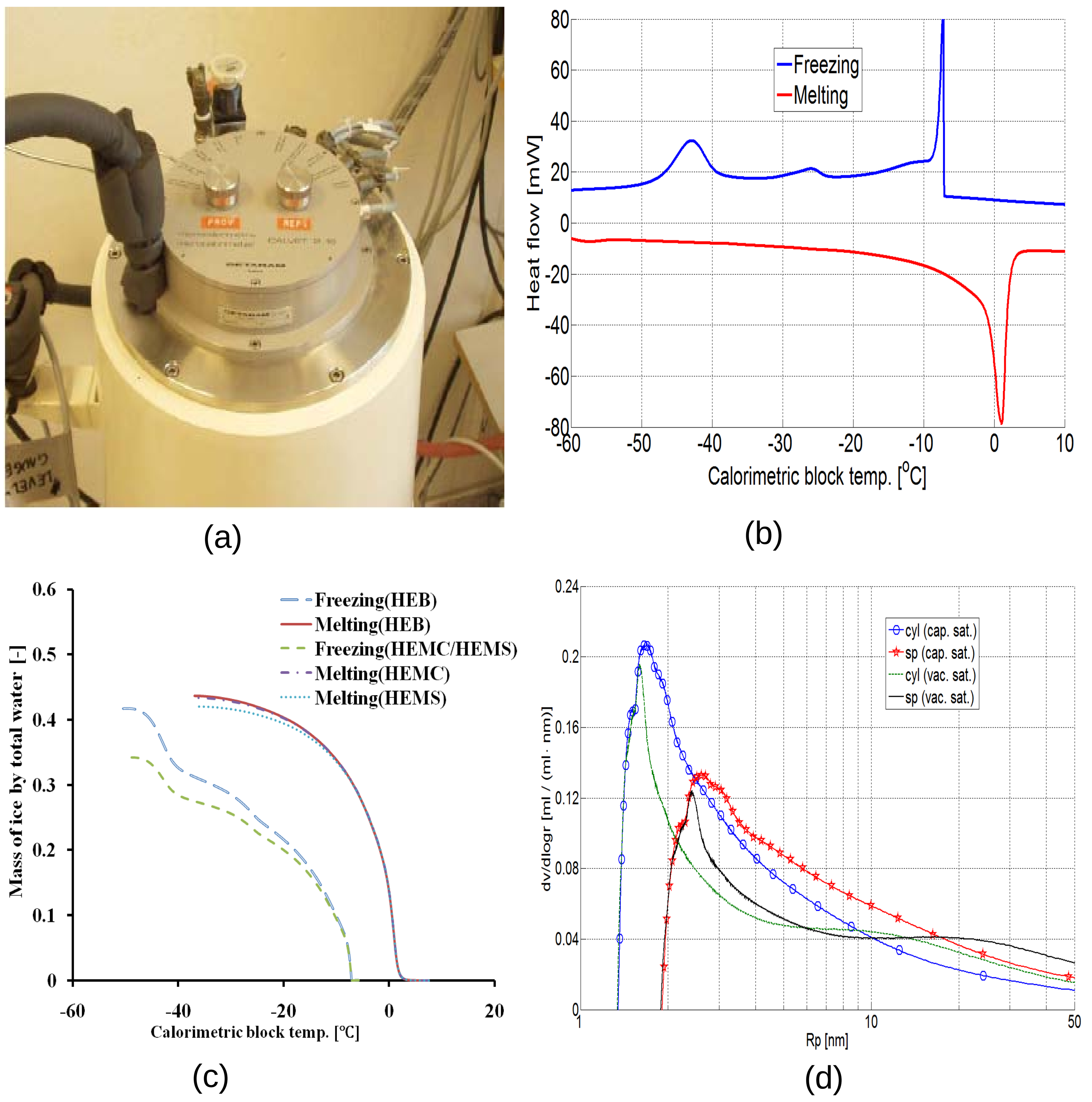

Figure 2. (a) an instrument for LTC measurement; (b) the measured heat flow during the freezing and the melting process of a cement-based material; (c) the influence of baseline calculation and the heat of fusion of the confined water on the calculated ice content [2]; (d) the impact of sample saturation on the detected pore size distribution curves of a cement-based material derived from the measured LTC melting data [3].

NMR cryoporometry (collaboration)

This method is based on the fact that the spin-spin relaxation time of ice is shorter than that of water and the relaxation time of the water is sensitive to its confinement in pores. By studying the signal intensity during the freezing/ melting process, the pore size distribution can also be calculated.

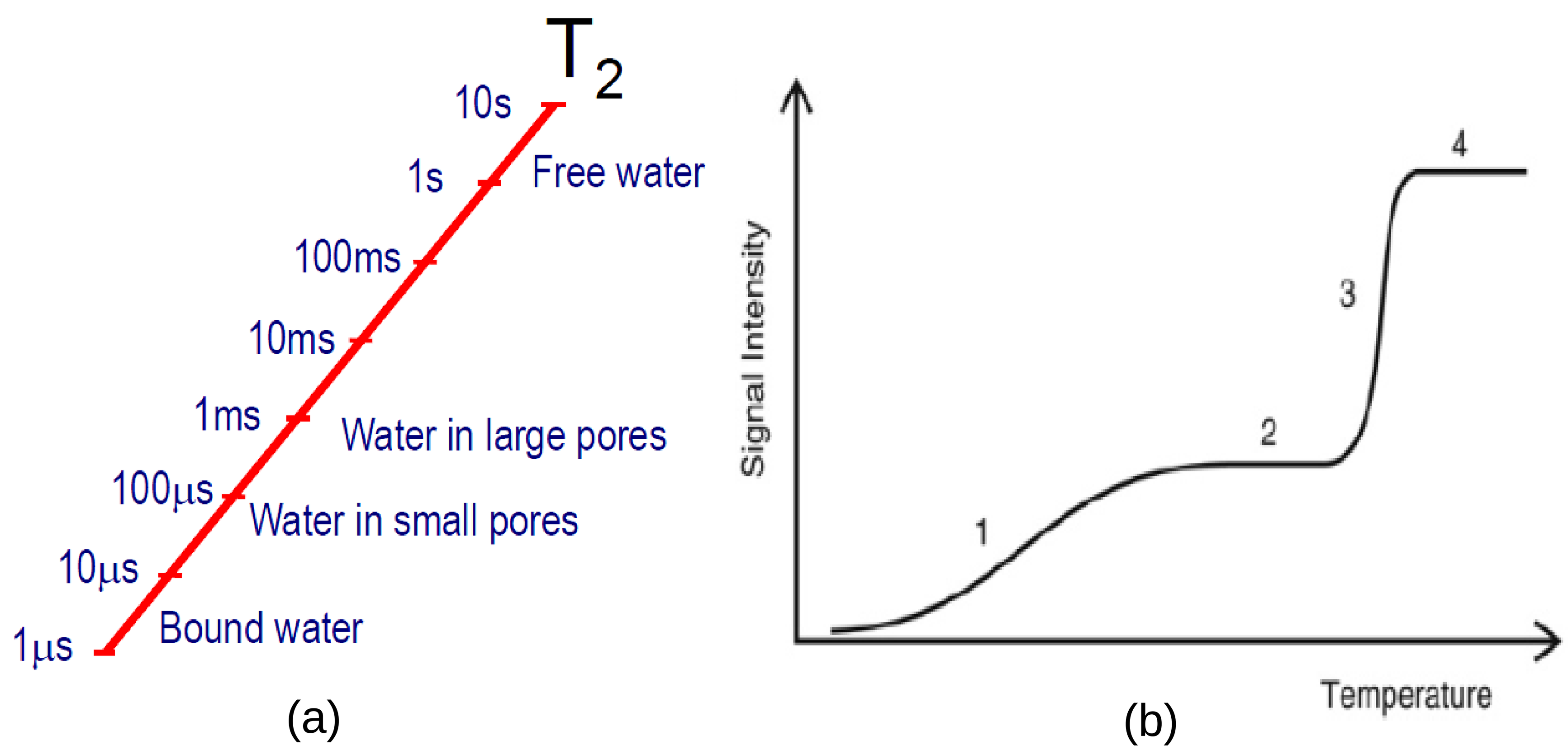

Figure 3. (a) T2 is the NMR spin-spin relaxation time, which is very sensitive to proton motion and confinement [4]. (b) An ideal NMR cryoporometry melting curve with four main features: (1) pore melting step; (2) total pore volume plateau; (3) bulk melting step; (4) total liquid volume plateau [5].

Moisture fixation

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

References

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