



# Influence of limestone and slag on the pore structure of cement paste based on mercury intrusion porosimetry and water vapour sorption measurements



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## HIGHLIGHTS

- DWVS is sensitive to changes in microstructure resulting from GGBFS and PLC inclusions.
- Microstructure characterization using both DWVS and MIP provides a description of pores diameters less than 1 nm to 8  $\mu\text{m}$ .
- DWVS and MIP yield complementary findings for 100% GU and 50% GU + 50% GGBFS cement pastes.

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## ABSTRACT

The objective of this study is to evaluate the responsiveness of mercury intrusion porosimetry (MIP) and dynamic water vapour sorption (DWVS) experiments to reflect changes to the pore structure of cement paste due to the influence of cementing material type. The DWVS isotherms are used to evaluate the pore size distribution and specific surface area. The MIP test is used to characterize the threshold diameter, and total porosity of the specimens. This study examines the influence of four cement paste mixture designs, namely 100% general use (GU) Portland cement, 50% GU + 50% ground granulated blast furnace slag (GGBFS), 100% Portland limestone cement (PLC), and 50% PLC + 50% GGBFS. The specimens were aged for one year in saturated limewater. The results indicate that DWVS is sensitive to changes in microstructure resulting from GGBFS and PLC inclusions. The Barrett Joyner Halenda (BJH) pore size distributions indicate that the effect of GGBFS and PLC inclusions have a marked effect on pores smaller than 4 nm diameter and a slight effect on pores between 4 and 10 nm in diameter. Characterization of the paste microstructure using both DWVS and MIP techniques provide a description of pores ranging from less than 1 nm in diameter to up to 8  $\mu\text{m}$ . Both methods are well suited to detect changes due to moisture-binder and the cementing material type (GGBFS and PLC).

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## 1. Introduction

Durable concrete needs to resist damage due to coupled and complex physical, chemical and electro-chemical processes such as sulphate attack, freezing and thawing cycles, alkali-silica reactions, and corrosion. These mechanisms are largely controlled by the concrete's resistance to ingress of moisture in the liquid or vapour state and aggressive ions through diffusion, permeation or absorption acting alone or in combination. As a result, characterization of the microstructure of hydrated cement paste remains an area of research that is warranted and continues to advance. Not only is accurate characterization of the microstructure of cement-based materials complex because the range of solid and

pores in hydrated cement paste spans over seven orders of magnitude, but also because moisture and ions, such as chlorides, sulphates or magnesium, interact physically and/or chemically when in contact with hydrated or unhydrated cementitious phases. For several years, research has reported alterations to the microstructure of concrete when exposed to moisture and ions [1,2]. The effect of physical and chemical interaction of free chlorides with the cementing material on reducing the diffusivity and sorptivity of concrete is established [3–6]. Accurate characterization of the pore structure of cement-based materials which accounts for moisture-binder and ion-binder interactions is an essential step towards the development of improved measures of transport and durability of concrete [7,8]. Inaccurate or incomplete descriptions of the microstructure can result in over or underestimation of the service life of concrete structures or infrastructure yielding potentially adverse economic and environmental implications [9–11].

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A challenge associated with characterizing the physical pore structure of cement-based materials is the understanding the sensitivity of test parameters to the effect of different binders and moisture–binder interactions over a range of pore sizes. The pore space of cement-based materials spans the millimetre to nanometre range, and comprises of capillary pores, gel pores, air voids, water voids, interfacial transition zone, and cracks attributed to thermal or relative humidity gradients [12]. Pores can be classified based on pore diameter in accordance with the International Union of Pure and Applied Chemistry (IUPAC) [13]. The pore diameter of micropores, macropores and mesopores are classified as pore widths less than 2 nm, between 2 nm and 50 nm, and larger than 50 nm, respectively.

In the past three to four decades, the study of concrete porosity and techniques to describe the pore structure has been extensively researched and reported on [14–19]. It still remains an important area of research owing to its direct correlation to concrete durability and sustainability. The two techniques of interest in this study are mercury intrusion porosimetry (MIP) and water vapour sorption experiments, both which have the potential to describe the pore size distribution and other pore characteristics of cement-based materials. Mercury intrusion porosimetry is a rapid technique that provides a pore size distribution ranging from nanometre to micrometre, as well as measures of total porosity and threshold diameter of cement-based materials [17,20]. Despite the well known limitations of the MIP technique, it is proven to capture differences in pore structure for cement-based materials containing supplementary cementing materials and the effect of chloride–binder interactions [15,19]. Gas sorption experiments measure the equilibrium mass uptake of gases by solid materials and can be used to determine the specific surface area and pore size distribution, which are important parameters for characterizing microstructure [13,21]. Sorption experiments on cement-based materials report that the majority of pores captured by water vapour sorption tests are in the gel pore (less than 3.6 nm in diameter) and small capillary pore range (3.6–37 nm in diameter) [13,14,22,23]. These fine pores are intimately connected to the properties and behaviour of the calcium–silicate–hydrate (C–S–H) gel that forms the bulk of hydrated cement paste and ultimately influences mechanical and transport properties [12,24,25].

DeBelie et al. [23] recently conducted a comparative study of the specific surface area of hydrated cement paste based on three methods, nitrogen sorption, water sorption using the saturated salt method, and water sorption using a dynamic vapour sorption balance. Consistent with findings from Mikhail et al. [21], the results revealed that Brunauer Emmett Teller (BET) specific surface areas based tests using nitrogen gas are markedly smaller than the specific surface areas based on water vapour sorption isotherms. Three primary explanations for the observations are: (i) the removal of bound water during sample preparation can allow water molecules to enter the space between the C–S–H layers which could result in erroneously high BET specific surface areas, (ii) the larger nitrogen molecules are not able to penetrate the entire pore space which results in a lower BET specific surface area in comparison to the smaller water vapour molecules, and (iii) the differences in the vaporization characteristics of nitrogen and water also contribute to the relatively lower nitrogen sorption BET specific surface areas [26]. The above mentioned factors justify the differences between nitrogen sorption and water vapour sorption specific surface area determinations. Similar BET specific surface areas are achieved for water vapour sorption using the saturated salt method and the dynamic water vapour sorption (DWVS) method with the latter yielding shorter test times and the ability to dynamically vary relative humidity [23]. The DWVS method allows for simultaneous examination of moisture uptake and porosity, and provides an assessment of material response through a range of environmental

conditions [27,28]. It should however be noted that there exist two ways to determine the WVVSs: the dynamic method and the equilibrium method (the “saturated salt solutions method”) which can provide different results since it is quite difficult to wait for equilibrium before a relative humidity shift.

The objective of this study is to evaluate the responsiveness of DWVS isotherms to changes in the pore structure of cement paste resulting from inclusion of granulated ground blast furnace slag (GGBFS) and Portland limestone (PLC) cement. Furthermore, this investigation evaluates the pore characteristics of cement paste determined using DWVS and MIP and investigates the relationship between the two test measurements. The DWVS isotherms are used to evaluate the pore size distribution and specific surface area. The MIP test is used to characterize the threshold diameter, and total porosity of the specimens.

## 2. Test methods

### 2.1. Dynamic water vapour sorption

In general, water vapour sorption isotherms are a measure of a material's equilibrium water mass over varying relative humidity at constant temperature. Cement-based materials characteristically exhibit Type II or IV isotherm form, illustrated in Fig. 1 [29,30]. At low relative humidity, adsorption occurs by diffusion. As the partial pressure of vapour increases, water molecules begin to deposit on the pore surfaces, first as monolayers, then in stacked multilayers [31]. The transition from monolayer to multilayer adsorption is indicated by point B in Fig. 1 for the Type II and Type IV isotherm form. At relative humidities greater than 60%, the multilayer water molecules begin to join across pores to form menisci, resulting in significant mass uptake through capillary condensation [32]. During adsorption, pores fill from small to large, and monolayer and multilayer adsorption resulting from diffusion of water molecules occurs without preference throughout the specimen. Thus, the description of pore sizes resulting from DWVS testing can be considered to be free of misleading network effects, in which the interconnectedness of pores affects how the pores are registered by the model.

As capillary condensation occurs and as the maximum relative humidity is achieved, in order for the adsorption–desorption loop to close, the equilibrium relative humidity must be very close to saturation. If full saturation cannot be achieved, the mass water content at 100% relative humidity has been shown to vary by 10% based on a comparative study of the saturated salt method and the DWVS approach where the former method achieves greater than 90% relative humidity.

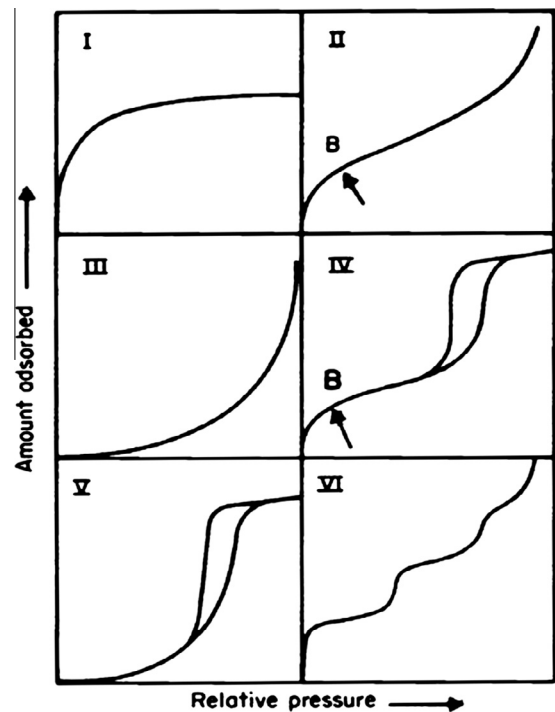


Fig. 1. Sorption isotherm classification [13].

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