XVI International Conference on Durability of Building Materials and Components
DBMC 2023, China
K.F. Li and D.P. Fang (Eds)

Microstructure of Cement Paste with Expansive Additives under Free and Restrained Conditions

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Abstract. *Expansive additives (EAs) have been widely used to reduce the risk of cracking due to shrinkage. Such additives cause early age expansion of concrete due to the formation of Portlandite and Ettringite in the pores of the cement paste, which affects the cement paste's microstructure. The presence of external restraints in the form of reinforcement and support affects the expansion and, ultimately the microstructure of cement paste. In the current study, the microstructure of cement paste with expansive additives is studied using mercury intrusion porosimetry (MIP). A free lime-based EA, with 10 and 15% (by weight) cement replacement ratios, is studied in the current study. The porosity and pore size distribution of the cement paste with EA were studied under free and restrained conditions. Results show that the under the stress-free condition, the porosity of the paste increased, while under the restrained condition, the porosity remained much similar to the reference case of OPC. Further, the pore size distribution of the cement paste with EA under free and restrained conditions is explained, and the effect of EA and restraint on the microstructure is discussed.*

Keywords: *Expansive Additives, Porosity, Free Expansion, External Restraint, Pore Size Distribution, Mercury Intrusion Porosimetry.*

1 Introduction

Concrete undergoes shrinkage when exposed to a lower humidity environment or due to autogenous shrinkage(Bissonnette et al., 1999; Neubauer et al., 1997). Cracks develop in concrete when the shrinkage stress exceeds the tensile strength of the concrete(Choi et al., 2015; Tongaroonsri & Tangtermsirikul, 2009; Zhu et al., 2020). This can lead to significant structural damage, compromising the integrity of the concrete structure. Different mitigation strategies such as super absorbent polymers (SAP), expansive cements, expansive additives (EAs), shrinkage-reducing admixtures etc., have been used to reduce shrinkage, among which EAs have been extensively used to reduce the risk of cracking and improve the service life of structures (Bentz et al., 2001; Fu et al., 1995; Maltese et al., 2005; Yang et al., 2019)**.**

EAs cause early age expansion due to the formation of expansive hydrates such as portlandite and ettringite, which compensate for the shrinkage (Nagataki & Gomi, 1998). Due to the formation of such additional species in the pore network and expansion, the microstructure of the cement paste is greatly influenced. As a result of which, the mechanical properties of the cement and concrete are also affected. The concrete structures are subjected to external restraints in the form of reinforcement or supports, which limits the expansion of concrete (Gupta et al., 2022; Semianiuk et al., 2017). Such external factors also affect the microstructure of concrete.

Thus, understanding the effect of EAs and external restraint on the microstructure of cement paste is essential for optimizing the performance of concrete in construction applications. In this context, the current study focuses on the microstructural changes that occur in cement paste with expansive additives under free and restrained conditions. A free lime-based EA with different replacement ratios was studied using Mercury intrusion porosimetry (MIP). The porosity and pore size distribution of the cement paste is evaluated under both free and restrained conditions, and the effect of external restraints on the microstructure of the cement paste is discussed.

2 Material and Methods

Ordinary Portland cement (OPC) and a free lime-based expansive additive (EA) were used as raw materials for the current study. The chemical compositions of the raw materials were calculated using XRF and are given in **Table 1**. The mineral compositions of OPC and EA are determined using the internal standard method, where 10% corundum (Al_2O_3) was mixed with the cement powder. Sample preparation for X-ray diffraction (XRD) was carried out using the back-loading method, and Rietveld analysis was performed using the TOPAS (Bruker) software (listed in **Table 2**)(Scrivener et al., 2016). The fineness and specific gravity of the cement were 3290 cm^2/gm and 3.16, respectively, whereas the specific gravity of the EA was 3.10.

Table 1. Chemical composition of the raw materials (OPC and EA).

Materials	Chemical composition (mass %)										Total
	LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	TiO_2	P_2O_5	SO_3	
OPC	3.03	20.9	6.96	2.43	64.43	1.29	0.49	0.26	0.18	2.84	99.79
EA	2.29	1.56	7.70	0.65	64.76	1.36	0.03	0.04	0.00	23.8	99.90

Table 2. Mineral composition of cement (OPC) and expansive additives (EA).

	C_3S	C_2S	C_3A	C_4AF	Gypsum	Anhydrite	Ye'elimite	Free Lime (CaO)	Bassanite	Calcite	Total
OPC	50.0	24.4	10.0	8.2	1.1	-	-	-	1.5	4.79	100
EA	-	-	-	3.47	-	34.78	11.14	44.69	-	1.34	99.6

2.1 Mix Design and Storage Conditions

Three different cement paste mixes were prepared by replacing a portion of the cement with EA. The weight ratios of EA used were 0%, 10%, and 15% of the total weight of the solids. A w/b ratio of 0.4 was used in the current study. The cement and EA were dry-mixed using a Hobart paddle mixer for 90 seconds at 140 rpm. Next, water was added, and initial mixing was carried out for 30 seconds at 140 rpm, followed by a 15-second rest and further mixing for 90 seconds at 285 rpm. The paste was then scraped from the sides, and the final mixing was conducted for 120 seconds at 285 rpm. To understand the effect of EA and external restraint on the microstructure of the cement paste, the resulting mixes were kept in two different conditions.

2.1.1 Free condition

After the cement paste mixing, the paste was kept in small petri dishes (diameter 35mm, height 13mm) and sealed using a parafilm tap. Samples were kept in an environmental chamber with a temperature of 20±0.5 °C for 8 hours. Further, to allow the expansion to occur in stress-free conditions, the samples were demolded and wrapped with plastic foil to maintain a sealed curing condition and kept again in the environmental chamber.

2.1.2 Restraint conditions

For the restraint conditions, the cement paste was poured and kept in aluminum tubes. The outer diameter and thickness of the tube were 20mm and 1mm, respectively. The length of the tube was 28 cm. The ends of the tube were closed with the help of end caps using epoxy to maintain restraint conditions in the longitudinal direction. Samples were kept in the environmental chamber.

2.2 Mercury Intrusion Porosimetry

After seven days the specimens were removed and broken into small pieced of size 3-8 mm for hydration stoppage. For the restraint specimens tubes were cut from the center, and cement paste samples were removed for the hydration stoppage. Solvent exchange methods is followed for hydration stoppage. The specimens were soaked in isopropanol for 72 hours and then vacuum filtered. Further, the specimens were kept for vacuum drying for seven days.

Table 3: Nomenclature of the different mixes analyzed by MIP

Name	EA (%)	Restraint condition
WC40_Ref	0	Free
WC40EA10_Free	10	
WC40EA15_Free	15	
WC40EA10_Rest	10	Restrained
WC40EA15_Rest	15	

For MIP, specimens were broken into small pieces with size smaller than 5mm. MIP tests were performed on dried specimens using a porosimeter (Quantachrome Poremaster, Tokyo, Japan). During the intrusion process, the equilibrium pressure (P) is related to the diameter (d) using the Washburn equation as given below:

$$P = \frac{-4\gamma \cos\theta}{d} \quad (1)$$

Here γ denotes the surface tension of mercury, which is taken to 0.45 N/m. θ represents the contact angle, and its value is taken to be 140°. The pore size distribution of the specimen can be obtained through the first derivative of the cumulative volume of the pores obtained from the MIP. Table 3 outlines all the specimens tested by the MIP and the nomenclature followed in the current study.

3 Experimental Results

This section will discuss the effect of EA and external restraint on the total porosity and pore size distribution of the cement paste. Figure 1(a) plots the cumulative pore volume of cement paste with different amounts of EA under stress-free conditions. The porosity of the specimens for the reference specimen was found to be 11.2%, while the porosity of samples with 10 and 15% EA was found to be 15.6 and 21.2 %, respectively. For the mix with EA the free expansion is expected to reach higher than 10000μm/m. It is apparent that the porosity of samples increased with the addition of EA when the samples were allowed to expand freely. Figure 1(b) shows the pore size distribution of the specimens under free conditions, and it is evident that pore structures have become coarser with higher addition of EA. The peak between 0.1-10μm has shifted to right side, indicating a higher pore diameter for the paste EA. It is also visible that the height of the peak has become higher, implying higher pore volume in this range (0.1 to 10μm). The peak between 0.01-0.1μm has shifted to the left side, implying the pore size in this range has become finer. Ou et al. (2023) conducted the MIP measurements for the cement paste with CSA-based additives using w/b 0.45 with 10%EA. The cumulative porosity of the paste increased from 10.3 to 12.4 % with the addition of EA along with the coarsening of the pore structure. Shen et al. (2020) studied the pore structure of ultra-high-performance concrete with EA (CSA based). They reported a decrease in the amount of pores between 0.01 to 0.1μm and a slight increase in pores in the range of 1 to 10μm.

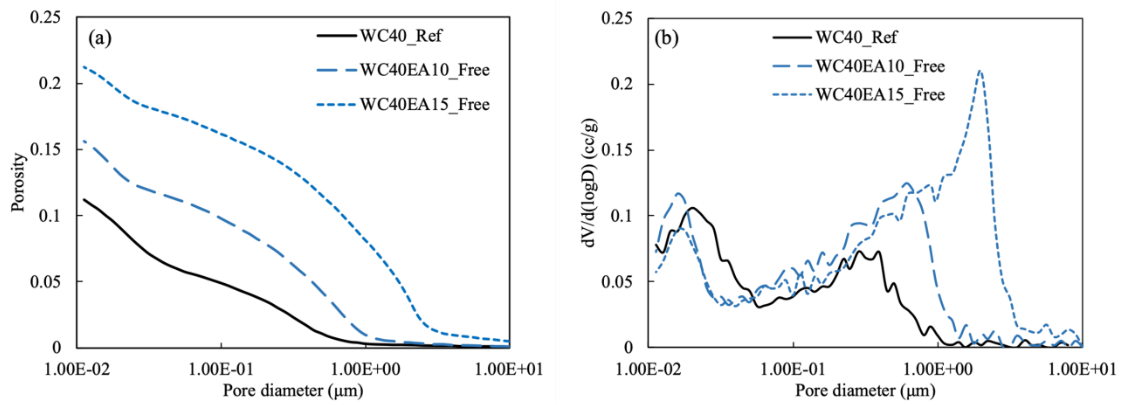

Figure 1. Cumulative pore volume (a) and pore size distribution (b) of cement paste with EA under stress-free conditions

Figure 2(a) plots the cumulative pore volume of paste with different amounts of EA under restrained conditions. It can be observed that, unlike free expansion, cumulative pore volumes for paste with different amounts of EA are close to each other under restrained conditions. The pore volume measured by MIP for 10 and 15% EA was 11.6 and 10.3%. Figure 2(b) shows the pore size distribution for the restrained case. For the specimen with 10%EA, the pores in the range of 0.1 to 10μm and an increase in the amount of pores in the range of 0.01 to 0.1μm increased. This observation differs from the one observed for the case of free expansion of cement paste. However, a different trend is observed for the 15% EA with a peak observed between the pore size range of 1 to 10μm, which is contrary to the observation from the literature (Duc Van et al., 2020). This might be attributed to inhomogeneity in filling the paste in the tube or compaction.

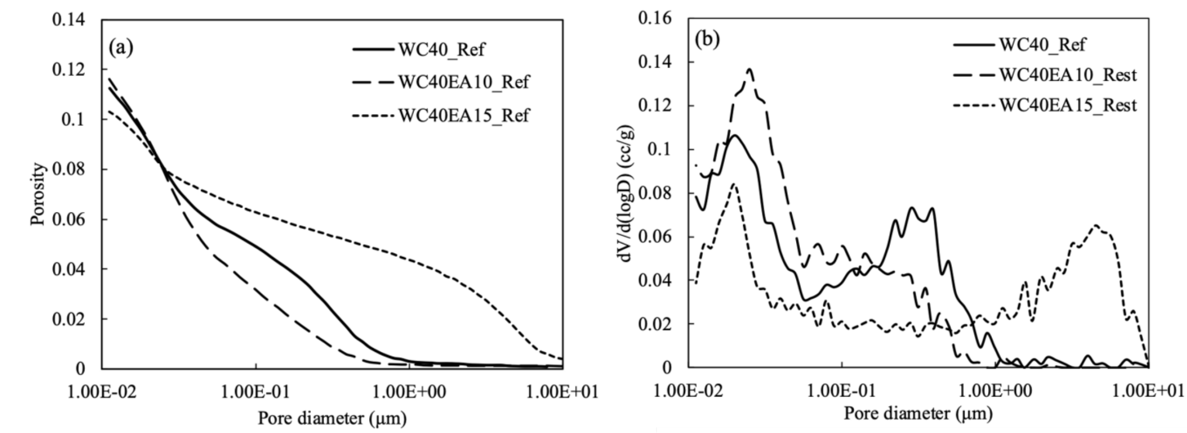

Figure 2. Cumulative pore volume (a) and pore size distribution (b) of cement paste with EA under restrained conditions

4 Discussion

The crystallization pressure theory is commonly used to explain the mechanism of internal expansive reactions and their origin. This theory is based on Corren's law, which states that the pressure acting on expansive crystals (such as portlandite and ettringite in the case of EA) is caused by a high degree of supersaturation in the pore solution(Correns, 1949; Flatt et al., 2007). This results in the conversion of chemical energy from the supersaturation of the pore solution to mechanical energy, causing expansion. Crystallization of expansive hydrates occurs in pores of different sizes due to a higher degree of supersaturation, with higher crystallization pressure acting through crystal precipitation in smaller pores and vice versa(Scherer, 1999, 2004). For cement paste subjected to stress-free expansion, peak between 0.01 to 0.1 μm shifts to the finer side as the expansive hydrates precipitate in such pores, causing the expansion. Consequently, the pore size in the range of 0.1 to 10 μm shifts to the coarser size, and overall porosity increases with an increase in the amount of EA.

According to the crystallization pressure theory, a higher degree of supersaturation is required to cause expansion under restrained conditions compared to free expansion. Due to the influence of external stress, crystal dissolution occurs from smaller pores and precipitates in larger available pores, causing smaller expansion. This observation is supported by the pore size distribution of the paste tested under restrained conditions.

As both EA and external restraint affect the porosity of cement paste, they influence its mechanical properties. Gupta et al. (2021) conducted measurements of elastic modulus and compressive strength for cement paste under stress-free conditions, observing a decrement in strength and modulus with an increase in the amount of EA. Similarly, Hua reported a similar decrement in paste strength with EA under free conditions (Hua et al., 2022). The increase in porosity of the cement paste can be attributed to the decrease in mechanical properties. However, further studies are needed to explore the effect of external restraint on the mechanical behavior of cement paste and concrete.

5 Conclusions

The effect of EA and external restraint on the microstructure of the cement paste is studied using the MIP. The primary conclusion derived from the current study are:

1) Under stress-free conditions, the porosity of the cement paste increases with an increase in the amount of EA.
2) Under free conditions, pores below 0.1μm become finer with the addition of EA. The pores between 0.1 and 10 μm becomes coarser with an increase in the volume of such pores.
3) Overall, the pore structure of the paste becomes coarser with additions of EA under free conditions.
4) For retrained conditions, pore in the range of 0.1 to 1μm decreases due to the precipitation of expansive hydrates and limited expansion of paste.

Acknowledgements

The authors gratefully acknowledge Denka Limited for their financial support and for providing expansive additives.

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