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# The Effect of Improper Curing on Properties Which May Affect Concrete Durability

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## Abstract

Good curing, enabling prolonged hydration and the development of a well-developed microstructure, is imperative if concrete is to perform at its full potential. This may become more important with the increasing use of composite cements containing more slowly reacting additions. Furthermore, the effects of improper curing, i.e. compromised durability, may not become visible for many years.

A series of concrete mixes have been prepared of 20 or 50 MPa target mean strength, using either CEM I or CEM I + 30% fly ash as the binder. Mixes were designed with two different workabilities, (10-30 and 60-180 mm slump) Samples were cured in a fog room at 20 ± 3$^0$C and 99 ± 1% RH or under ambient conditions 20 ± 3$^0$C and 42 $\pm$ 5% RH. Performance was evaluated in terms of compressive strength, transport properties and resistance to carbonation. Equivalent paste samples were characterised by TGA, XRD and SEM to follow hydration and microstructural development.

Improper curing did not greatly affect compressive strength. However, the effects on transport properties, and therefore properties that may affect durability, were more profound. The effects of non-ideal curing were greater for lower strength mixes, those containing fly ash and, to a less extent, less workable mixes.

**Keywords:** curing; durability; Permeability & pore-related properties; microstructure

**Research Significance:**

As society strives to reduce the carbon footprint of construction, the cement industry is reducing the clinker factor by the use of additions such as fly ash. Fly ash is known to hydrate more gradually than Portland cement, hence may require prolonged curing. This study shows that the impact on transport properties, which may have implications for durability, are more pronounced than the impact on compressive strength. This study has shown that good site practice and proper curing is necessary to ensure long-term concrete performance.

## 1. Introduction

Portland cement concrete underpins modern life. Global concrete production is approaching $20\times 10^{12}$ kg per annum. Sabir *et al.* (Sabir et al., 2001) wrote that concrete's consumption is second only to water as the most utilized man-made substance on the planet (Meyer, 2005, Gaimster and Munn, 2007). Furthermore, as vast developing nations upgrade and invest in their infrastructure, this will continue to increase (Purnell and Black, 2012).

Portland cement is an indispensable component of concrete, but the production of almost 4 billion tonnes of Portland cement per year has environmental impacts (Flower and Sanjayan, 2007, Collins, 2010). The cement industry is accountable for 5-10 % of the anthropogenic $CO_2$ (Meyer, 2009, Damtoft et al., 2008, Mehta, 2001). The global greenhouse gas emissions generated by production of cement has led to research into how these emissions can be reduced. One such approach is the increased use of supplementary cementitious materials. These materials may be in their natural form, industrial by-products, or those that necessitate only minimal further processing to produce. Fly ash, blast furnace slag, silica fume and metakaolin are additions that can be used as partial replacements for Portland cement. These materials can enhance concrete durability, lessen the risk of thermal cracking in mass concrete and possess lower embodied energy- and $CO_2$ than Portland cement (Berndt, 2009, Siddique, 2004).

Fly ash is an essential pozzolan, a by-product of coal combustion in the generation of electricity. Most fly ash particles are spherical and amorphous, ranging in size between 10 and 100 μm (Shi et al., 2012). Fly ash is an important pozzolan, and it has been established that fly ash may improve the long term strength of concrete with proper curing (Ramezanianpour and Malhotra, 1995, Haque, 1990, Toutanji et al., 2004, Thomas et al., 1989). However, fly ash hydrates more slowly than cement, and thus concrete containing fly ash may require longer curing times. Curing of concrete is also essential for optimal durability (Al-Gahtani, 2010), and may be compromised by improper curing practices. If the concrete is not properly cured, then the surface layer, about 30 to 50 mm (Neville, 2011, Gowripalan et al., 1990), is most affected due to the potential for evaporation of water from the concrete surface. This is particularly significant as it constitutes the cover zone for most reinforced concrete construction (Gowripalan et al., 1990). This means that regulation of moisture is not

just for improving the compressive strength of a structure, rather it also reduces surface permeability and increases hardness, so as to improve the longevity of a structure, especially one exposed to harsh environments. Basheer *et.al* (Basheer et al., 2001) wrote that improper curing is one of the factors that have reduced the service life of many structures or resulted in mandatory comprehensive repairs, with great economic costs.

The effects of improper curing may not appear to demonstrably affect concrete quality immediately, and thus its impact on strength may not be an appropriate parameter with which to measure the durability of a structure. Cabrera *et.al* (Cabrera et al., 1989) commented that assessing curing methods based on the strength of the concrete does not adequately predict the performance of the concrete in a structure, since the durability of the concrete is controlled more by its porosity and permeability than its strength.

One of the most important parameters influencing the durability of concrete is its permeability. Permeability of concrete determines the ease with which potentially deleterious substances can penetrate the concrete. Thus, permeability dictates the extent to which concrete can be affected by external agents, and may thus be used as a measure of concrete durability.

Gopalan (Gopalan, 1996) wrote that the most influential characteristics affecting concrete durability were the pore structure and alkalinity of the cover concrete. These factors can be followed by measuring the sorptivity and carbonation resistance respectively. Furthermore, both of these factors are greatly influenced by the curing conditions, with improper curing increasing permeability and reducing the quantity of carbonateable matter in the hardened cement paste.

In addition to the use of additions to reduce the embodied carbon of concrete, judicious concrete mix design can also achieve these aims (DAMINELI et al., 2013). Replacement of 20% of the Portland cement in concrete can reduce the embodied carbon by 14% (Black and Purnell, 2016, Purnell and Black, 2012). Meanwhile, reductions in embodied carbon may be achieved by the use of a less workable concrete mix. However, a truly sustainable approach should consider not just the carbon footprint, but also consider long-term performance. Therefore, while stiff

mixes containing additions may have a lower carbon footprint, their durability must also be considered.

This study investigates the effect of improper curing on concrete specimens. It has also looked at the effect of improper curing on factors affecting durability, in particular resistance to carbonation. The results have been interpreted with reference to the microstructures and phase assemblages of ideally and improperly cured samples.

## 2. Materials and Methods

### 2.1 Materials

Eight different concrete mixes were prepared according to the method of Teychenne (Teychenné et al., 1997). The mixes were designed to examine the effects of variables known to have a considerable impact on the embodied carbon of concrete (Purnell and Black, 2012, Black and Purnell, 2016), namely mean compressive strength, binder type and workability. The different levels of each variable are shown in Table 1 and the mix designs shown in Table 2. This table also shows the nomenclature used throughout the rest of this paper for naming samples.

Concrete specimens were cast using CEM I 52.5N, compliant with BS EN 197-1 – 2011 (British Standards Institution, 2011). The fly ash used complied with BS EN 450-1:2012 (British Standards Institution, 2012). The chemical compositions of these materials, as determined by XRF, are shown in Table 3.

10 mm diameter uncrushed coarse aggregate and quartz sand of diameter 150 µm to 5mm was used. Figure 1 shows the particle size distributions of the aggregates. The aggregates were oven dried before use. Potable mains water within the laboratory was used.

Concrete specimens were prepared for compressive strength, permeability, sorptivity and carbonation testing. Samples were cast in steel moulds and covered with polythene sheeting. After 24 hours samples were stripped from the moulds and cured under one of two conditions; one in the fog room at temperature of 20 $\pm$ 3 $^0$C and 99 $\pm$ 1%RH and the second one under ambient conditions at 20 ± 3$^o$C, 42 ± 5%

RH. These curing conditions are henceforth referred to as ideal and ambient respectively.

## 2.2 Methods

### 2.2.1 Compressive strength test

The compressive strength of 100 X 100 X 100 mm concrete cubes was determined in triplicate after curing for 28 days. The unconfined compressive strength was measured using a Retrofit Tonipact Concrete and Transverse Beam Machine according to BS EN 12390-3 (British Standards Institution, 2009). The ideally-cured samples were tested immediately after removal from the fog room, the cubes just being made surface dry with a towel before testing. The samples cured under ambient conditions however were immersed in water for 3 hours prior to testing. This was to remove any influence of the degree of sample saturation on the measured strength (Chen et al., 2012, Shoukry et al., 2011).

### 2.2.2 Permeability test

Concrete permeability was measured by using a gas permeability cell developed by Cabrera and Lynsdale (Cabrera and Lynsdale, 1988). Figure 2 shows the Components of the Leeds cell used for the permeability test. Concrete samples of 50mm diameter and 40mm height were cast and cured for 28 days. Following curing, the samples were placed in an oven to dry to constant weight at 40°± 2°C. This drying temperature was selected as being high enough to allow for pore water to be driven off in a reasonable time and not so high as to result in decomposition of the C-S-H or the ettringite. Concrete samples (S) were placed into the rubber cylinder (A) which was placed into the plastic ring cylinder (B). This was placed into the Leeds cell and the metal O ring (C) was put in place before the cell cap (D) was positioned. A force was applied vertically downward to form a seal to ensure that all nitrogen passing through the system would go directly through the sample.

Nitrogen gas was forced through the sample at a defined pressure after which the flow was allowed to normalise to a steady flow (generally 10-15 minutes). The time for a known volume of nitrogen to pass through the sample was recorded by using a

bubble flow meter. This was repeated three times to provide an average and standard deviation and to ensure that the flow had fully normalised.

This procedure was repeated for each sample at different applied pressures; 0.5,1, 1.5,2 and 2.5 bar (above atmospheric). Ten millimetres (10mm) flowmeter tube was used for more permeable samples, and five millimetres (5mm) for less permeable samples. All mixes were tested in triplicate at 28 days following curing under standard and ambient conditions.

Permeability was then calculated according to the method proposed by Grube and Lawrence (Grube and Lawrence, 1984), with a slight alteration to take account for the change in gas, as shown in equation below.

$$\mathbf{k} = \frac{\mathbf{2P_2 \, x \, \upsilon_L 1.78 \, x \, 10^{-6}}}{\mathbf{A}\left(\mathbf{P_1}^2 - \mathbf{P_2}^2\right)}$$

Where: $P_1$ is the absolute applied pressure (bar), $P_2$ is pressure at which the flow rate is measured in bar and is 1.01325 bar, υ is measured in $cm^3/s$, ʟ and a measured in m and $1.78 \, x \, 10^{-6}$ = dynamic viscosity of nitrogen at 20℃ (g/cm/s).

### 2.2.3 Sorptivity tests

Sorptivity tests were carried out using similar methods by Tasdemir (Tasdemir, 2003) and Güneyesi (Güneyisi and Gesoğlu, 2008), on concrete cubes which had been cured for 28 days and dried in an oven at 40℃ to constant mass. A schematic diagram showing the set-up of the sorptivity test is presented in Figure 3. After drying, the lower areas on the sides of the specimens were coated with petroleum jelly so as to ensure unidirectional flow; the coated samples were placed in a trough of water, with the water level kept at about 5mm from the base of the specimens. The specimens were removed from the trough and weighed at different time intervals up to one hour to evaluate mass gain. At each of these times, the mass of water adsorbed by each of the specimen was obtained, and from this the sorptivity coefficient *(k)* was calculated according to

$$K = \frac{Q}{A\sqrt{t}}$$

Where $Q$ is the amount of water adsorbed in $m^3$, t is the time in secs, $A$ is the cross-sectional area of the specimen that was in contact with the water in $m^2$, and $k$ is the sorptivity coefficient in $m^3/m^2s^{1/2}$.

### 2.2.4 Carbonation

The depth of carbonation was determined on cubes cured for 28 days. After curing, samples were allowed to dry in an ambient environment for two weeks, as stated in clause 6.2.7 of BS 1881 – 210(British Standards Institution, 2013). This step is necessary to stabilize the internal relative humidity of the concretes, and reduce the variation in the internal relative humidity between the concrete samples before subjecting them to accelerated carbonation tests. Samples were then exposed to pure $CO_2$ for two weeks at 20℃ and 65% RH (using a saturated NaBr solution). Carbonation depths were determined by spraying a freshly broken surface with alcoholic 1% phenolphthalein solution. The average depths of carbonation were measured at two points perpendicular to three faces of the broken concrete cubes. The depths of penetration of the trowelled face were ignored due to the influence of trowelling.

## 2.3 Sample preparation for SEM, XRD and TG Samples

Paste samples were used for SEM, XRD and TG analysis, as the presence of quartz in concrete samples can affect the accuracy of the tests. Samples were prepared by using the same water / binder ratio as used for the concrete mixes. The fresh prepared pastes were poured into 14-16mm diameter, 8 ml plastic tubes, fitted with tight lids and placed in tube rotator at ten rph overnight to prevent any bleeding and segregation. The samples were removed from the rotator after 12 hours, whereupon the ideally cured samples were sealed and cured in a water bath at 23°C, while the plastic tubes for the ambient-cured samples were left open at both ends, and left to cure under ambient conditions. The samples were cured for 1, 7 and 28 days. The cured samples were cut using an isomet slow speed saw. After removal of the outer 1mm, two 2 mm thick slices were cut from each end and hydration stopped by solvent exchange. The four slices were immersed in isopropyl alcohol (IPA) overnight using a solution-to-sample ratio of 100:1 followed by drying in a vacuum

desiccator for another 12 hours. The outermost of these 2 mm slices was used for SEM imaging, imaging the surface 3 mm from the end of each tube.

### 2.3.1 XRD

Hydration stopped paste samples from the second slice, i.e. 3-5mm into the sample, were ground to a fine powder with a pestle and mortar before being placed in a 10mm diameter holder using the back loading method. In order to minimise the effect of preferred orientation, samples were prepared carefully and minimal pressure was applied when back loading the samples onto the sample holders (Aranda et al., 2012). A Bruker D2 Phaser with a Cu X-ray source working at 300W (30KV at 10mA) was used. The scan step size was $0.02^{o}$, the collection time 1s, and patterns were collected over the range $7^{0}$ to $70^{0}$ 2θ with effective total time of 3403 s. The divergence slit, air scatter, filter and Soller slits were set to 1, 1, 0.5 and 2.5 respectively.

### 2.3.2 TGA

Powder samples prepared as for XRD analysis were also used for thermal analysis. The portlandite (CH) and bound water $W_n$ contents were measured by thermal gravimetric analysis (TGA). A Stanton Redcroft Thermal Gravimetric Analyser TG 760 was used. Samples of ≈ 6-8mg were loaded in a clean crucible and heated under nitrogen from $20^{0}$ to $1000^{0}$C at a constant rate of $20^{0}$C/min. The tangent method was used to evaluate the portlandite (CH) content and the equivalent $(CH)_{eq}$ from the calcium carbonate ($CaCO_3$) content. The sum of the CH and $(CH)_{eq}$ contents were used to assess the degree of hydration of each sample. The following equations were used to calculate the CH

$$\%Ca(OH)_2 = \left(ML_{CH} \times \frac{M_{CH}}{M_{H_2O}}\right) \div \text{Residue}$$

$$CaCO_3 = \left(ML_{CaCO3} \quad X \quad \frac{M_{CaCO_3}}{M_{CO_2}}\right) \div \text{Residue}$$

$$[Ca(OH)_2]_{eq} = \left(ML_{CaCO3} \times \frac{M_{Ca(OH)_2}}{M_{CO_2}}\right)$$

Where:

$ML_{CH}$ mass loss due to dehydroxylation of CH

$ML_{CaCO3}$ mass loss due to decarbonation of $CaCO_3$

$M_{CH}$ molar mass of CH, taken as 74g/ mol

$M_{H2O}$ molar mass of water, taken as 18g/mol

$M_{CaCO3}$ molar mass of $CaCO_3$. $M_{CaCO3}$ = 100g/mol

$M_{CO2}$ molar mass of $CO_2$ = 44g/mol

Total CH = $\%Ca(OH)_2 + [Ca(OH)_2]_{eq}$

The bound water was taken as the mass loss between 50 ℃ and 550 ℃, at which point it was assumed that all the phases containing water had fully decomposed. $W_n$, normalised to the total mass loss at 550℃ was calculated using the equation as described by Whittaker *et al*. (Whittaker et al., 2014).

$$W_n = \left(\frac{w_{50-w_{550}}}{w_{550}}\right) \text{ X } 100$$

Where:

$W_{550}$ mass loss at $550^0$C

$W_{50}$ mass loss at $50^0$C

**2.3.3 SEM**

SEM samples were prepared from ambient and ideal cured samples which had been cured for 28 days. The 2mm thick sample which had been cut and hydration stopped was resin impregnated and polished using silicon carbide paper and then diamond paste. The sample was placed in the resin such that the cut surface was to be viewed. As such, the degree of hydration was measured at a depth of 1mm from the surface of the paste. A Jeol 5900 LV scanning electron microscope fitted with a backscatter electron detector was used for imaging. An accelerating voltage of 15KeV and 10mm working distance was used. The degree of hydration was measured on 25 images obtained at 400X magnification using the method described by Whittaker(Whittaker et al., 2014).

A consistent analysis technique was applied to all the images by using a grey level histogram as shown in below Figure 4. The histogram was obtained for each image. Four components of the hydrated paste microstructure: capillary porosity, calcium hydroxide (CH), calcium silicate hydrate gel along with other hydration products, and unhydrated cement could be identified in the histogram. The histogram also indicates the number of pixels in the image having each possible brightness value (between 0 and 225). Pores in the microstructure appear as dark spots on the electron images and can be easily distinguished from the hydrated phases (C-S-H and CH), C-S-H are dark grey

The Java applet Image J was used to calculate the amount of unreacted cement, from which the equation below was used to calculate the degree of hydration for cement and fly ash blends.

$$DH\frac{cem}{SEM}\ (t) = 1 - \frac{V_{(t)cem}}{V_{(o)\ cem}}$$

$$DR\frac{Add}{SEM}\ (t) = 1 - \frac{V_{(t)Add}}{V_{(o)Add}}$$

Where:

$V_{(o)cem}$ is the volume fraction of cement before hydration

$V_{(o)Add}$ is the volume fraction of unreacted addition before hydration

$V_{(t)cem}$ is the volume fraction of unhydrated cement at hydration time t, and

$V_{(t)Add}$ is the volume fraction of unreacted addition at hydration time t.

## 3. Results

### 3.1 Compressive strength

The 28 day compressive strengths of ambient- and ideal-cured CEM I and fly ash blend concretes is presented in Figure 5. In all instances the ideal-cured samples had higher strengths than the ambient-cured samples, in agreement with other findings (Xue et al., 2015, Ozer and Ozkul, 2004, Nahata et al., 2014). This effect was more pronounced for the lower strength samples. While it is known that samples

exposed to ambient conditions should develop lower strengths due to reduced degrees of hydration, it is assumed that the effect of improper curing diminishes with increasing compressive strength because the lower water/binder ratios inhibit water loss under ambient conditions. This was supported by the reduced permeability and sorptivity results for the higher strength samples (see below).

Comparing the results from the CEM I and fly ash blends, ambient curing appeared to have a slightly more detrimental effect on the composite cement mixes. Similar results were obtained by other researches (Ramezanianpour and Malhotra, 1995, Cao et al., 2000, Ozer and Ozkul, 2004, Tan and Gjorv, 1996, Nassif and Suksawang, 2002, Güneyisi et al., 2005, Aprianti et al., 2016) .This is perhaps as expected, since drying of the samples under ambient conditions is not instantaneous, thus the CEM I concretes will have hydrated to a greater degree than the blended cement systems, binding water and thus not allowing it to evaporate. Running counter to this, however, the increased rate of early-age clinker hydration when fly ash was present (see later), due to the filler effect and higher effective water/cement ratio, may have made the loss of performance less than may have been expected.

### 3.2 Sorptivity

Figure 6 shows the sorptivity data from the various samples. The sorptivity coefficients of ambient-cured concretes were considerably higher than those of the ideal-cured samples. This is in agreement with the findings of Khatib and Mangat who found that trowelled faces of concrete cubes cured to ambient conditions showed higher sorptivity and water absorption values than the centre of their cubes. It also agrees with the findings of others (Tasdemir, 2003, Bai et al., 2002, Khatib and Mangat, 1995). The figure also shows that sorptivity decreased with increasing compressive strength, and that non-ideal curing had a greater effect on the lower strength mixes.

Wet mixes often showed higher sorptivities than the corresponding stiff mixes, and non-ideal curing also seemed to have a slightly greater effect on the wet mixes than the corresponding stiff mixes, but the effect was not too clear cut. The increased

paste volume in the wet mixes may be the cause of this slightly elevated permeability.

Finally, the importance of ideal curing conditions for composite cements is clear in the figure. For the ideally cured samples, the fly ash-containing blends normally had lower sorptivities than their corresponding CEM I blends (Nath and Sarker, 2011, Camões et al., 2003, Gopalan, 1996, Khan and Lynsdale, 2002, Kelham, 1988, Bai et al., 2002). However, under ambient curing conditions, the situation was reversed and the fly ash blends all showed worse performance than the equivalent CEM I concretes. Again, similar findings were seen by Khatib and Mangat as a function of depth from the trowelled face of ambient-cured concrete specimens.

### 3.3 Permeability

The permeability results from the various samples are presented in Table 4. For the ideally cured samples, permeability decreased with increasing strength, as would be expected. Workability also had an effect on permeability, with stiff mixes generally having lower permeabilities then their corresponding wet mixes. The effect of binder type was less clear-cut. Stiff fly ash-containing concretes had higher permeabilities than their CEM I equivalents, but wet fly ash-containing mixes showed lower permeabilities.

The impact of improper curing on all samples was immediately clear, there being at least a ten-fold increase in permeability for almost all ambient-cured samples compared to their ideal-cured equivalents. The key factor in determining the effect of improper curing on permeability was the compressive strength, with the 20 MPa samples exhibiting an average increase in permeability about 50% greater than the 50 MPa samples. While wet mixes generally showed higher permeabilities than their equivalent stiff mixes, both following ideal and ambient curing, concrete workability did not appear to influence the extent of the increase in permeability upon ambient curing. Somewhat surprisingly, binder type did not appear to have a clear effect on permeability but addition of fly ash reduces the permeability which can be seen in the table that permeability of ideal cured fly ash samples having lower permeability than CEM1 samples and this is in agreement with other researchers (Beglarigale et al., 2014, Dhir et al., 1987, Khan and Lynsdale, 2002).

### 3.4 Resistance to carbonation

Figure 7 shows the carbonation depth measured for the 20 MPa samples. Ambient curing had a detrimental effect on carbonation resistance. The improvement in carbonation resistance with improved curing conditions has been shown previously (Das et al., 2011, Atiş, 2003, Ewertson and Petersson, 1993, Roziere et al., 2009, Lo and Lee, 2002, Balayssac et al., 1995, Fattuhi, 1988, Younsi et al., 2013). Stiff mixes showed slightly higher carbonation rates than wet mixes, but the impact of improper curing was not related to concrete workability. Similar conclusions could be made concerning binder type, where there were no clear, consistent trends in the data but ideally cured fly ash blends performed better than the equivalent CEM I mixes (Das and Pandey, 2011, Das et al., 2011, Atiş, 2003). This is in line with the permeability data.

### 3.5 Degree of hydration

In the previous sections, it has been demonstrated that non-ideal curing affected strength development and transport properties. The loss of water from the hydrating cement, prior to complete hydration, had impaired performance. As discussed, the presence of water is essential for continued hydration. Therefore, paste samples were prepared with matching water/binder ratios to equivalent concrete samples and cured under ideal or non-ideal conditions. These paste samples were then characterised to determine the degree of cement hydration, which could then be related to the engineering performance measures detailed above.

Figures 8 and 9 show selected SEM images of ambient and ideal cured samples of CEM I and fly ash paste. The field of view in each image is 326 x 244 µm. The samples show a mixture of hydrated cement paste, residual anhydrous material and coarse porosity (with the smallest feature being ca. 2x2 microns) (Whittaker et al., 2014). The figures therefore allow an assessment of the degree of cement hydration and of coarse capillary porosity.

The figures show how, as expected, capillary porosity was greater for the lower strength samples. The wet mixes were more porous than the equivalent dry mixes. This is in line with the intrinsic permeability measurements presented earlier.

Meanwhile, changing the binder from CEM I to a fly ash blend did not appear to have a significant effect on the microstructure.

Most clearly, ambient-cured samples exhibited reduced degrees of hydration, evidenced by the increased presence of anhydrous clinker (appearing as bright angular features), and much higher capillary porosities than their ideal-cured counterparts. The figures are a clear indication of how ideal curing can improve the pore structure of concrete, as has been reported by others previously (Mangat and El-Khatib, 1992, Khatib and Mangat, 1999, El-Sakhawy et al., 1999, Toutanji et al., 2004, Elahi et al., 2010).

Using the approach developed elsewhere (Kocaba, 2009, Whittaker et al., 2014), and as described in 2.3.3, it was then possible to determine the degree of clinker hydration in each of the samples. Table 5 and Figure 10 show the degree of clinker hydration for each of the samples. In all cases, the ideal cured samples showed a higher degree of hydration than their corresponding ambient cured ones, confirming that prolonged moist hydration increases the degree of hydration (Bentz et al., 1997, Chen and Wu, 2013). Allowing the samples to dry under ambient curing conditions affected hydration, in turn leading to higher porosity and coarser pore structure compared to ideal-cured concrete (Patel et al., 1988).

Workability had no discernible effect on the degree of hydration.

Figure 10 also shows that the degree of clinker hydration was consistently higher for the fly ash-containing blends than their corresponding CEM I samples, irrespective of curing conditions. This may be ascribed to the filler effect and increased effective water/cement ratio at early ages (Lawrence et al., 2003, Hanehara et al., 2001, Pane and Hansen, 2005, Lam et al., 2000, Sakai et al., 2005). Note also that, while the degree of clinker hydration is higher, this doesn't necessarily relate to the overall degree of binder hydration because the fly ash will only have reacted to a small degree (Poon et al., 2000, Lam et al., 2000, Li et al., 2000).

### 3.6 Bound water content

The hydration of cementitious materials can be examined by the bound water content. Bound water content cannot be directly related to the overall degree of

hydration but gives an indication of the progress of hydration (Whittaker et al., 2014, Pane and Hansen, 2005). Figure 11 shows the bound water content calculated for each sample at 28 days. As expected, the bound water contents of the ideal cured samples were higher than those of the ambient cured samples. This is a reflection of the higher degree of hydration, as evidenced by the SEM data and supporting the improved engineering performance.

As with many of the other measures, the workability of the sample did not appear to have an effect on the bound water content. The binder type, however, did affect the bound water content. All of the fly ash-containing samples showed lower bound water contents than the corresponding CEM I mixes. This is due to the low reactivity of the fly ash compared to the cement clinker and explains why, despite the higher degree of clinker hydration (as determined by SEM-BSE analysis) the engineering performance of the fly ash-containing concretes was not significantly better than that of the corresponding CEM I concretes. Furthermore, comparing the difference in bound water content for ideal and ambient cured samples according to binder type, the presence of fly ash in the concrete did not appear to affect the susceptibility to improper curing.

### 3.7 Thermogravimetry

Tables 6 and 7 show the CH content calculated from thermogravimetric analysis (TGA) of all the samples. Note that, since CH is a hydration product of cement, the CH content of the fly ash blends had been normalised to cement content (by dividing the obtained data by 0.7) in order to compare the results with the CEM I results.

For the CEM I systems, the portlandite contents were always higher when the samples were cured under ideal conditions, i.e. hydration had been allowed to proceed, so had led to the formation of portlandite from the hydration of alite and belite. However, the situation for the fly ash-containing samples was different. In these samples, the ideally cured specimens invariably had lower portlandite contents. This is due to the consumption of portlandite during the pozzolanic reaction.

### 3.8 XRD

Qualitative XRD analysis was carried out to examine the effect of curing on the hydration of the clinker phases present in the mixes. Figures 12 and 13 show typical XRD patterns obtained from CEM I and fly ash-containing pastes obtained after hydration for 1, 7 and 28 days. The reflections for portlandite (at 18°, 34° and 47° 2θ) can be considered as a good indicator of the evolution of hydration reactions (Mounanga et al., 2004, WANG et al., 2005). Ideal curing led to increased portlandite contents in the CEM I samples, while the portlandite contents in the ambient-cured samples did not increase so dramatically. Similarly, the decrease in the intensity of the peaks due to alite and belite, (near 29.5° 2θ) was more profound under ideal curing conditions.

As with the thermogravimetric data, the production of portlandite under ideal curing conditions was not so pronounced in the fly ash-containing mixes. The pozzolanic reaction led to portlandite consumption when the samples were cured under ideal conditions. However, the aforementioned consumption of alite and belite could be observed.

## Conclusions

The study shows that improper curing is detrimental to the performance of concrete. Improper curing leads to reduced compressive strength development and increased sorptivity and permeability. This is due to reduced levels of cement hydration as water evaporates from the concrete surface. This study has shown that the impact on sorptivity and permeability is far greater than the impact on compressive strength, with implications for the long-term durability of concrete.

The extent of the impact of improper curing depends on the properties of the concrete. Low strength concrete, which has a slightly higher water content and also an inherently higher porosity, is more greatly affected by improper curing than high strength concrete. This is presumed to be due to the ease with which water can evaporate from the surface of the more porous cement paste.

Composite cements, containing 30% fly ash, showed comparable strengths to CEM I concretes and improved transport properties when ideally cured. Improper curing however led to reduced performance. Strength was compromised by improper curing to a greater degree than for equivalent CEM I mixes. However, it was sorptivity and

permeability which were most severely affected. This was due to the reduced degree of cement hydration which will have affected the pozzolanic reaction between the fly ash and portlandite.

Concrete workability has been found to be a factor which can help to reduce the embodied carbon of concrete (Purnell & Black, 2012), with stiffer mixes having lower carbon footprints. This study has shown that while stiff concrete mixes may show improved transport properties, their lower paste volumes leave them more susceptible to carbonation.

This study confirms the need for good site practice, and also shows that embodied carbon should not be the only factor when considering the environmental performance of concrete. Rather, durability and whole life performance should also be considered.

Table 1 Variables used in the mix design

| Variable | Level |
|---|---|
| UCS | 20 MPa<br>50 MPa |
| Workability (slump) | 10-30 mm (stiff)<br>60-180 mm (wet) |
| Binder | CEM I 52.5N<br>70 % CEM I 52.5N + 30% fly ash |

Table 2: Mix designs used for the samples in this study. Note, water contents were those used having accounted for moisture uptake by the oven dried aggregates.

| Binder type | Target strength (MPa) | Slump (mm) | Nomenclature | W/b ratio | Water (kg/m$^3$) | Cement (kg/m$^3$) | PFA (kg/m$^3$) | Fine aggregate (kg/m$^3$) | Coarse aggregate (kg/m$^3$) |
|---|---|---|---|---|---|---|---|---|---|
| CEM1 | 20 | 10-30 | 20 S-C | 0.77 | 212.9 | 275 | 0 | 849 | 1033 |
| | 50 | | 50 S-C | 0.42 | 207. 4 | 499 | 0 | 650 | 1007 |
| CEM1 | 20 | 60-180 | 20 W-C | 0.75 | 257.2 | 343 | 0 | 911 | 805 |
| | 50 | | 50 W-C | 0.40 | 250.5 | 624 | 0 | 675 | 760 |
| 30% fly ash | 20 | 10-30 | 20 S-FA | 0.78 | 197.7 | 223 | 96 | 844 | 1026 |
| | 50 | | 50 S-FA | 0.42 | 191.6 | 405 | 174 | 631 | 978 |
| 30% fly ash | 20 | 60-180 | 20 W-FA | 0.76 | 237.0 | 277 | 119 | 906 | 801 |
| | 50 | | 50 W-FA | 0.40 | 229.6 | 504 | 216 | 651 | 732 |

Table 3 Compositions of raw material, determined by XRF (% weight)

| Compound | % | CEM I | Fly ash |
|---|---|---|---|
| $SiO_2$ | % | 19.85 | 50.73 |
| $Al_2O_3$ | % | 4.93 | 25.49 |
| $Fe_2O_3$ | % | 2.14 | 10.05 |
| CaO | % | 63.95 | 2.28 |
| MgO | % | 2.01 | 1.63 |
| $SO_3$ | % | 3.13 | 0.41 |
| $K_2O$ | % | 0.58 | 3.46 |
| $Na_2O$ | % | 0.37 | 0.90 |

Table 4 Ambient and Ideal Permeability coefficients.

| | Ideal cured ($10^{-16}$ $m^2$) | Ambient cured ($10^{-16}$ $m^2$) | Percentage increase upon ambient curing |
|---|---|---|---|
| 20MPa Stiff mix CEM I | 0.29 | 6.26 | 2160 |
| 50MPa Stiff mix CEM I | 0.14 | 1.65 | 1180 |
| 20MPa Wet mix CEM I | 0.61 | 7.00 | 1150 |
| 50MPa Wet mix CEM I | 0.23 | 2.01 | 870 |
| 20MPa Stiff mix fly ash | 0.45 | 6.38 | 1420 |
| 50MPa Stiff mix fly ash | 0.16 | 1.64 | 1030 |
| 20MPa Wet mix fly ash | 0.30 | 5.60 | 1870 |
| 50MPa Wet Mix fly ash | 0.21 | 2.57 | 1220 |

Table 5 Degree of hydration obtained from SEM Images

| | Ambient DOH | Ideal DOH |
|---|---|---|
| 20 Stiff mix CEM1 | 79.58 | 82.81 |
| 50 Stiff mix CEM1 | 60.9 | 73.68 |
| 20 Wet mix CEM1 | 73.08 | 81.76 |
| 50 Wet mix CEM1 | 66.22 | 73.02 |
| 20 Stiff mix fly ash | 86.83 | 91.55 |
| 50 Stiff mix fly ash | 80.87 | 87.62 |
| 20 Wet mix fly ash | 81.13 | 92.15 |
| 50 Wet mix fly ash | 81.63 | 90.61 |

Table 6: Portlandite contents determined by thermal analysis for each CEM I mix

| Mix | Age | $Ca(OH)_2$ | $CaCO_3$ | $Ca(OH)_{2eq}$ | CH total |
|---|---|---|---|---|---|
| 20MPa Stiff mix | Ambient | 6.52 | 5.41 | 3.03 | 9.55 |
| 20MPa Stiff mix | Ideal | 9.74 | 0.90 | 0.50 | 10.24 |
| 50MPa Stiff mix | Ambient | 6.91 | 2.26 | 1.35 | 8.26 |
| 50MPa Stiff mix | Ideal | 8.42 | 0.00 | 0.00 | 8.42 |
| 20MPa Wet Mix | Ambient | 7.51 | 3.56 | 2.02 | 9.53 |
| 20MPa Wet Mix | Ideal | 10.37 | 2.72 | 1.51 | 11.89 |
| 50MPa Wet mix | Ambient | 7.29 | 2.59 | 1.51 | 8.80 |
| 50MPa Wet Mix | Ideal | 7.37 | 1.02 | 0.59 | 7.96 |

Table 7: Portlandite contents determined by thermal analysis for each fly ash-containing mix (note data have been normalised to cement content).

| Mix | Age | $Ca(OH)_2$ | $CaCO_3$ | $Ca(OH)_{2eq}$ | CH total |
|---|---|---|---|---|---|
| 20MPa Stiff mix | Ambient | 8.06 | 2.51 | 1.51 | 9.57 |
| 20MPa Stiff mix | Ideal | 8.21 | 1.98 | 1.18 | 9.38 |
| 50MPa Stiff mix | Ambient | 7.43 | 5.21 | 3.20 | 10.63 |
| 50MPa Stiff mix | Ideal | 7.05 | 1.95 | 1.18 | 8.23 |
| 20MPa Wet Mix | Ambient | 6.04 | 4.73 | 2.86 | 8.90 |
| 20MPa Wet Mix | Ideal | 8.04 | 1.86 | 1.09 | 9.14 |
| 50MPa Wet Mix | Ambient | 6.43 | 4.10 | 2.52 | 8.96 |
| 50MPa Wet Mix | Ideal | 7.18 | 1.84 | 1.09 | 8.28 |

## List of Figure Captions

**List of Table Captions**

Table 1 Variables used in the mix design

Table 2: Mix designs used for the samples in this study. Note, water contents were those used having accounted for moisture uptake by the oven dried aggregates.

Table 3 Compositions of raw material, determined by XRF (% weight)

Table 4 Ambient and Ideal Permeability coefficients.

Table 5 Degree of hydration obtained from SEM Images

Table 6: Portlandite contents determined by thermal analysis for each CEM I mix

Table 7: Portlandite contents determined by thermal analysis for each fly ash-containing mix (note data have been normalised to cement content).

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Figure 1

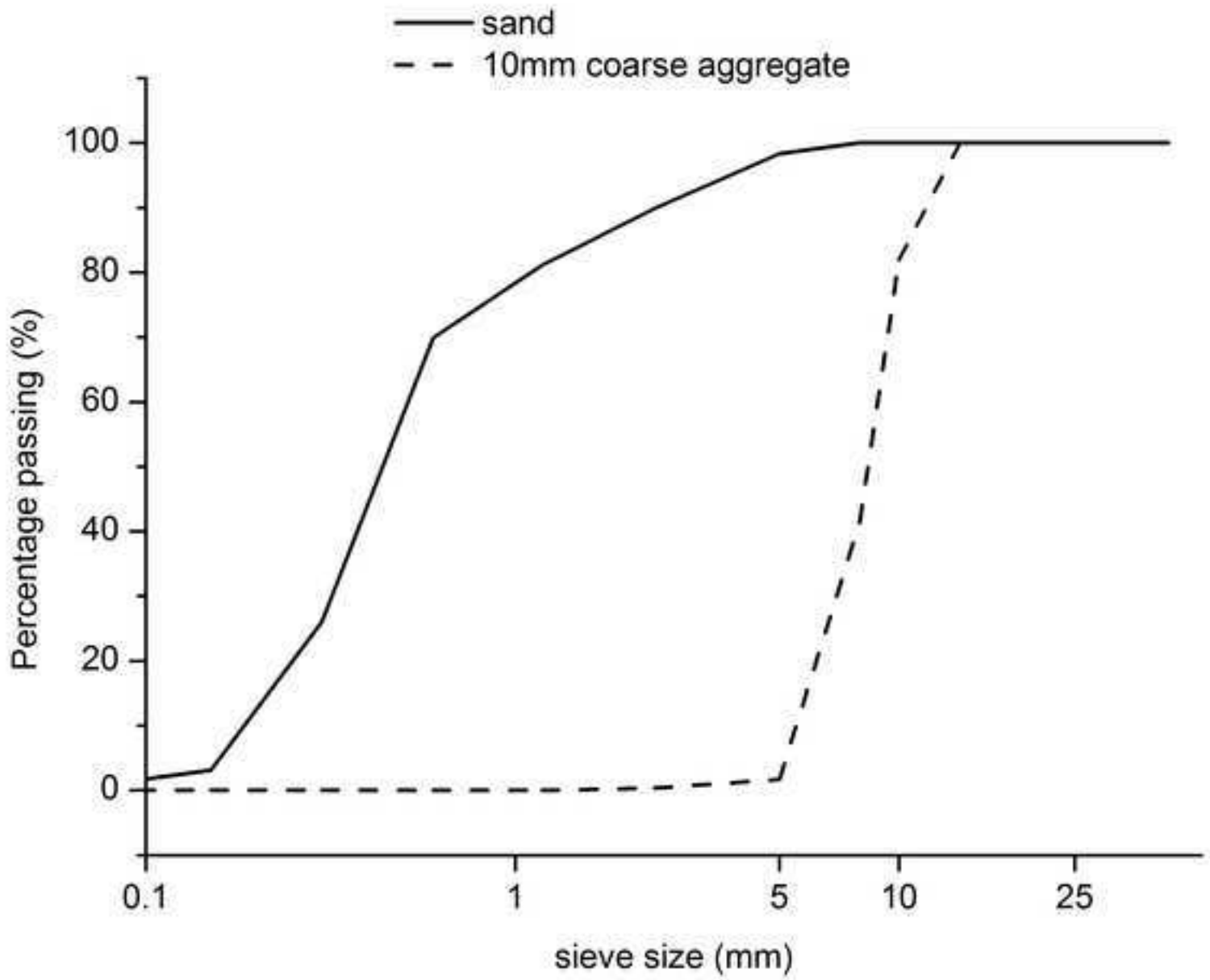

sand
10mm coarse aggregate
Percentage passing (%)
100
80
60
40
20
0
0.1
1
5
10
25
sieve size (mm)

Figure 2

Figure 3

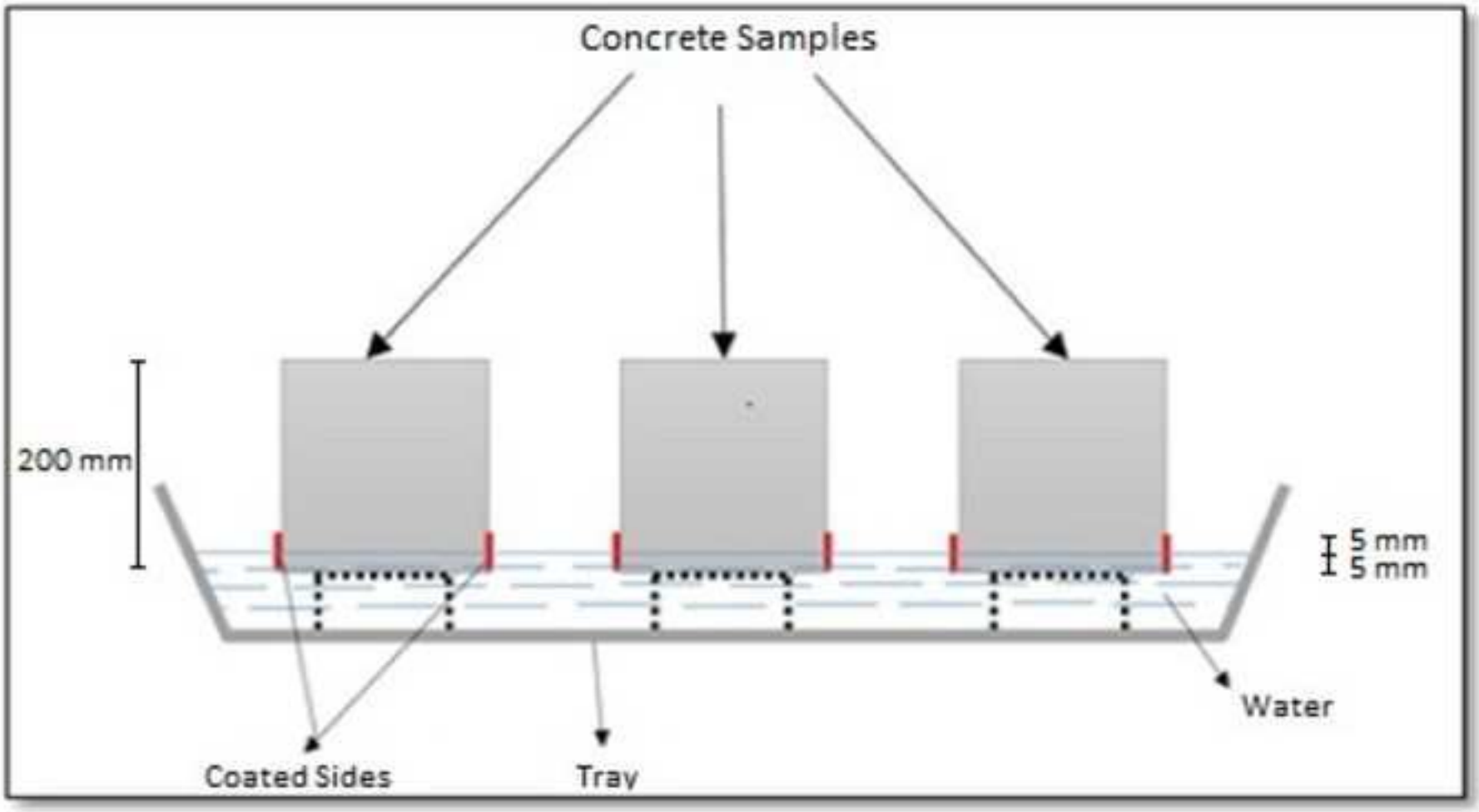


Figure 4

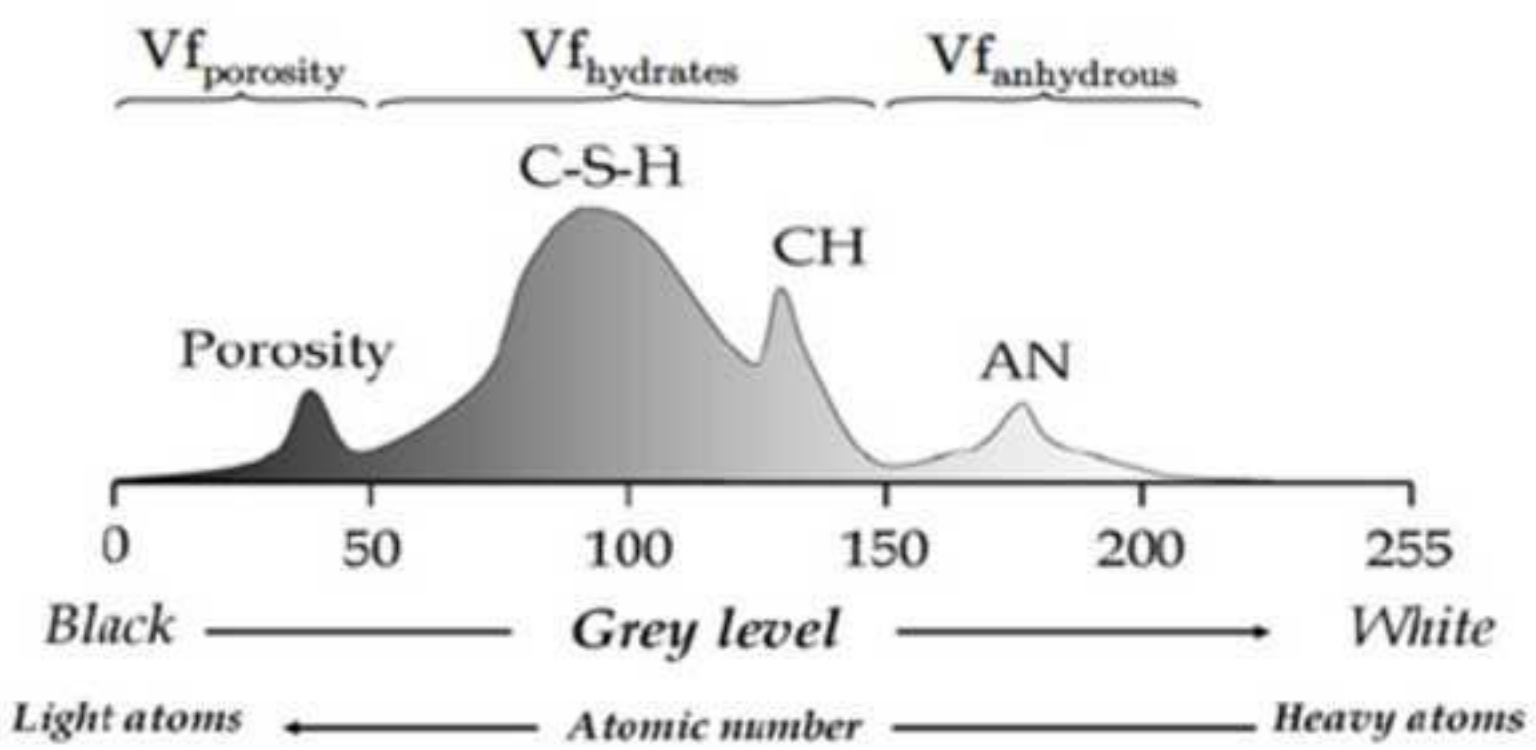

Vf porosity
Vf hydrates
Vf anhydrous
C-S-H
CH
Porosity
AN
0
50
100
150
200
255
Black
Grey level
White
Light atoms
Atomic number
Heavy atoms

Figure 5

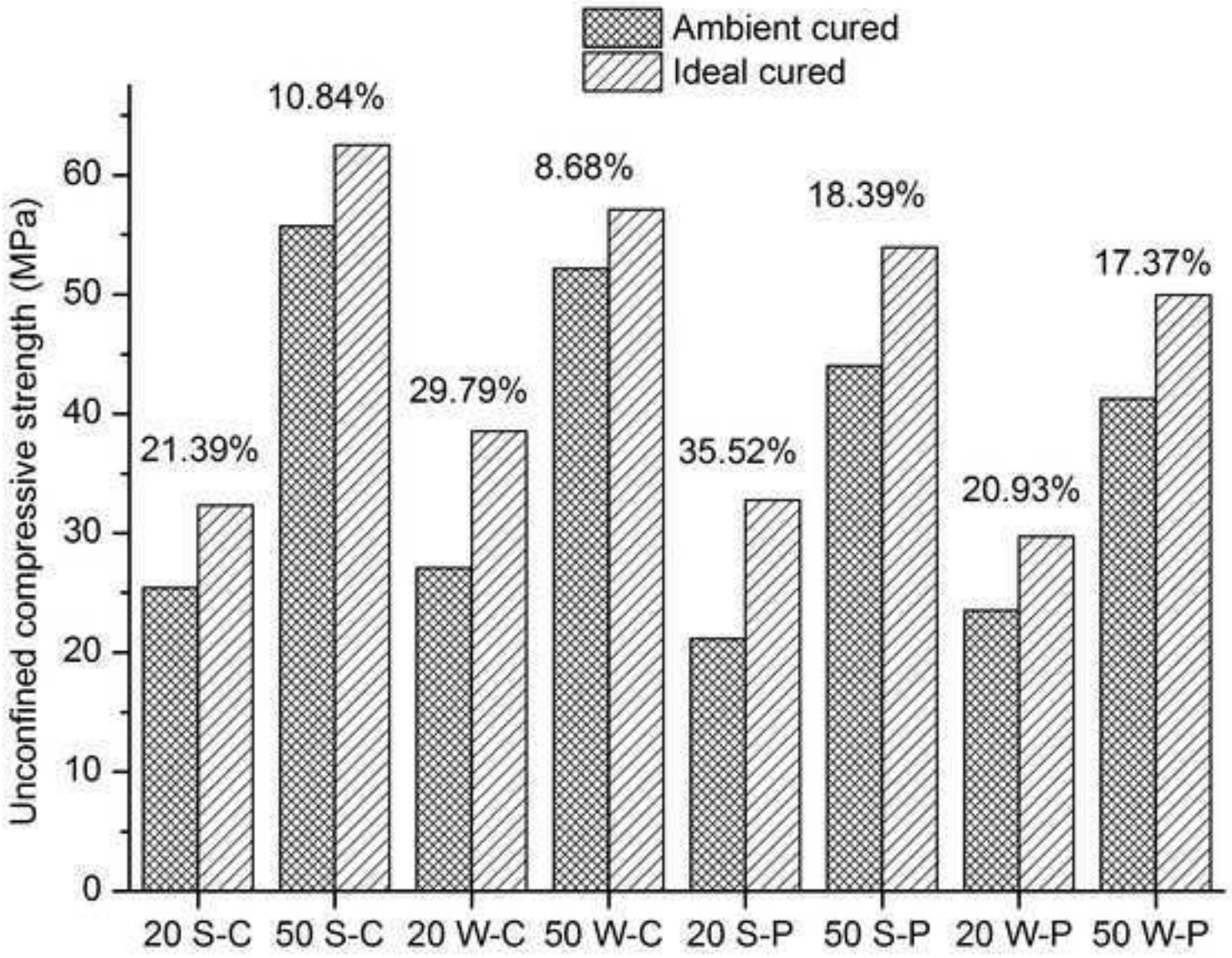

Ambient cured
Ideal cured
Unconfined compressive strength (MPa)
0
10
20
30
40
50
60
21.39%
10.84%
29.79%
8.68%
35.52%
18.39%
20.93%
17.37%
20 S-C
50 S-C
20 W-C
50 W-C
20 S-P
50 S-P
20 W-P
50 W-P

Figure 6

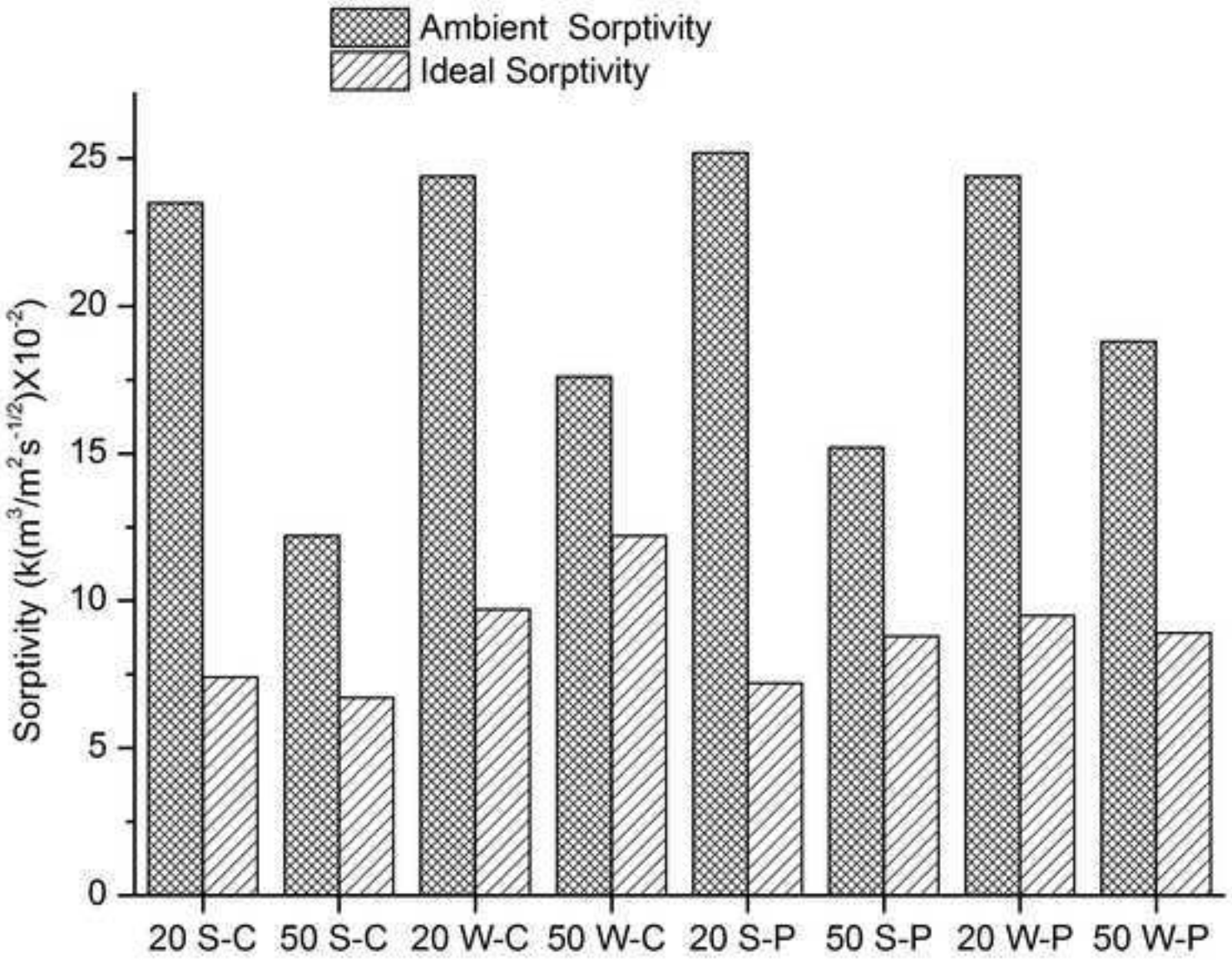

Ambient Sorptivity
Ideal Sorptivity
Sorptivity (k(m³/m²s⁻¹/²)X10⁻²)
0
5
10
15
20
25
20 S-C
50 S-C
20 W-C
50 W-C
20 S-P
50 S-P
20 W-P
50 W-P

Figure 7

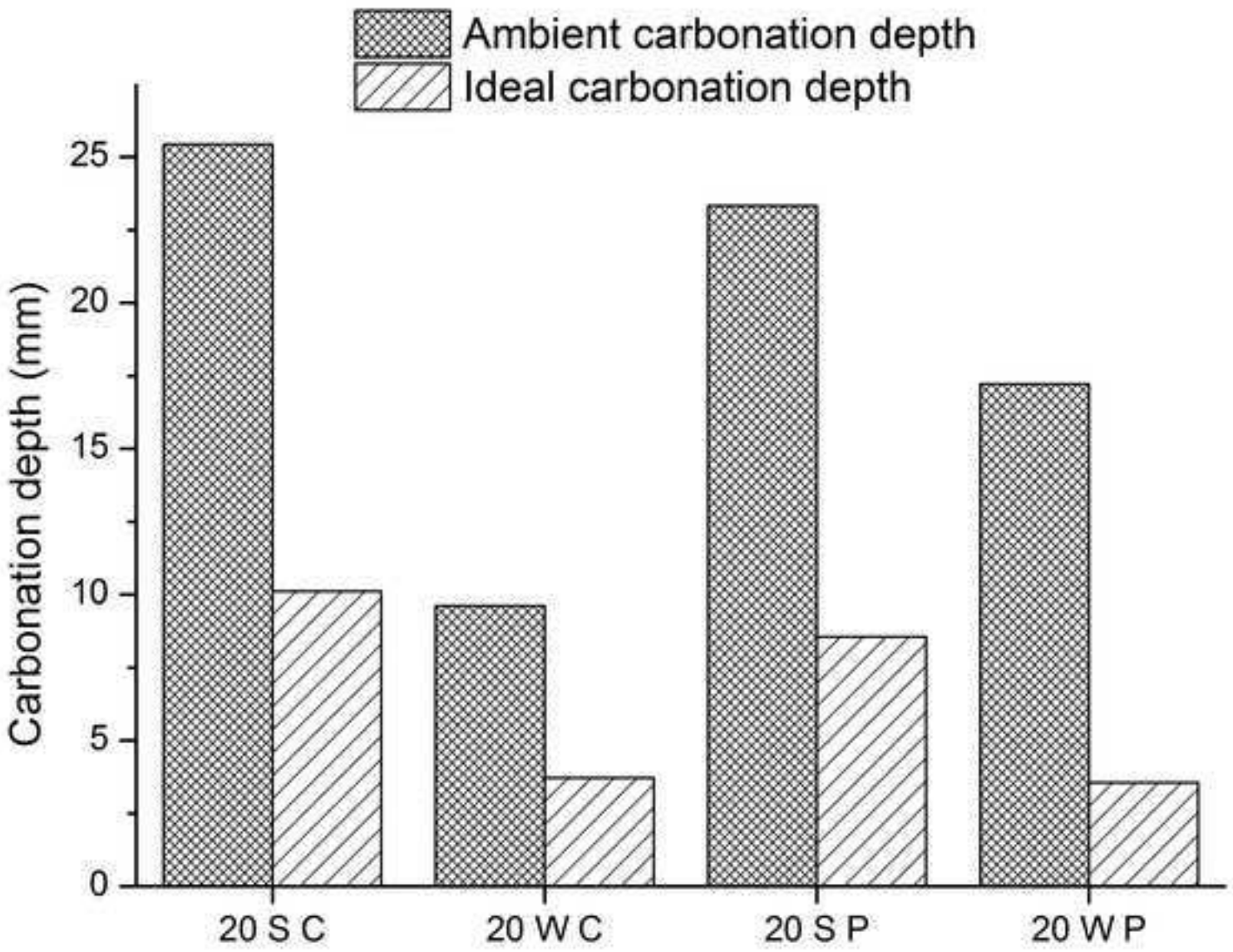

Ambient carbonation depth
Ideal carbonation depth
Carbonation depth (mm)
0
5
10
15
20
25
20 S C
20 W C
20 S P
20 W P

Fig 8a

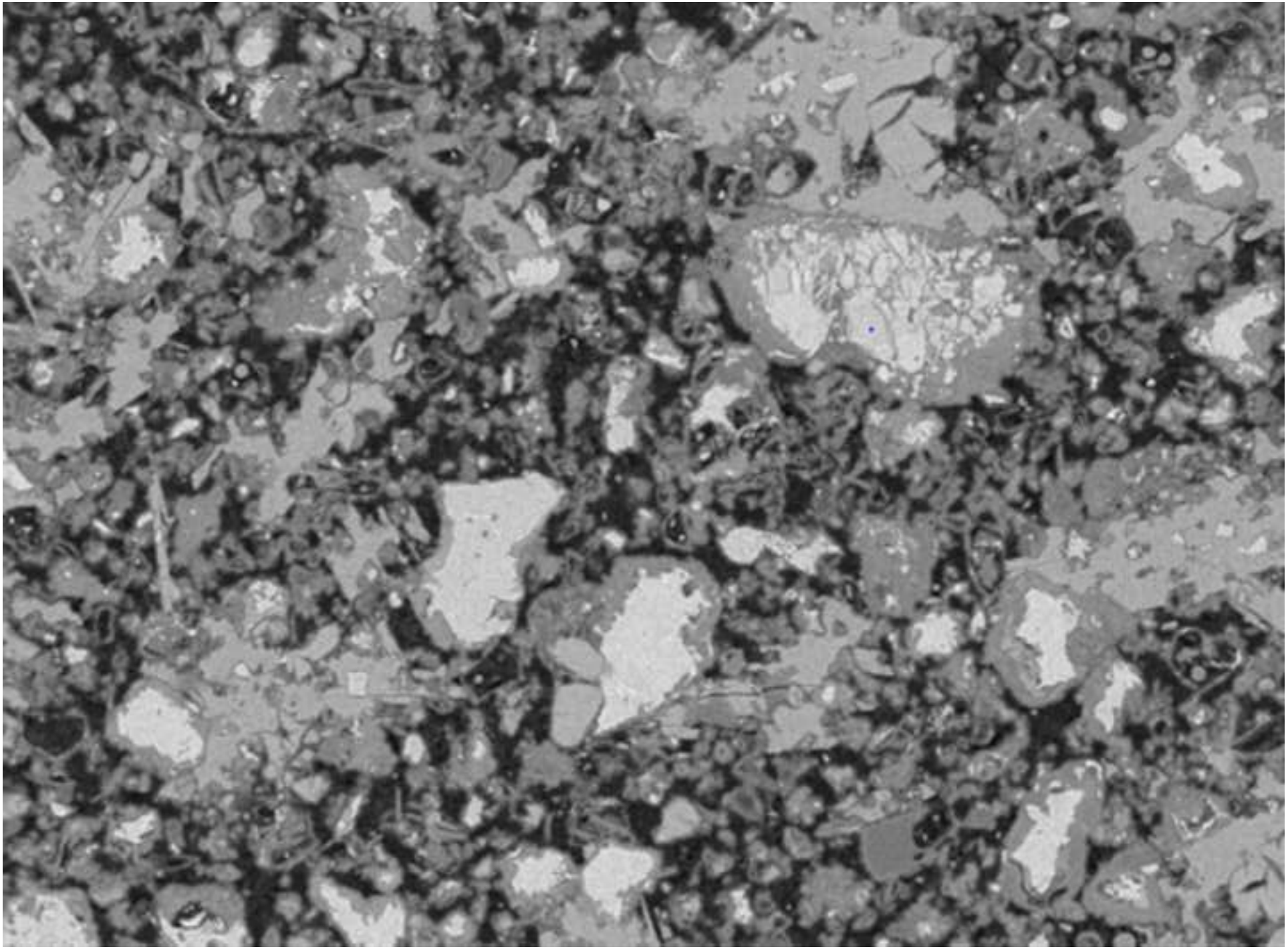


Fig 8b

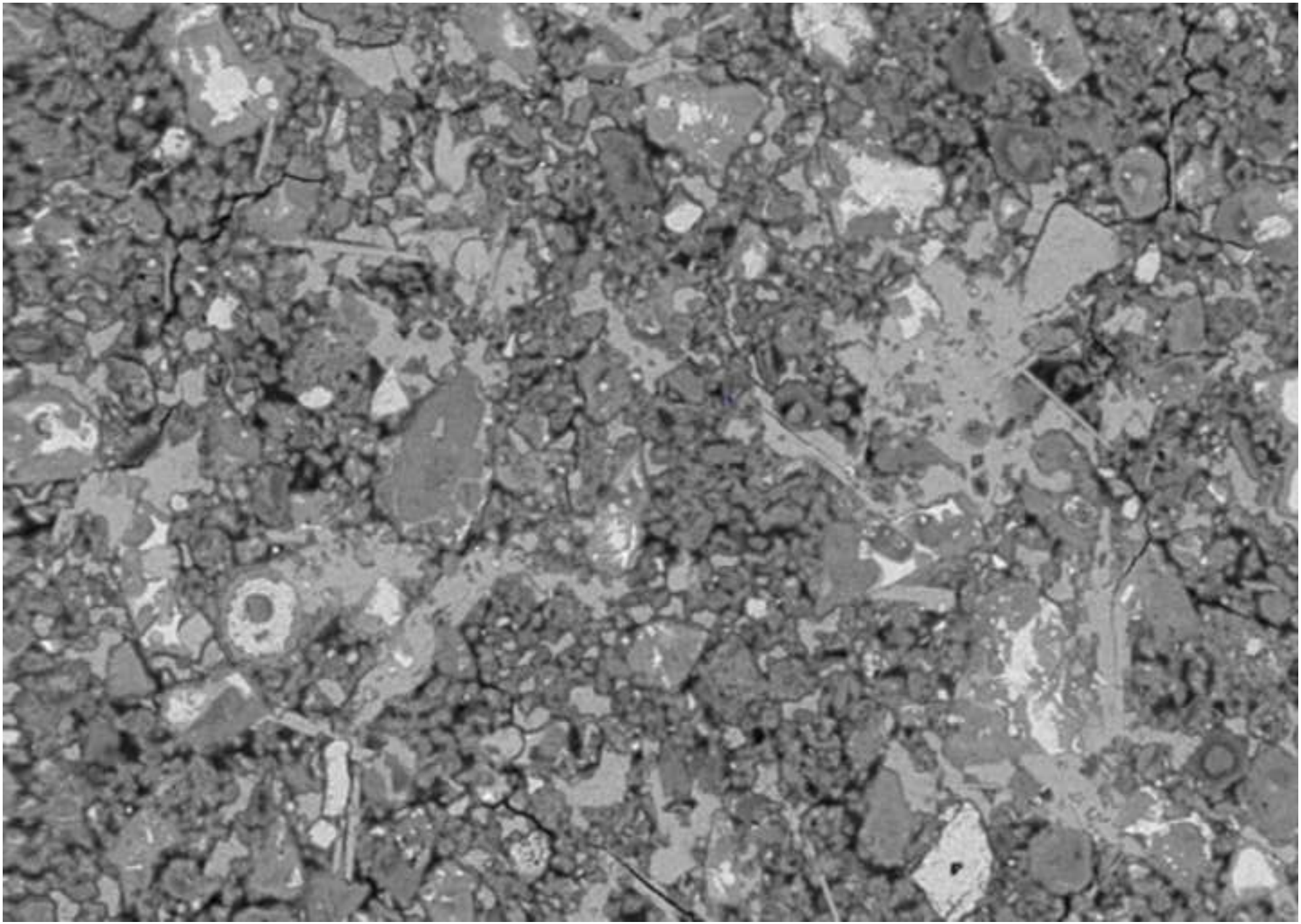


Fig 8c

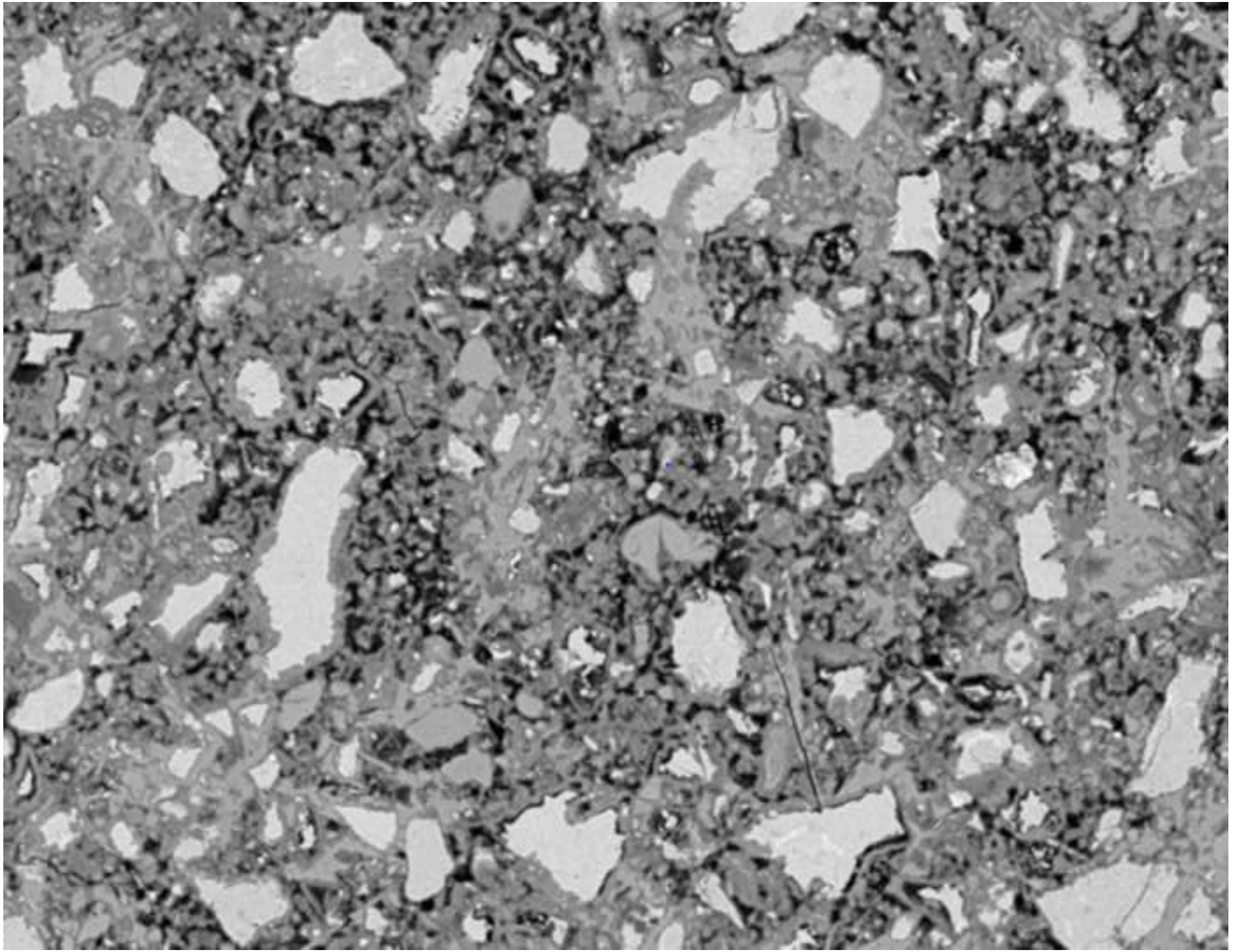


Fig 8d

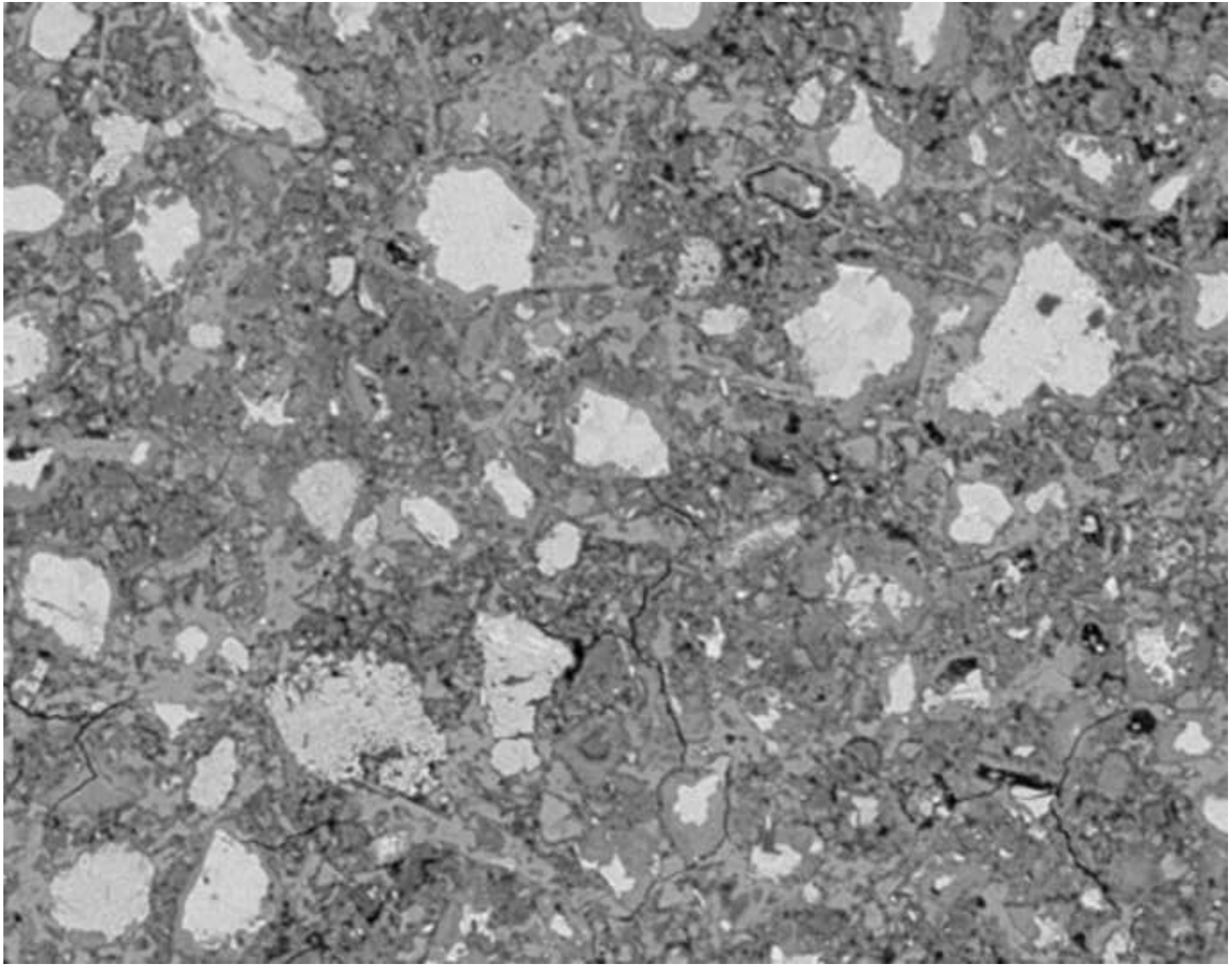


Fig 9a

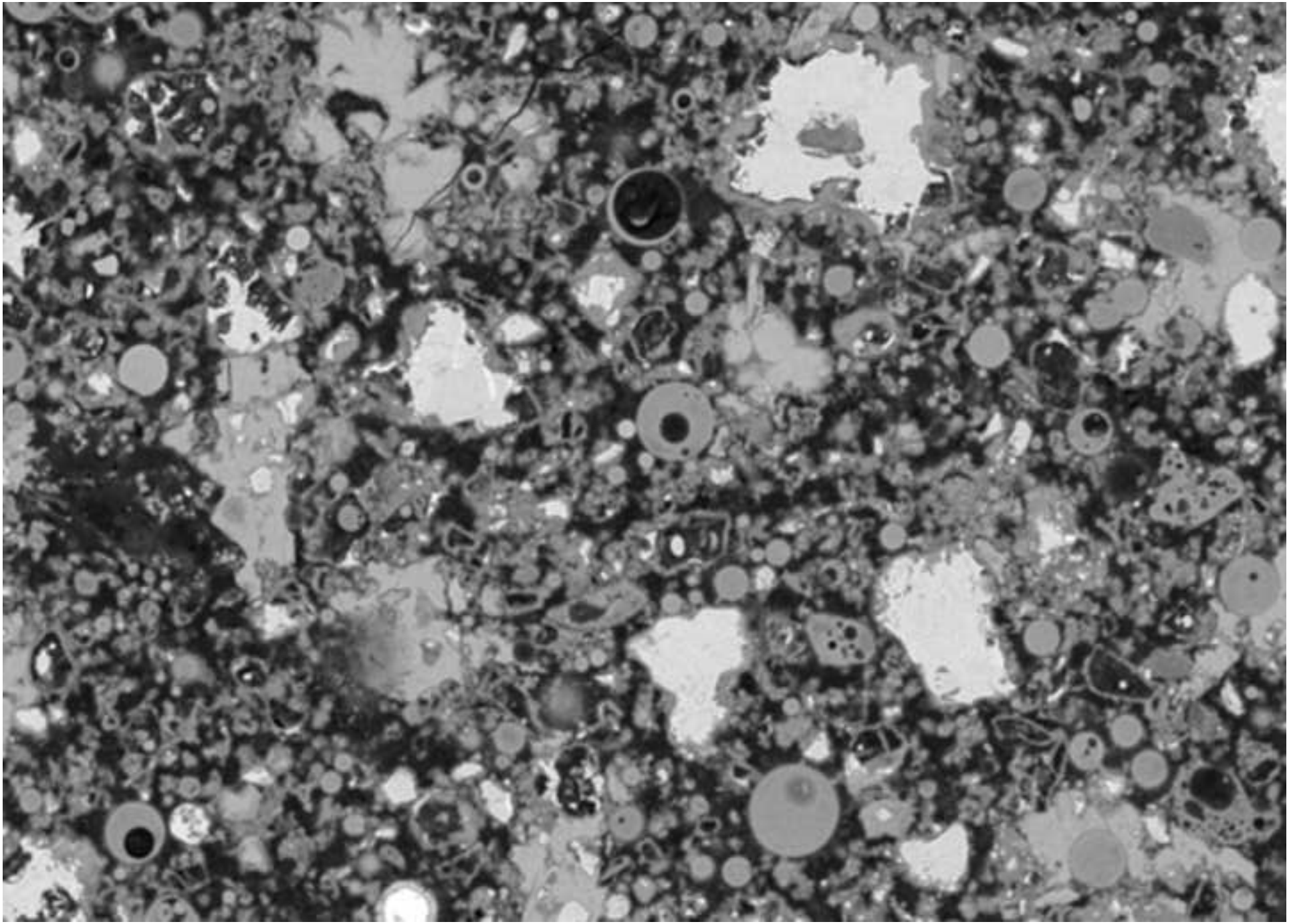


Fig 9b

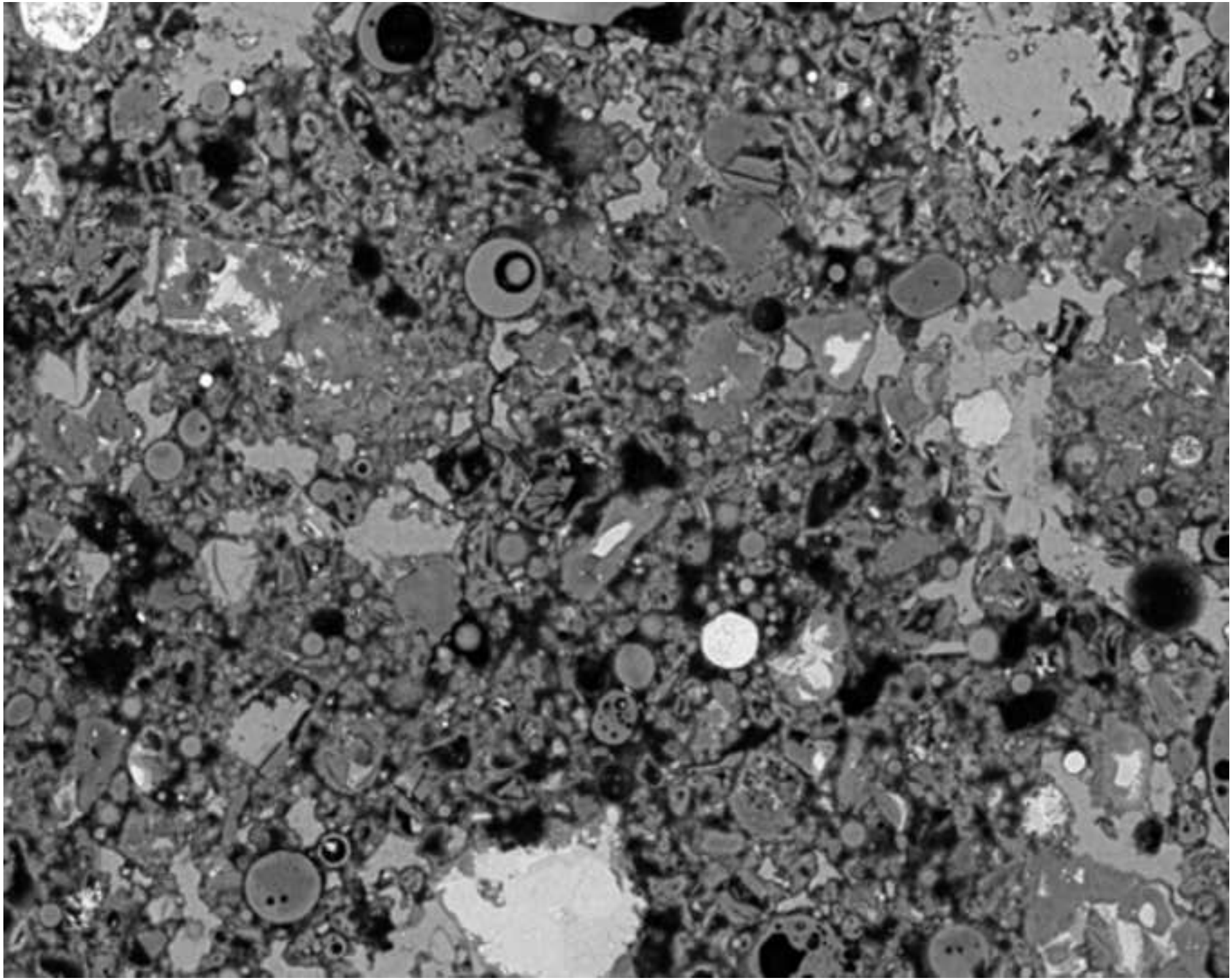


Fig 9c

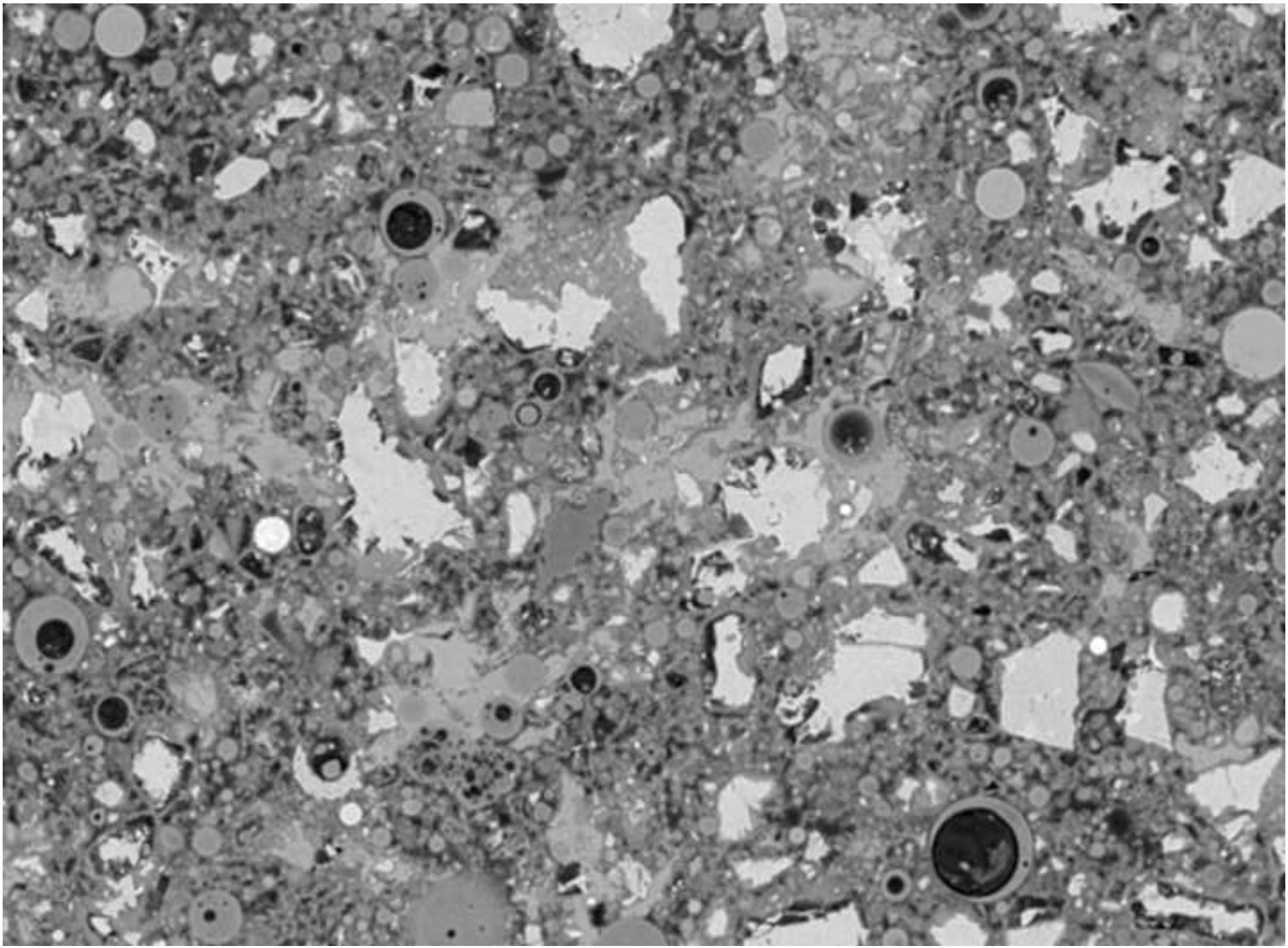


Fig 9d

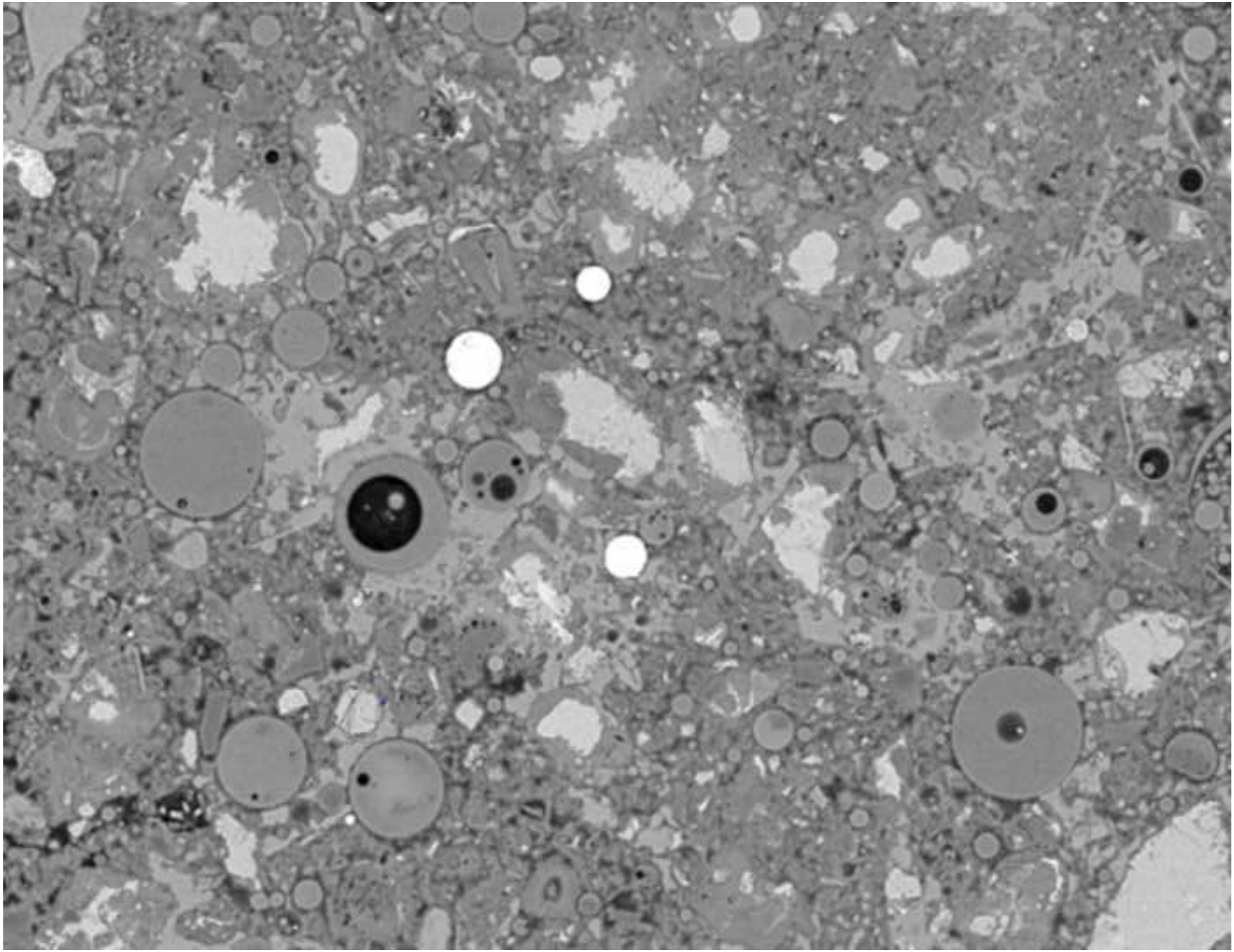


Figure 10

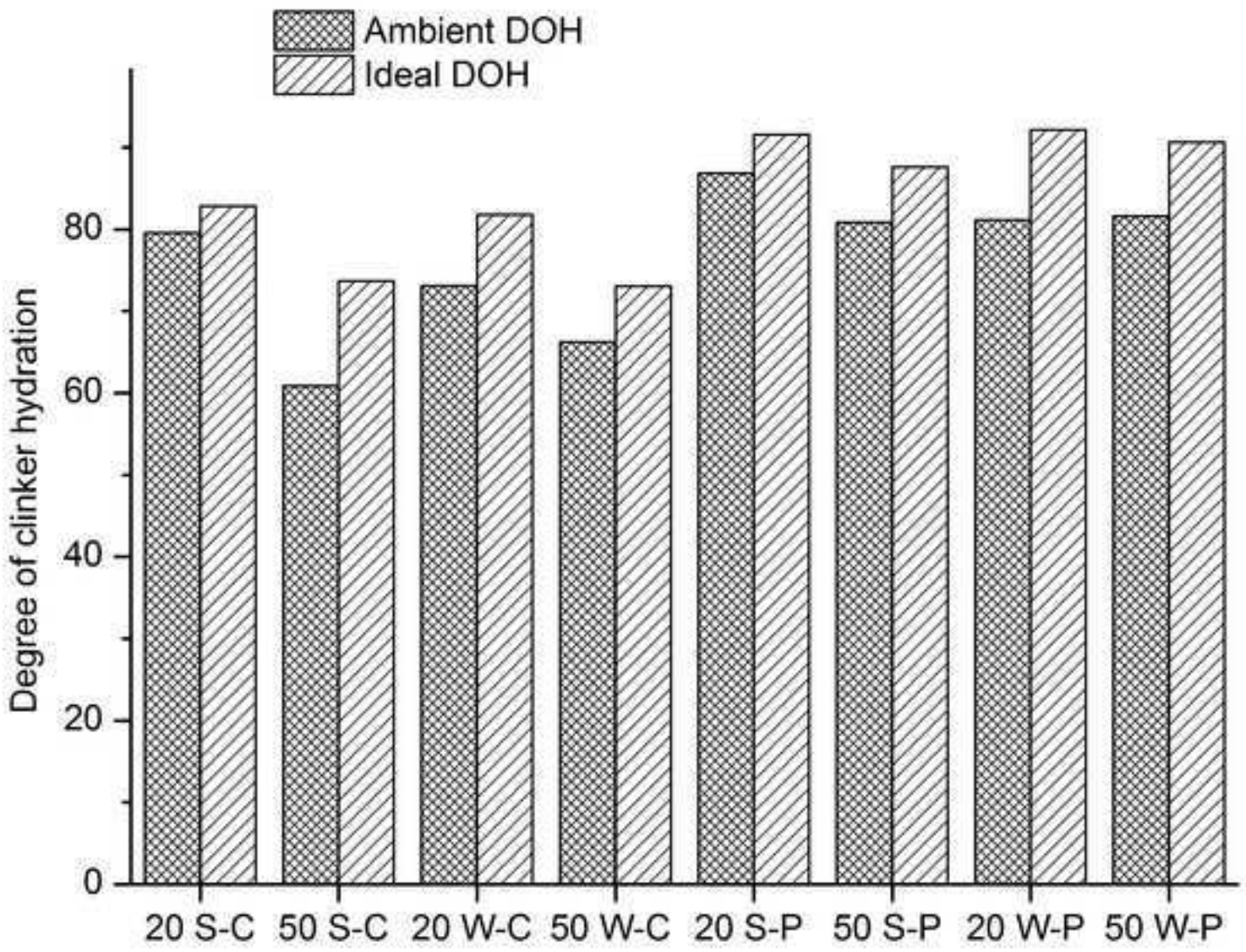

Ambient DOH
Ideal DOH
Degree of clinker hydration
0
20
40
60
80
20 S-C
50 S-C
20 W-C
50 W-C
20 S-P
50 S-P
20 W-P
50 W-P

Figure 11

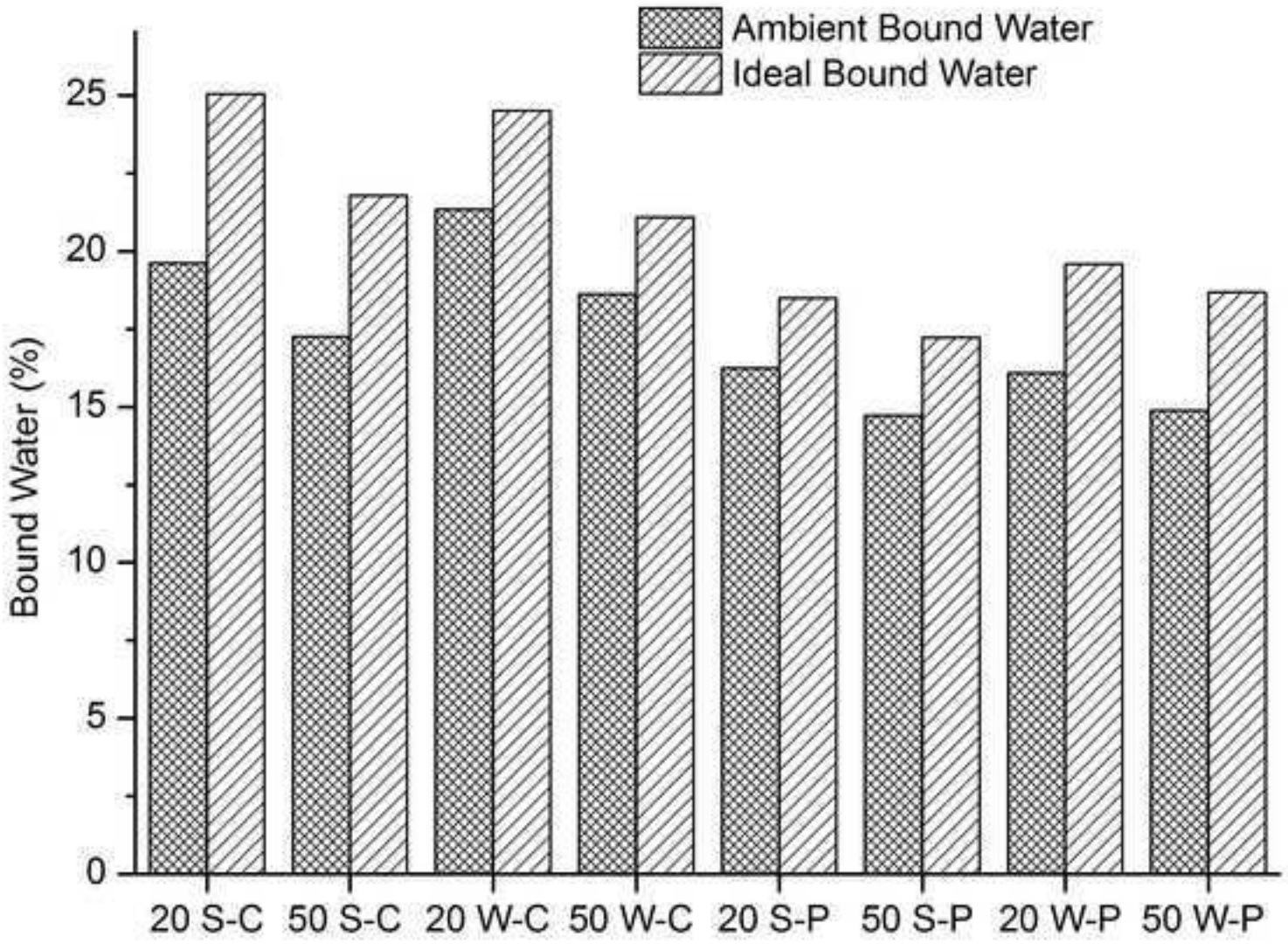

Ambient Bound Water
Ideal Bound Water
Bound Water (%)
0
5
10
15
20
25
20 S-C
50 S-C
20 W-C
50 W-C
20 S-P
50 S-P
20 W-P
50 W-P

Figure 12

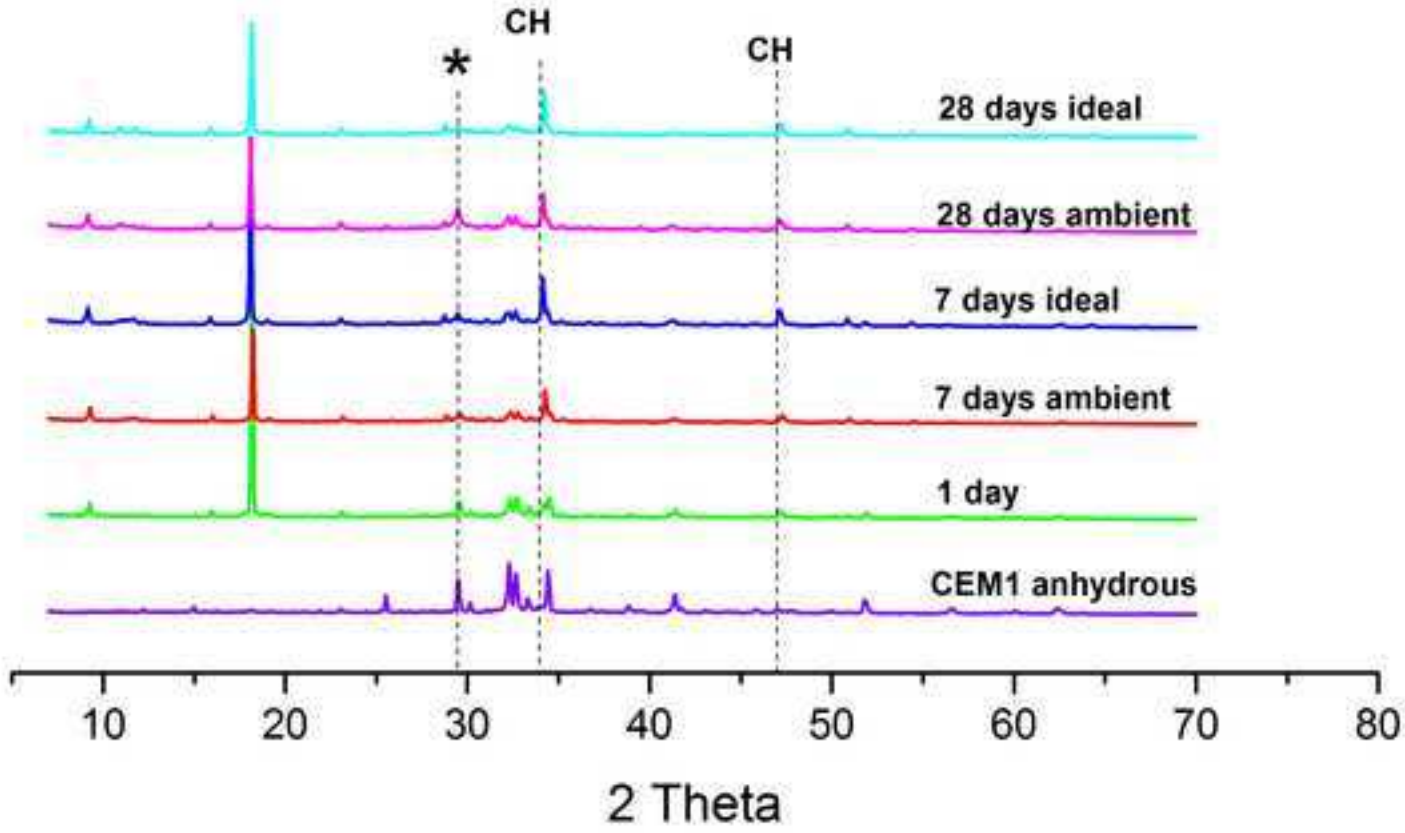
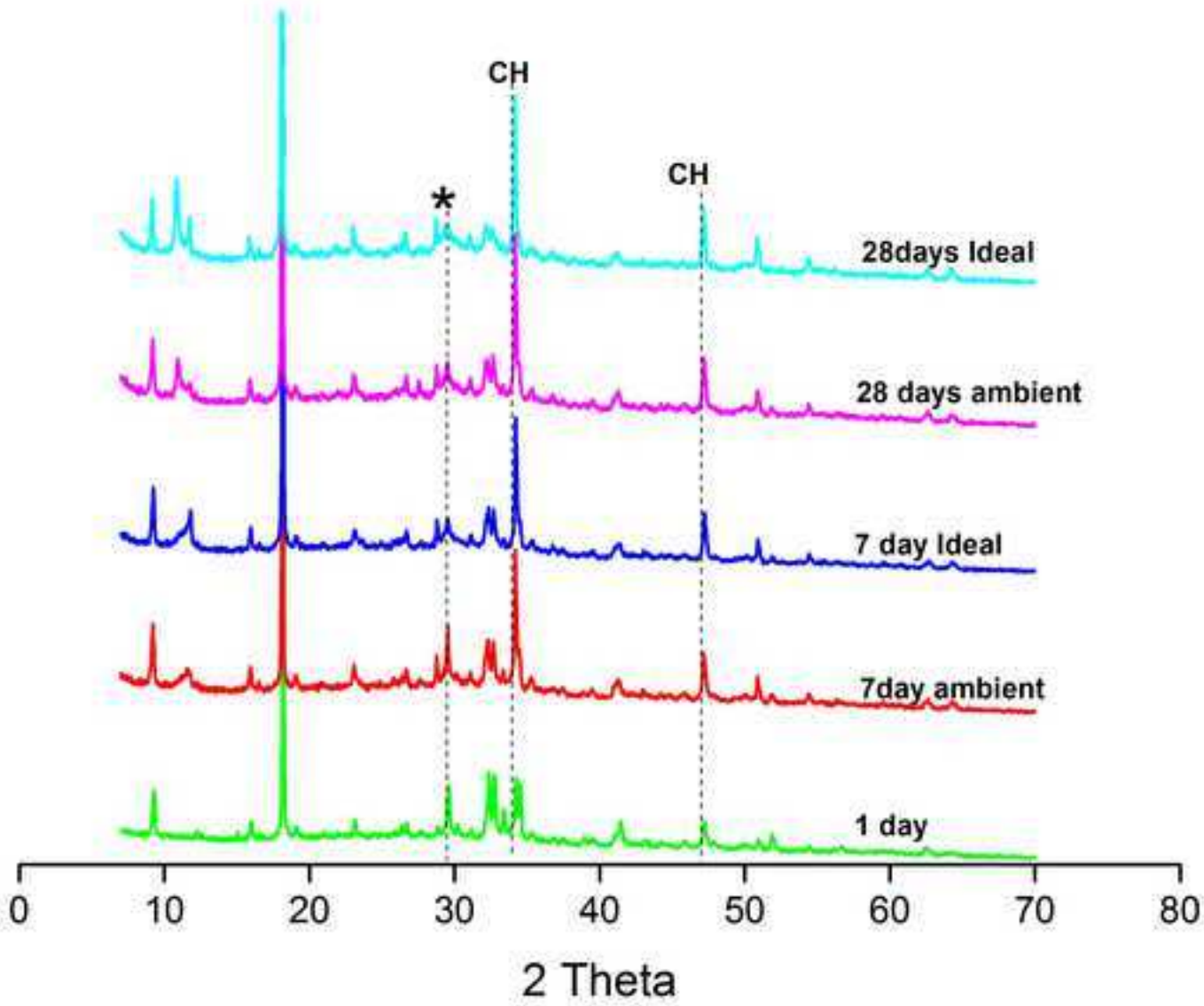

CH
*
CH
28 days ideal
28 days ambient
7 days ideal
7 days ambient
1 day
CEM1 anhydrous
10
20
30
40
50
60
70
80
2 Theta

Figure 13


CH
CH
*
28days Ideal
28 days ambient
7 day Ideal
7day ambient
1 day
0
10
20
30
40
50
60
70
80
2 Theta