

Manuscript Number:

Title: Soil Stabilisation using Alkaline Activation of Fly Ash for Self Compacting Rammed Earth Construction

Article Type: Research Paper

Keywords: Soil Stabilisation; Alkaline Activation; Fly Ash; Rammed Earth

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Abstract: This paper studies the effectiveness of alkaline activation of low-calcium fly ash on the improvement of residual granitic soils to be used on rammed-earth construction. Different liquid:solid ratios, alkali concentrations and Na₂O : ash ratios were tested. Effect of calcium hidroxide, sodium chloride and concrete superplasticiser is also reported. Compressive strength up to 7 days at 60°C was determined. Results show that, in terms of mechanical strength, there is an optimum value for the activator:solids ratio and the alkali concentration, and that a decrease in the Na₂O:ash ratio results in strength increase. No improvement was observed with the sodium chloride or the superplasticiser, while the calcium produced only a short term increase in strength. SEM/EDS analysis were used to analyse microstructural development, showing that strength is fairly related to the Si:Al and Na:Si ratios.

Research highlights

- Low calcium fly ash activated with a sodium-based solution.
- No significant improvement with sodium chloride or superplasticiser.
- There is an optimum liquid : solid ratio and alkali concentration for strength development.
- An alkali : ash ratio reduction results in a significant strength increase.
- Microstructure is significantly affected and can be related to strength variation.

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February 2012

Soil Stabilisation using Alkaline Activation of Fly Ash for Self Compacting Rammed Earth Construction

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Abstract

This paper studies the effectiveness of alkaline activation of low-calcium fly ash on the improvement of residual granitic soils to be used on rammed-earth construction. Different liquid:solid ratios, alkali concentrations and Na₂O : ash ratios were tested. Effect of calcium hydroxide, sodium chloride and concrete superplasticiser is also reported. Compressive strength up to 7 days at 60°C was determined. Results show that, in terms of mechanical strength, there is an optimum value for the activator:solids ratio and the alkali concentration, and that a decrease in the Na₂O:ash ratio results in strength increase. No improvement was observed with the sodium chloride or the superplasticiser, while the calcium produced only a short term increase in strength. SEM/EDS analysis were used to analyse microstructural development, showing that strength is fairly related to the Si:Al and Na:Si ratios.

Keywords: Soil Stabilisation; Alkaline Activation; Fly Ash; Rammed Earth

1. Introduction

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5 Soil is certainly one of the most ancient building materials. The most common earth
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7 construction techniques are rammed-earth and adobe masonry [1]. Adobes are bricks prepared
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9 with moist earth (to which straw is added sometimes) by simply moulding it in a wooden
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11 frame and drying it out in the sun. Then the masonry is prepared by ‘gluing’ the adobes with
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13 earth mortar. A rammed earth wall is made by compacting thin layers of packed earth using a
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15 rammer. The development of modern construction materials gradually made the application of
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17 earth as a construction material less and less attractive, which had more to do with social than
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19 with technical disadvantages. Inclusively, this trend had a significant influence on the lack of
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21 production of specific regulation codes. However, the recent growing concern with a
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23 meaningful and sustainable development is a serious motivation for seeking environmentally
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25 friendly construction materials. Also, a curious change of mentality occurred, since what was
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27 until recently a social embarrassment is now seen as a very modern material, capable of
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29 producing comfortable and visually impressive buildings. Rammed earth walls are relatively
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31 thick (0.5 to 0.9 m), and the dwellings are usually one or two stories high, in order to limit the
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33 compression stresses. That is because these walls have a reduced compressive strength when
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35 compared with other building materials, which currently causes serious limitations in terms of
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37 architectural and structural design.
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48 One of the major techniques used to overcome the problems created by under-performing
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50 soils is the mixing with a cementitious binder. Usually these binders are cement and/or lime,
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52 which glue the soil particles together through chemical reactions. In the case of cement, the
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54 reactions are mainly hydraulic, while with lime they are pozzolanic. This means that cement
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56 needs only water to react and increase in strength; while lime needs water and a pozzolanic
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58 material, like soil. Both binders share the fact that their reactions with water depend largely
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1 on their specific surface. Moreover, although the type of reaction is different for lime and
2 cement, the final product is very much alike, based on calcium and silicium compounds. In
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4 terms of mechanical strength, cement-based binders usually deliver significantly better results
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6 than lime-based binders.
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11 Traditionally, the soils used for rammed earth construction have a distinctive clayey
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13 behaviour, since the cohesion plays an important role in the structural stability of the walls,
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15 and it also helps preventing disaggregation of the wall surface. Since the clay fraction is
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17 significant, the chemical improvement of these soils is very often made with lime, due to the
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19 very effective and permanent reactions between the calcium compound and the pozzolanic
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21 soil. Also, although the compressive strength achieved is lower than with cement, it is usually
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23 enough for the purpose of current earth construction designs. However, soils from northern
24
25 Portugal are mostly granitic, with a clay fraction composed mainly by kaolinite in quantities
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27 lower than 10% [2]. Therefore, and since a clay fraction of at least 10% is needed for the soil
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29 to be stabilised with lime [3,4,5], more appropriate binders need to be used. One obvious
30
31 choice would be cement, however, environmental and durability concerns with cement
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33 production and application are increasing rapidly and can no longer be ignored. The
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35 environmental issues related with cement production was – together with strength and
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37 durability concerns – the main motivation to study the application of a geopolymeric binder
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39 (alkaline activation) for the improvement of residual granitic soil to be used in rammed earth
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41 construction. This technology – alkaline activation of fly ash – was already tested for soil
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43 improvement in geotechnical applications [6,7]. The geopolymeric binder is usually obtained
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45 through alkaline activation of waste materials, and its application is rapidly increasing in the
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47 construction industry, not only as a technically sound construction material; but also as an
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49 important contribution for the reduction in cement consumption, since it allows its
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1 substitution in significant percentages, while at the same time uses an industrial by-product,
2 namely fly ash.
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7 In general terms, alkaline activation is a reaction between alumina-silicate materials and alkali
8 or alkali earth substances, namely: ROH , $R(OH)_2$, R_2CO_3 , R_2S , Na_2SO_4 , $CaSO_4 \cdot 2H_2O$,
9 $R_2 \cdot (n)SiO_2$, in which R represents an alkaline ion like sodium (Na^+) or potassium (K^+), or an
10 alkaline earth ion like calcium (Ca^{2+}). It can be described as a polycondensation process, in
11 which the silica (SiO_2) and alumina (AlO_4) tetrahedra interconnect and share the oxygen ions.
12 The process starts when the high hydroxyl (OH^-) concentration of the alkaline medium
13 favours the breaking of the covalent bonds $Si-O-Si$, $Al-O-Al$ and $Al-O-Si$ from the vitreous
14 phase of the raw material, transforming the silica and alumina ions in colloids and releasing
15 them into the solution. The extent of dissolution depends upon the quantities and nature of the
16 alumina and silica sources and the pH levels. In general, minerals with a higher extent of
17 dissolution will result in higher compressive strength after the process is complete. At the
18 same time, the alkaline cations Na^+ , K^+ or Ca^{2+} act like building blocks of the structure,
19 compensating the excess negative charges associated with the modification of the aluminium
20 coordination during the dissolution phase. The resulting products accumulate for a period of
21 time, forming a ion “soup” of high mobility. If calcium is present in the mixture in significant
22 amounts the dissolved $Al-Si$ complex will diffuse from the solid surface and produce a
23 dominant C-S-H gel phase. Otherwise there is the poly-condensation of the dissolved gel with
24 the Si and Al ions precipitating around the nuclei points, sharing all oxygen ions and forming
25 a $Si-O-Al$ and $Si-O-Si$ three-dimensional structure (the formation of $Al-O-Al$ is not favoured),
26 more stable than those existing in the original aluminosilicate source. The resulting polymeric
27 structure of $Al-O-Si$ bonds is the main structure of the new material. Materials formed using
28 reactions between silica and alumina and alkali cations like sodium or potassium are very
29 similar, at a molecular level, with natural rocks, sharing their stiffness, durability and strength.
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2 The aim of this paper is to determine, through a parametric analysis using laboratory tests, the
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4 optimum fly ash – based alkaline activated binder for the improvement of soil to be used in
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6 rammed earth construction. This evaluation was made in terms of mechanical strength of each
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8 mixture after specific curing periods. Different compositions were considered, with the
9
10 following specific objectives:
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- 13 - Effect of maximum particle size.
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- 15 - Influence of hydrated lime, sodium chloride and concrete superplasticisers.
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- 17 - Effect of liquid : solid ratio.
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- 19 - Effect of activator concentration.
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- 21 - Effect of Na₂O : ash ratio.
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- 23 - Relationship between strength and microstructure.
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36 **2. Methodology**

37 *2.1 Material characterization*

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46 Laboratory tests were performed with granitic residual soil recovered near University of
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48 Minho, in Guimarães. It can be considered as representative of the typical soils from the north
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50 of Portugal. After being submitted to characterisation analysis according to BS 1377 Parts 1, 2
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52 & 4, 1990: particle size distribution, Atterberg limits and compaction parameters using
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54 Proctor tests [8,9,10] (Table I) – it was classified as ‘SM – silty sand’ [11]. Additional
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56 characterisation included Scanning Electron Microscopy (SEM) and Energy Dispersive
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58 Spectroscopy (EDS) to determine chemical composition.
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2 Table 1: Soil properties
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9 The fly ash used had low calcium content (class F) and was obtained from a Portuguese
10 thermo-electric plant. Its characterisation was carried by scanning electron microscopy (SEM)
11 and chemical analysis by energy dispersive spectroscopy (EDS) (Table 2). Its total mass
12 available for dissolution is approximately 74% (Si and Al).
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21 Table 2: Chemical composition of the fly ash (wt. %)
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28 The sodium silicate was originally in solution form, with a specific gravity of 1.45 g/cm^3 , a
29 sodium oxide (Na_2O) content of 13% and a $\text{SiO}_2 : \text{Na}_2\text{O}$ ratio of approximately 2 : 1 by mass.
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31 The sodium hydroxide was originally supplied in flake form with a specific gravity of 2.13
32 g/cm^3 at 20°C and 95-99 % purity, and was dissolved in water to achieve different
33 concentrations of 10, 12.5 and 15 molal before being mixed with the silicate in a sodium
34 silicate : sodium hydroxide ratio of 2 : 1. This ratio was previously studied by different
35 authors [12,13,14], who concluded that a value of 2 favours the strength gain.
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48 The sodium chloride (NaCl) and the calcitic hydrated lime ($\text{Ca}(\text{OH})_2$) were both 99% pure.
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50 Regarding the water-reducing admixture, Rheobuild 1000 from BASF was used.
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55 2.2 *Sample preparation and testing procedure* 56 57 58 59 60 61 62 63 64 65

1 The parametric analysis to evaluate the effectiveness of each composition was based on
2 unconfined compressive strength (UCS) tests, performed after a pre-determined curing period
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4 under specific conditions. UCS samples were cylindrical, with 38mm in diameter and 76mm
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6 height, and one result was the average of three tests.
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11 The soil was oven dried and sieve down before being thoroughly mixed with the fly ash and
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13 only then the activator was added and mixed. A kitchen appliance was used for mixing, and
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15 every mixture batch was just enough to form the three samples necessary to produce one
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17 result, in order to avoid excessive delays between mixture preparation and moulding. This
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19 propeller type mixer achieves its purpose due to axial and turbulent flow, and is suitable for
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21 suspensions with viscosities up to 20 Pa.s [15]. After mixing for 3 minutes and cast into the
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23 moulds in three layers, the samples were vibrated by tapping the mould continuously for 25-
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25 30 seconds for each layer, in order to release the trapped air bubbles [16].
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33 Finally, the moulds were covered with a Plexiglas cap and stored in an oven at 60°C for
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35 twelve hours. After this the samples were removed from the moulds and stored back in the
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37 oven (again at 60°C) in a sealed bag up until two hours before trimming and testing. The
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39 curing periods considered were 1, 3 and 7 days. Curing at high temperatures allowed a
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41 significant reduction in the necessary period for significant reactions between the activator
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43 and the fly ash to occur [12]. The value of 60°C was chosen since it has been reported [17]
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45 that curing geopolymers at temperatures higher than 80°C can be detrimental to its properties.
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47 Also, it is inside the interval reported by Bondar et al (2011) [18] as optimum for curing
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49 alkali-activated natural pozzolans.
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57 Specific tests to optimise sample preparation showed that a circular saw can easily cut
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59 through this material without any damage to its structure, and was therefore used to trim the
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1 samples to an approximate length while also creating perfect horizontal plans for the contact
2 with the compression machine plates. A low testing speed (0.30 mm/min) was used to
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4 mitigate any influence of this parameter on the test result.
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11 **3. Results and discussion**
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16 *3.1 Effect of maximum particle size*
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21 Full details of all the mixtures tested are shown in Table 3. A set of UCS tests was performed
22 to evaluate the most suited maximum particle size. According to the New Zealand Standards
23 (NZS 4298:1998) [19], the soil used in earth construction needs to be sieved down if a
24 significant amount of large aggregate particles are present in its composition. Therefore, the
25 question of which would be the most effective maximum particle size was raised. Two
26 different sieves were used: 4.75 (SG4.75) and 6.30 mm (SG6.30). The soils resulting from the
27 sieving process are shown in Figure 1.
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41 Table 3: Identification and composition of the mixtures tested
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53 Figure 1: Particle size distribution of samples sieved down to 4.75 and 6.30 mm
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58 Sample with a maximum particle size of 4.75 mm has a more uniform distribution than that
59 with 6.30 mm, meaning that the latter has a higher uniformity coefficient. In soil mechanics,
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1 this can usually be related to higher mechanical strength, since the more particle sizes
2 available result in a higher density. However, UCS results from Figure 2 show that it is
3 beneficial, in terms of strength gain, to use a finer soil fraction. This can be concluded after
4 the 4.75 mm mixtures produced better UCS values than the 6.30 mm. It should be noted that
5 these results might be influenced by the size of the sample, meaning that in a bigger volume,
6 like a rammed earth wall, the inclusion of the particles between 4.75 and 6.30 mm, or even
7 bigger, might have a positive effect on compressive strength.
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Figure 2: UCS results of alkaline activation mixtures SG4.75 and SG6.30

3.2 *Influence of CaO, NaCl and concrete superplasticisers on strength gain evolution*

31 After setting the best option in terms of maximum particle size, a second set of tests was
32 carried out with the purpose of evaluating the influence of calcium hydroxide ($\text{Ca}(\text{OH})_2$),
33 sodium chloride (NaCl) and a common concrete superplasticiser (CSP).
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36 Calcium silicate hydrate (CSH) is known to be the major component regarding the binding
37 properties of Portland cement, while alkaline activation of low calcium raw materials – like
38 fly ash type F – produces binders resulting from aluminosilicate structures. However, Yip et
39 al (2005) [20] found that it is possible to have geopolymeric and CSH gels forming
40 simultaneously within the same binder. In high concentrations of NaOH (> 7.5 molal), the
41 geopolymeric gel is predominant, with small calcium precipitates scattered across the
42 mixture. The effect of these precipitates – namely an initial strength improvement relatively to
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1 the mixture without calcium – is not observed unless a significant source of calcium
2 hydroxide is available at the initial phase. Hu et al (2009) [21] concluded that adding calcium
3 to the activator accelerates the synthesis of the fly ash-based geopolymers. In the present
4 work, and with the objective of evaluating the effect of additional calcium on strength gain
5 rate, samples with a lime content of 40% of the fly ash – or 10% of the total solids content –
6 were prepared. Higher values could favour the formation of a CSH gel based matrix instead of
7 an aluminosilicate network with CSH gel as a secondary reaction product [22].
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19 Several authors [23,24,25] have previously reported the enhancement of soil – lime
20 stabilisation with sodium chloride (NaCl). Although no information could be found on the
21 effect of such additive on alkaline activation, and the exact chemical reactions that might
22 occur in this case are not fully understood, it was decided to test its effect on the strength gain
23 rate of the mixtures. Specific mixtures were prepared in which 8% of the fly ash mass (2% of
24 the total solids mass) was substituted by NaCl. The optimal NaCl percentage in soil–lime
25 stabilisation is usually between 1 and 2% of the soil mass [23,24,25]. Higher values increase
26 the Na⁺ ions in the solution which in turn increase the double layer around the soil particles.
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41 Previous research (personal communication) concluded that the reduction in Na₂O : ash ratio
42 might be beneficial for the strength gain process. Therefore, a superplasticiser was used in
43 some mixtures to try to reduce the liquid phase necessary for mixture homogenisation. The
44 capacity of these additives to lower the water demand might compensate some strength
45 reduction, previously reported by Kong & Sanjayan (2010) [26]. Some samples were prepared
46 with the ash partially substituted by a commercial superplasticiser. Hardjito & Rangan (2005)
47 [14] concluded that the use of superplasticiser beyond 2% of the fly ash mass will likely cause
48 deterioration in compressive strengths. Sathonsaowaphak et al (2009) [27] also reported a
49 decrease in strength but only for superplasticiser contents higher than 6% of the fly ash. A
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1 value of 6% was therefore used in this study. Based on previous experience, the reduction in
2 the liquid : solids ratio due to the CSP, relatively to the remaining mixtures, was
3 approximately 7%. During sample preparation, and based on the homogenisation level of the
4 mixtures, it was discover that this reduction could have been more pronounced, and therefore
5 optimisation of the liquid : solids ratio, when using a superplasticiser for workability increase,
6 will be carefully addressed in a future study.
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17 Results from Figure 3, comparing strength gain of the mixtures with quicklime, salt and
18 superplasticiser, show that mixtures $SGCa(OH)_2$ presented a significant short term
19 improvement, but technically no further improvement could be observed after one day curing.
20 This behaviour is common in calcium-based systems. Winnefield et al (2010) [17], when
21 studying the compressive strength behaviour of high and low calcium fly ash, reported a
22 similar behaviour for both types of ash analysed, i.e. short-term improvement (at two days
23 curing) followed by no improvement over the next 28 days. However, the curing temperature
24 used was 80°C, which might explain why the UCS of the low calcium fly ash geopolymers –
25 although improving significantly more than the high calcium geopolymers after two days –
26 did not improve beyond that period. The higher curing period probably accelerated the
27 reactions to such an extent that the observed behaviour during the present study also occurred
28 in the research by Winnefield et al (2010) [17], but it all happened in the first two days of
29 curing.
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58 Figure 3: UCS results of alkaline activation mixtures with NaCl, CaO and CSP additives
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1 Using the values obtained with the mixtures SG4.75 as a reference, NaCl and CSP additives
2 had, in general, a negative effect on mechanical strength (Figure 3). The effect of these
3 additives was significantly different from that obtained with the Ca(OH)₂, since they showed
4 a lower short term improvement but a very significant strength gain at 7 days curing when
5 compared with the SGCa(OH)₂ mixtures. Relatively to the SG4.75 results, the NaCL mixtures
6 showed significantly lower UCS values than SG4.75 at 1 and 3 days curing, and closer values
7 at 7 days. Therefore, the sodium chloride did not perform the role of catalyser that was
8 observed previously in soil stabilisation with lime also using sodium chloride [23,24,25]. The
9 reduction in the liquid : solid ratio – relatively to the SG4.75 mixtures – achieved with the
10 CSP did not correspond to an increase in performance, since UCS for every curing period was
11 higher for the specimens without the CSP. It could be that a potential increase in strength, by
12 decreasing the Na₂O : ash ratio when adding the CSP, was eliminated by a reaction between
13 the activator and the CSP that reduced the effectiveness of the Na₂O [26]. Further tests are
14 necessary to analyse any potential interaction between the sodium and the superplasticiser.
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36 *3.3 Effect of liquid : solid ratio*

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41 To evaluate the effects of the liquid (activator) : solid (soil + ash) ratio on UCS, additional
42 mixtures were prepared and tested, with liquid : solid ratios of 0.350 (SG1A), 0.400 (SG1B)
43 and 0.425 (SG1C). Results obtained with mixture SG4.75 (0.375) were also included (Figure
44 4). The percentage of fly ash in these additional mixtures was slightly adjusted so that the
45 activator : ash ratio of 1.5 and the Na₂O : ash ratio of 0.257, used for the SG4.75 mixtures,
46 was kept constant. It is apparent, from the results shown in Figure 4, that there is an optimum
47 liquid : solids value, around the 0.375 mark, which results in the highest compressive
48 strength. This might be explained using the dry unit weight – liquid content relationship
49 theory used for soil compaction. According to this theory, water content values higher than
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1 the optimum result in the water occupying the voids that could be filled with solid particles,
2 while lower than optimum values make the compaction process more difficult, and therefore
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4 lower densities are obtained. This last case is even more evident when no compaction energy
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6 is used (in this study the mixtures were poured in the mould and tapped on the table). In fact,
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8 specimens of mixture SG1A were especially difficult to prepare due to its high viscosity. It is
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10 possible that by using some kind of compaction energy, like repeated blows from the mass of
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12 a hammer, higher UCS values are obtained for the mixtures with the lower liquid : solids
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14 ratio, meaning that the optimum ratio is also lower than 0.375. Another possible way to use a
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16 lower liquid : solids ratio, overcoming the workability issues is the use of a superplasticiser,
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18 as previously analysed.
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34 Figure 4: Influence of activator : solid ratio on UCS at 3 and 7 days curing
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38 3.4 *Effect of activator concentration* 39 40 41 42

43 NaOH concentration plays a very significant role in strength development of alkaline
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45 activated systems. An increase in NaOH concentration usually results in strength increase
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47 [21,28]. The effect of the activator concentration was measured by preparing three additional
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49 mixtures: SG2A, SG2B and SG2C, with NaOH concentrations of 7.5, 10.0 and 15.0 molal,
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51 respectively. The results were compared with those obtained with the 12.5 molal mixtures
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53 (SG4.75). Again, the percentage of fly ash in these mixtures was adjusted so that the activator
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55 : solids ratio and the Na₂O : ash ratio were maintained at 0.375 and 0.257, respectively.
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60 Results from Figure 5 show that strength increase was significantly influenced by sodium
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hydroxide concentration. UCS values increased with activator concentration up to 12.5 molal. There is no significant difference between 12.5 and 15 molal, which might be explained by the fact that the 15 molal mixture had very poor workability, and the moulding of these specimens could become very complicated if the process took more than 4 to 5 minutes to be completed. This difficulty should be considered when designing field applications. An optimum concentration of 12.5 molal was therefore determined, based not only on UCS values, but also on the cost (12.5 molal mixtures are obviously cheaper) and the previously mentioned difficulties in preparing high concentration mixtures (15 molal). Explanation for this optimum value is probably related with the fact that immediately after mixing the ash with the activator the vitreous phase from the ash starts to dissolve. The more alkali is available in the mixture, the higher the speed of dissolution and less time is available for the gel to grow into a well – crystallise structure [29].

Figure 5: Influence of activator concentration on UCS

However, the initial faster dissolution in 15 molal specimens is probably responsible for its better performance at short term curing (Figure 6). Since the faster formation of the polymeric matrix significantly decreases further dissolution of the raw silica, and since for longer curing periods the 12.5 molal concentration specimens had time to develop an effective crystallise structure, the better performance of the 15 molal specimens at short term curing cannot be sustained for longer curing periods (3 and 7 days).

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7 Figure 6: Influence of activator concentration on UCS after 1 day curing
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12 *3.5 Effect of Na₂O : ash ratio*
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16 Another set of tests focused on the influence of the Na₂O : ash ratio on unconfined
17 compressive strength (Figure 7). This was achieved by preparing mixtures with Na₂O : ash
18 ratios of 0.428 (SG3A), 0.321 (SG3B) and 0.214 (SG3C), to be compared with the 0.257 ratio
19 from mixtures SG4.75. Other authors used smaller ratios [13,17] since the only dry mass
20 available was the fly ash, while in this case the liquid phase had to be enough for
21 homogenisation of the ash and the soil. Based on the results from Figure 7, it is possible to
22 conclude that the lower the Na₂O : ash ratio the higher the strength of the mixture, for both 3
23 and 7 days curing. Therefore, the maximum UCS was obtained for the lowest ratio of 0.214,
24 and the 7 days curing result for the SG3C mixture achieved the highest overall strength during
25 this study. This ratio could be used as a guideline for composition design, based on
26 mechanical performance requirements. To obtain mixtures with even lower ratios it is
27 necessary to reduce the alkali level or increase the ash content. For the first case two
28 possibilities are raised: reducing the alkali concentration in the activator, which results in a
29 strength decrease (Section 3.4), or reducing the activator (or liquid) content, which might
30 imply the use of some kind of compaction energy to avoid a significant reduction in strength
31 (Section 3.2). The second case (increasing the ash content), might be a better option, since it
32 only implies a change in the ash / soil balance.
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7 Figure 7: Influence of Na₂O / fly ash ratio on UCS evolution
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11 *3.6 SEM / EDS analysis*
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16 The different mechanical behaviour evidenced by the mixtures tested is strongly related with
17 the microstructure of the binding agent. Figures 8 through 10 represent a set of SEM images
18 showing the morphology of the most relevant mixtures in terms of composition and reaction
19 evolution. This morphology analysis is useful to understand the reactions that occur during
20 the strength gain process.
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31 SEM images of mixtures SG4.75 at 1, 3 and 7 days curing are shown in Figure 8 (8a through
32 8d). From this set it is clear the evolution from compact or hollowed spheres of different size
33 – but with regular and smooth surfaces – to a system in which the amorphous aluminosilicate
34 gel [30] is predominant. As this gel develops, it increases the strength of the mixture by not
35 only acting as a bonding agent but also by filling the pore space. Comparing Figures 8a, 8b
36 and 8d it is clear the reduction in pore volume. A similar reduction in void space was also
37 reported in Horpibulsuk et al (2010) [31], when studying the microstructure development of
38 cement-stabilised clay. In all images are visible the spaces previously occupied by round fly
39 ash particles, which were tear when the samples were created. Although unreacted spherical
40 particles are still visible after 7 days curing, they seem to be hollow after being partially
41 dissolved during the reaction process (Point 1 in Figure 8d). The described evolution
42 represents the progressive dissolution of the raw fly ash by the activator. Figure 8c shows
43 deposits of small sodium crystals (Area A) that did not yet reacted with the SiO₂.
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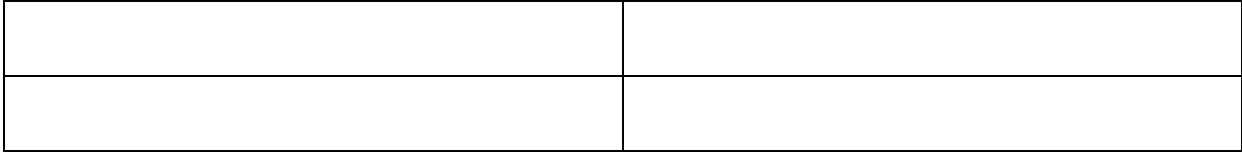


Figure 8: SEM images of mixture SG4.75 after 1 (8a), 3 (8b and 8c) and 7 (8d) days curing

Images in Figure 9 show the microstructure of mixtures SGN_aCl (a) and SGCSP (b), after 7 days curing. In both cases aluminosilicate gel can be seen surrounding original soil particles (Area B), but ash spheres with just a few small dissolution surface points indicate that a significant reaction did not occur, which might explain the lower strength levels achieved by these mixtures regarding the reference SG4.75 mixture. These results confirm that the sodium chloride and the superplasticiser did not have the expected effect of accelerating and improving strength gain.



Figure 9: SEM images of mixture SGN_aCl (9a) and SGCAP (9b) after 7 days curing

Figure 10 correspond to SEM images of mixtures SG1A (a) and SG3C (b), after 7 days curing. Mixture SG1A was prepared with the lowest activator : solids ratio used in this study, while SG3C mixture was selected for microstructural analysis because its Na₂O : ash ratio was the lowest that of all the mixtures in this study (Table 3). From Figure 10a, it appears that a reduced liquid (activator) content resulted in a small pore volume. Also, the significant gel

matrix development showed in Figure 10b is probably responsible for the high compressive strength achieved by this mixture.



Figure 10: SEM images of mixture SG1A (10a) and SG3C (10b) after 7 days curing

EDS analysis of the mentioned areas is presented in Table 4. The Si : Al ratio decreased with curing period for the mixture SG4.75. This was expected since the aluminium is continuously released into the aluminosilicate gel matrix with the fly ash dissolution [17]. The Na : Si ratio also decreased with curing time (SG4.75), reflecting an increase of the alkalis intake in the reaction products. Overall, it is possible to detect a trend between compressive strength and the Si : Al (Figure 11 left) and Na : Si (Figure 11 right) ratios. Also significant is the narrow interval for the Na : Al ratio, showing the high dependency between the alkali and the aluminium, which is explained by the role of the Na⁺ ions on the alkaline activation reaction, compensating the excess negative charges resulting from the progressive changes in the aluminium : oxygen coordination.

Table 4: EDS analysis of the mixtures from Figures 8 through 10

Label	Curing	Na wt %	Al wt %	Si wt %	K wt %	Ca wt %	Fe wt %	Si:Al	Na : Si	Na:Al
SG4.75	1 d	21.7	24.7	41.5	2.4	1.8	5.3	1.68	0.52	0.88
SG4.75	3 d	18.4	21.5	42.7	3.1	2.6	8.7	1.99	0.43	0.86
SG4.75	7 d	15.6	15.4	55.2	3.8	1.7	5.2	3.58	0.28	1.01
SGNaCl	7 d	19.1	18.7	37.1	3.6	2.0	11.7	1.98	0.51	1.02
SGCSP	7 d	19.4	18.4	48.0	2.3	2.3	5.8	2.61	0.40	1.05
SG1A	7 d	18.3	22.4	42.9	2.8	2.6	7.6	1.92	0.43	0.82
SG3C	7 d	15.4	14.5	57.6	2.9	1.6	5.2	3.97	0.27	1.06



Figure 11: Relation between UCS and Si : Al (left) and Na : Si (right) ratios

4. Conclusions

This study clarified some issues regarding the application of alkaline activation of fly ash to soil stabilisation. This technique, not yet fully understood and developed, poses additional complications when used with soil, since the soil brings an additional set of intrinsic relations and delicate compromises. However, it is possible, based on the research here presented, to detect what might be the most influencing factors. It is clear that the Na₂O : ash ratio plays a decisive role in the development of unconfined compressive strength. This ratio should be minimised to obtain maximum strength values. Alkali concentration is also another important factor to consider, especially since a compromise with the liquid : solid ratio needs to be achieved. This is because the optimum value for the former (lower levels of water to increase concentration) will probably not coincide with the optimum value for the latter (higher levels of liquid to improve workability and homogenisation). The maximum particle size of the soil should be minimised prior to mixing with the ash, although some further analysis is necessary to conclude if that is a general rule for every type of soil and if it has a minimum value. The additives used did not seem to justify the time, trouble and money spent, since the strength results obtained were always inferior to those achieved without any additive. However, if time is an issue, the use of hydrated calcium might be a solution, although in that case maximum strength will be compromised. Finally, microstructure of the mixture can be related to

1 strength development, with a clear relationship between UCS and the Si : Al and Na : Si
2 ratios.
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Table 1: Soil properties

Plastic Limit (%)	-
Liquid Limit (%)	33.8
Optimum water content (%)	12.2
Maximum dry density (kN/m ³)	19.2
Clay content (%) [$\phi < 0.002$ mm]	5.7
Silt content (%) [$0.002 < \phi < 0.06$ mm]	14.0
Sand content (%) [$0.06 < \phi < 2$ mm]	45.6
Gravel content (%) [$2 < \phi < 20$ mm]	34.8
D ₁₀ (mm)	0.007
D ₃₀ (mm)	0.179
D ₆₀ (mm)	1.482
Cu	201.9
Cc	2.9

Table 2: Chemical composition of the fly ash (wt. %)

Si	Al	Fe	Ca	K	Ti	Mg	Others	L.O.I. ^a
49.85	23.55	9.09	7.37	3.07	1.91	1.62	3.54	2.59

^a L.O.I. = Loss on ignition

Table 3: Identification and composition of the mixtures tested

Label	NaOH concent (molal)	Ash / soil / additives (wt%)	Water content (wt%)	Activ content (*) (wt%)	Liq / solid ratio (*)	Activ / ash ratio	Na2O / ash ratio
SG4.75	12.5	25.0 / 75.0 / 0	18.182	27.273	0.375	1.5	0.257
SG6.30	12.5	25.0 / 75.0 / 0	18.182	27.273	0.375	1.5	0.257
SGNaCl	12.5	23.0 / 75.0 / 2	18.182	27.273	0.375	1.630	0.279
SGCa(OH) ₂	12.5	15.0 / 75.0 / 10	18.182	27.273	0.375	2.500	0.428
SGCSP	12.5	23.5 / 75.0 / 1.5	17.284	25.926	0.350	1.489	0.255
SG1A	12.5	23.3 / 76.7 / 0	17.284	25.926	0.350	1.5	0.257
SG1B	12.5	26.6 / 73.4 / 0	19.048	28.571	0.400	1.5	0.257
SG1C	12.5	28.3 / 71.7 / 0	19.883	29.825	0.425	1.5	0.257
SG2A	7.5	21.2 / 78.8 / 0	20.979	27.273	0.375	1.769	0.257
SG2B	10.0	23.2 / 76.8 / 0	19.481	27.273	0.375	1.616	0.257
SG2C	15.0	26.5 / 73.5 / 0	17.045	27.273	0.375	1.415	0.257
SG3A	12.5	15.0 / 85.0 / 0	18.182	27.273	0.375	2.500	0.428
SG3B	12.5	20.0 / 80.0 / 0	18.182	27.273	0.375	1.875	0.321
SG3C	12.5	30.0 / 70.0 / 0	18.182	27.273	0.375	1.250	0.214

* The terms 'activator' and 'liquid' refer to the same material

Table 4: EDS analysis of the mixtures from Figures 8 through 10

Label	Curing	Na wt %	Al wt %	Si wt %	K wt %	Ca wt %	Fe wt %	Si:Al	Na : Si	Na:Al
SG4.75	1 d	21.7	24.7	41.5	2.4	1.8	5.3	1.68	0.52	0.88
SG4.75	3 d	18.4	21.5	42.7	3.1	2.6	8.7	1.99	0.43	0.86
SG4.75	7 d	15.6	15.4	55.2	3.8	1.7	5.2	3.58	0.28	1.01
SGNaCl	7 d	19.1	18.7	37.1	3.6	2.0	11.7	1.98	0.51	1.02
SGCSP	7 d	19.4	18.4	48.0	2.3	2.3	5.8	2.61	0.40	1.05
SG1A	7 d	18.3	22.4	42.9	2.8	2.6	7.6	1.92	0.43	0.82
SG3C	7 d	15.4	14.5	57.6	2.9	1.6	5.2	3.97	0.27	1.06

Figure1
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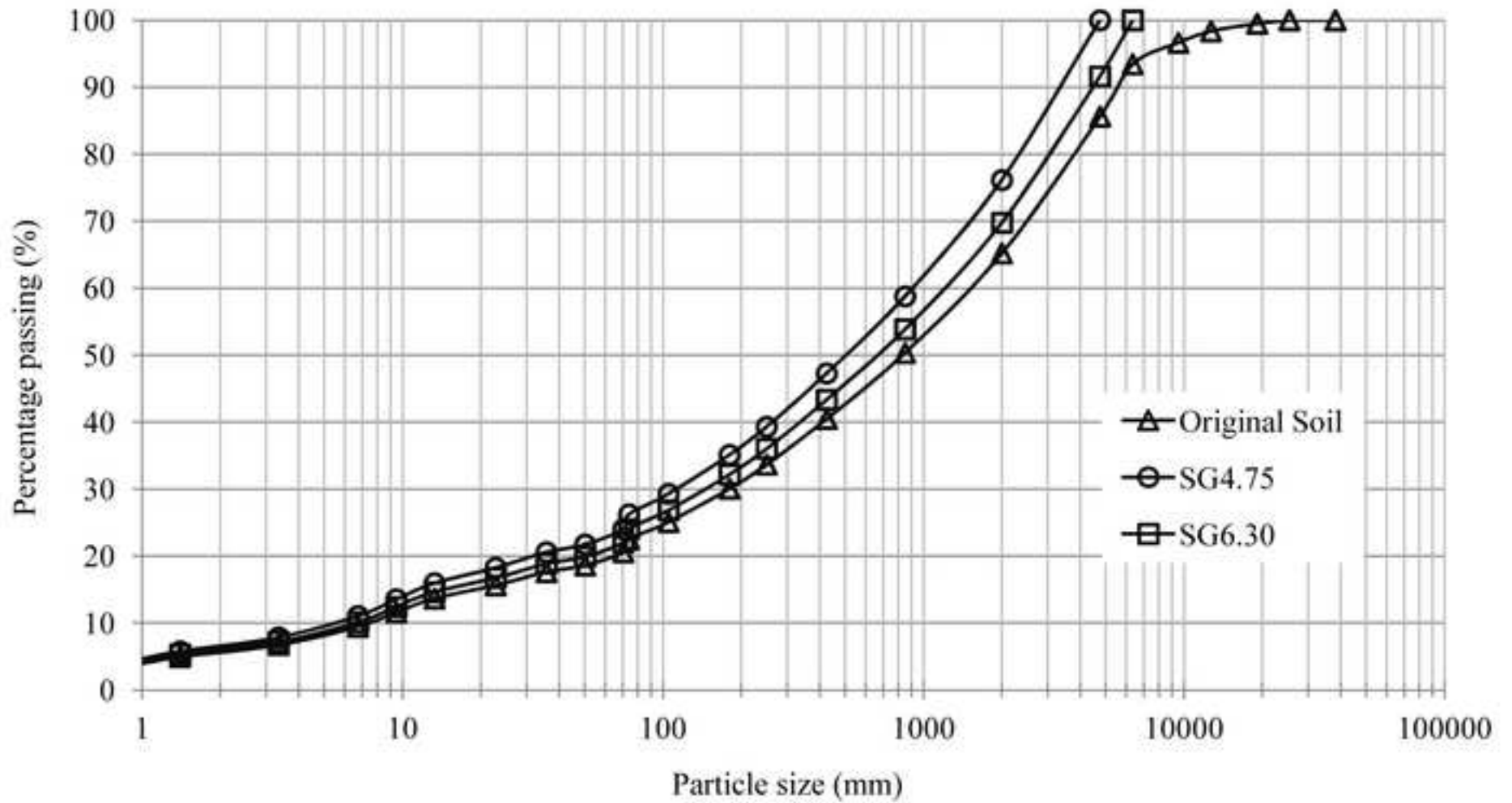


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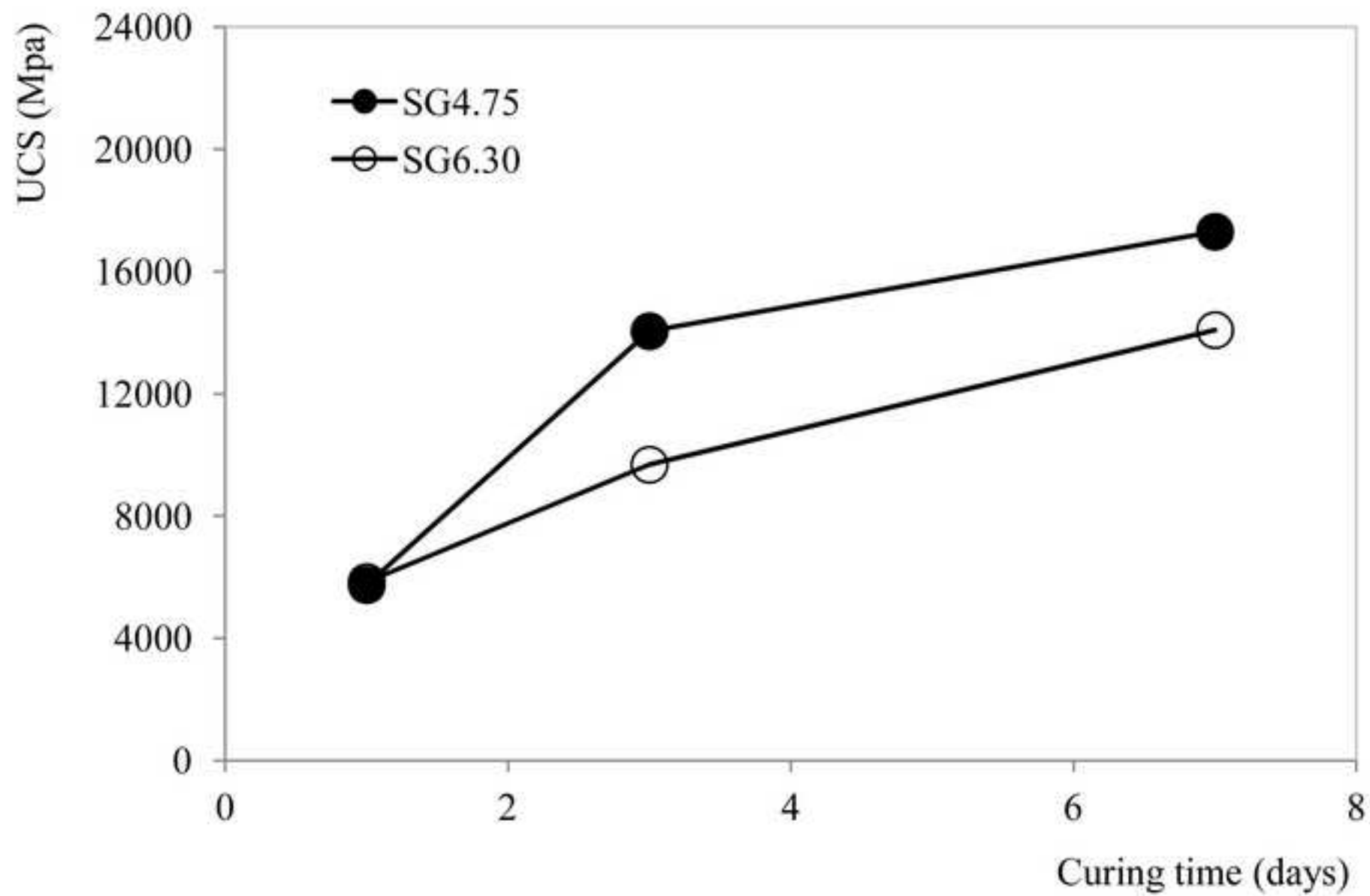


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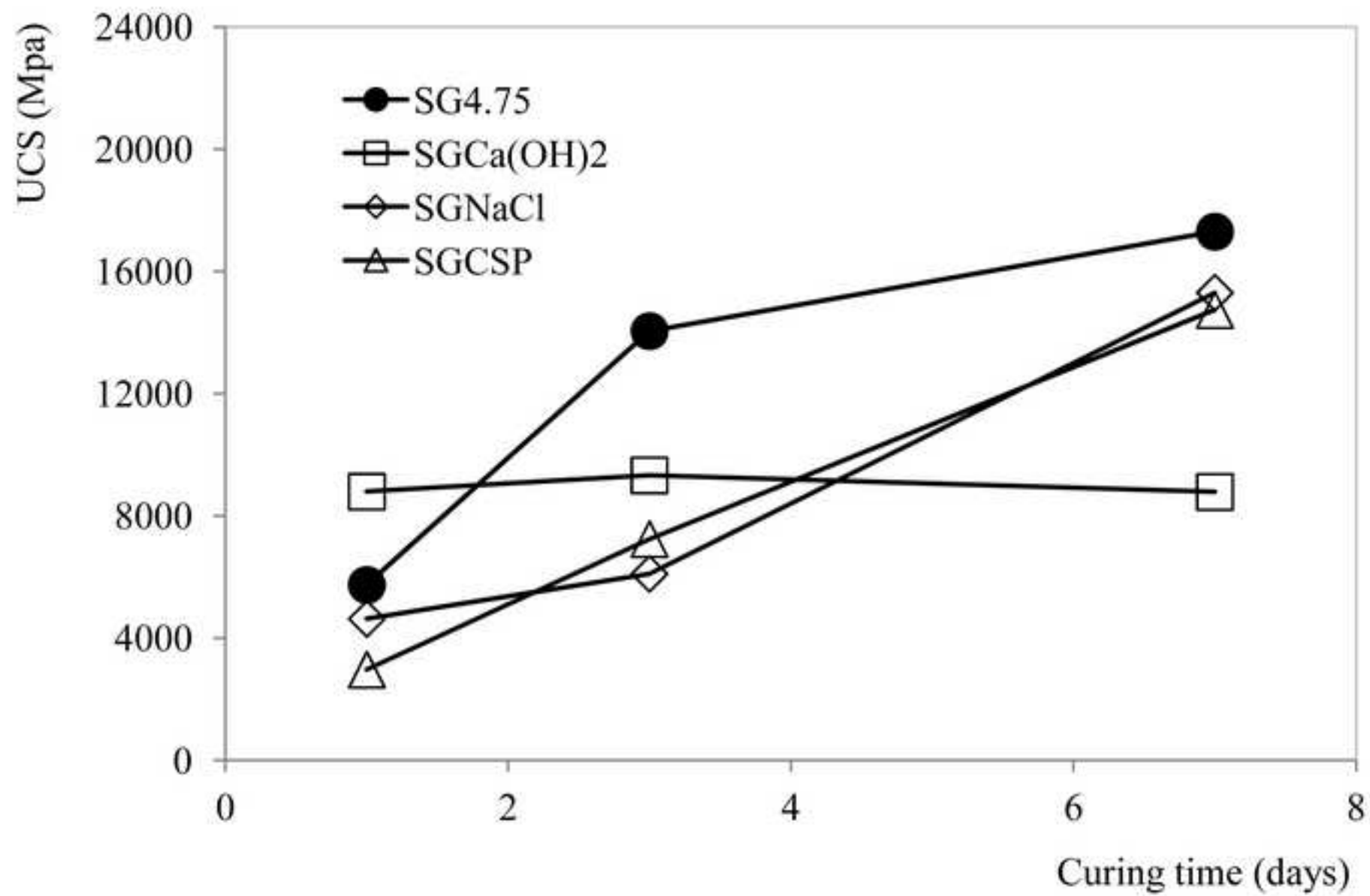


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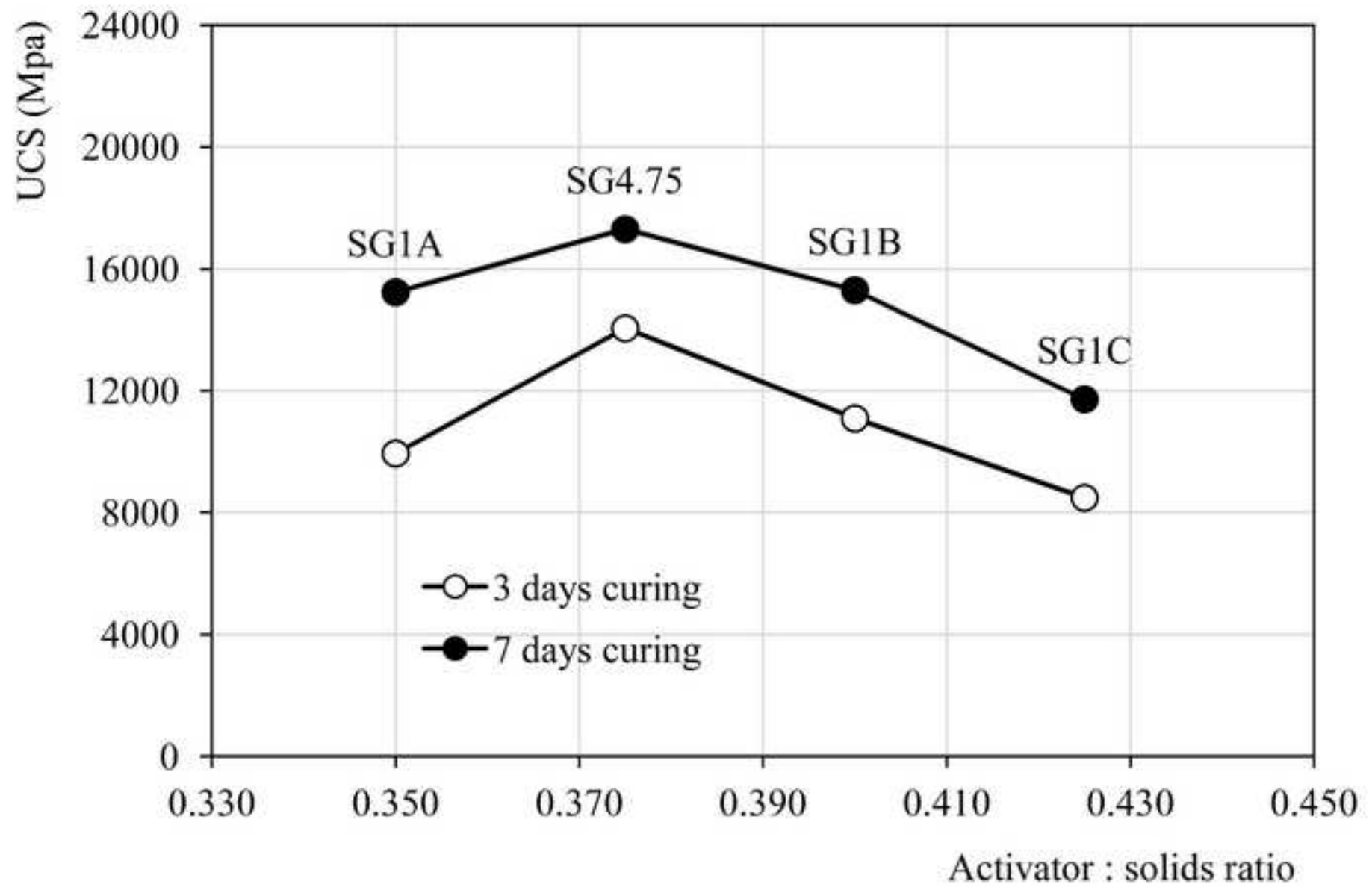


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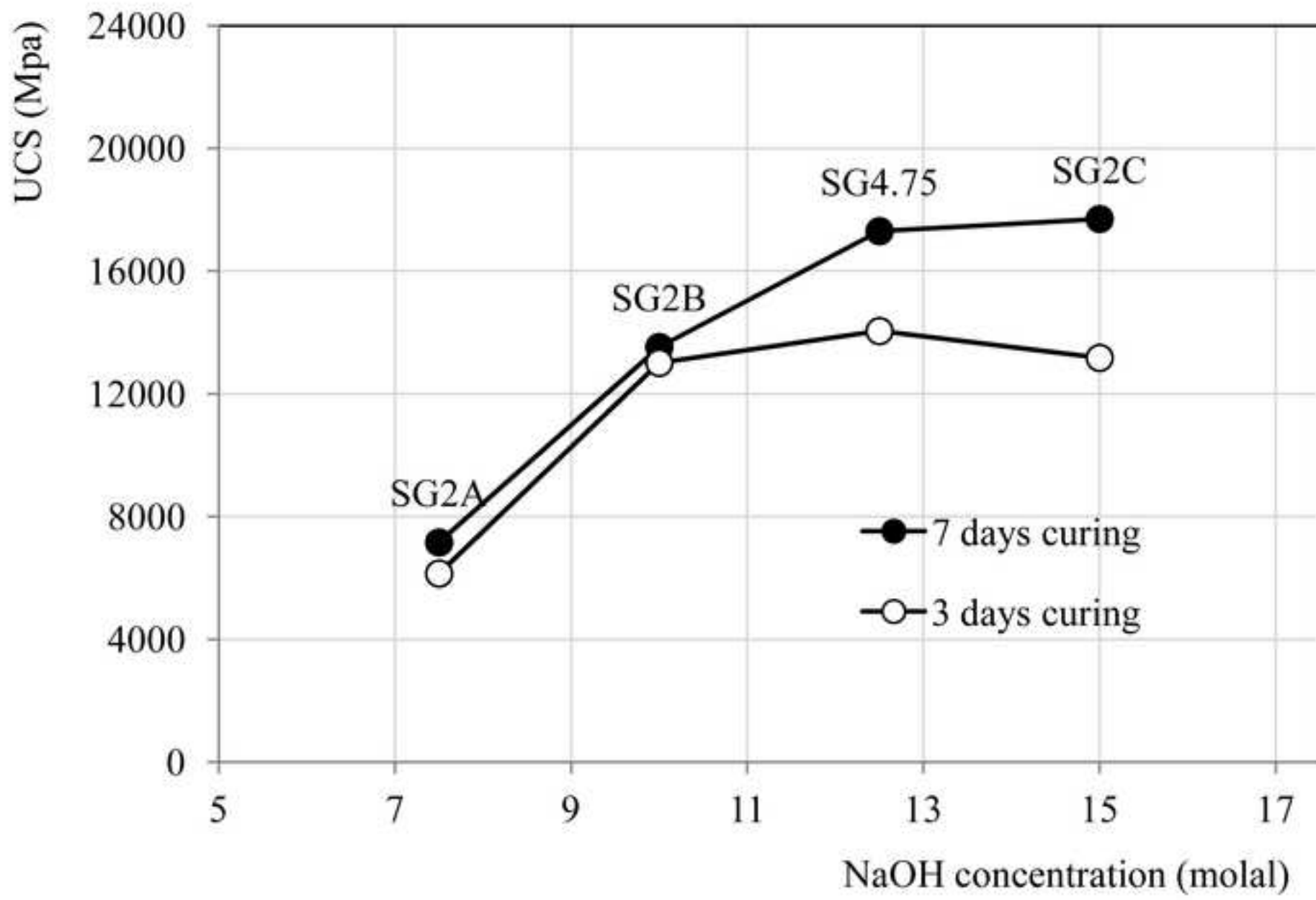


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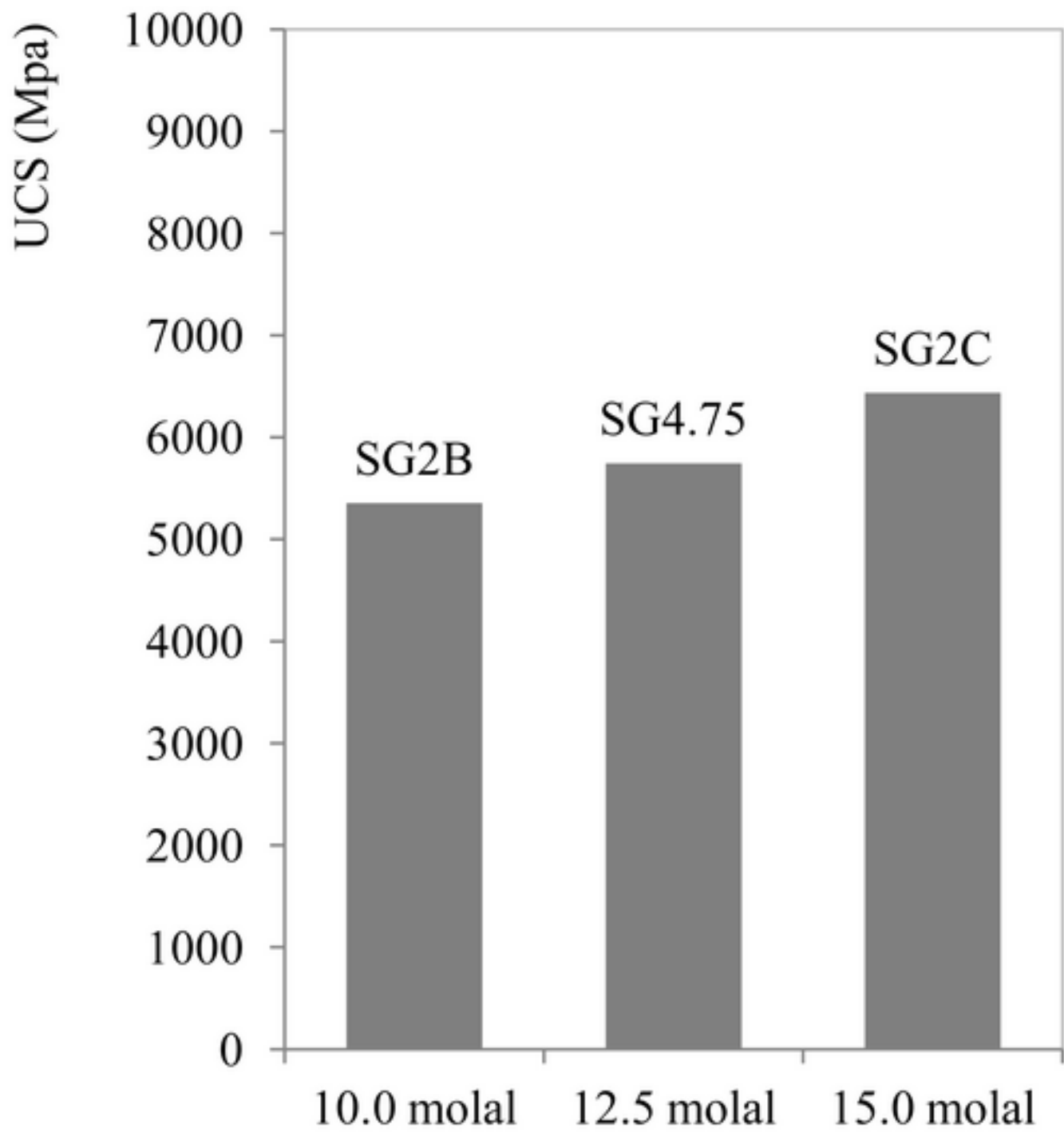


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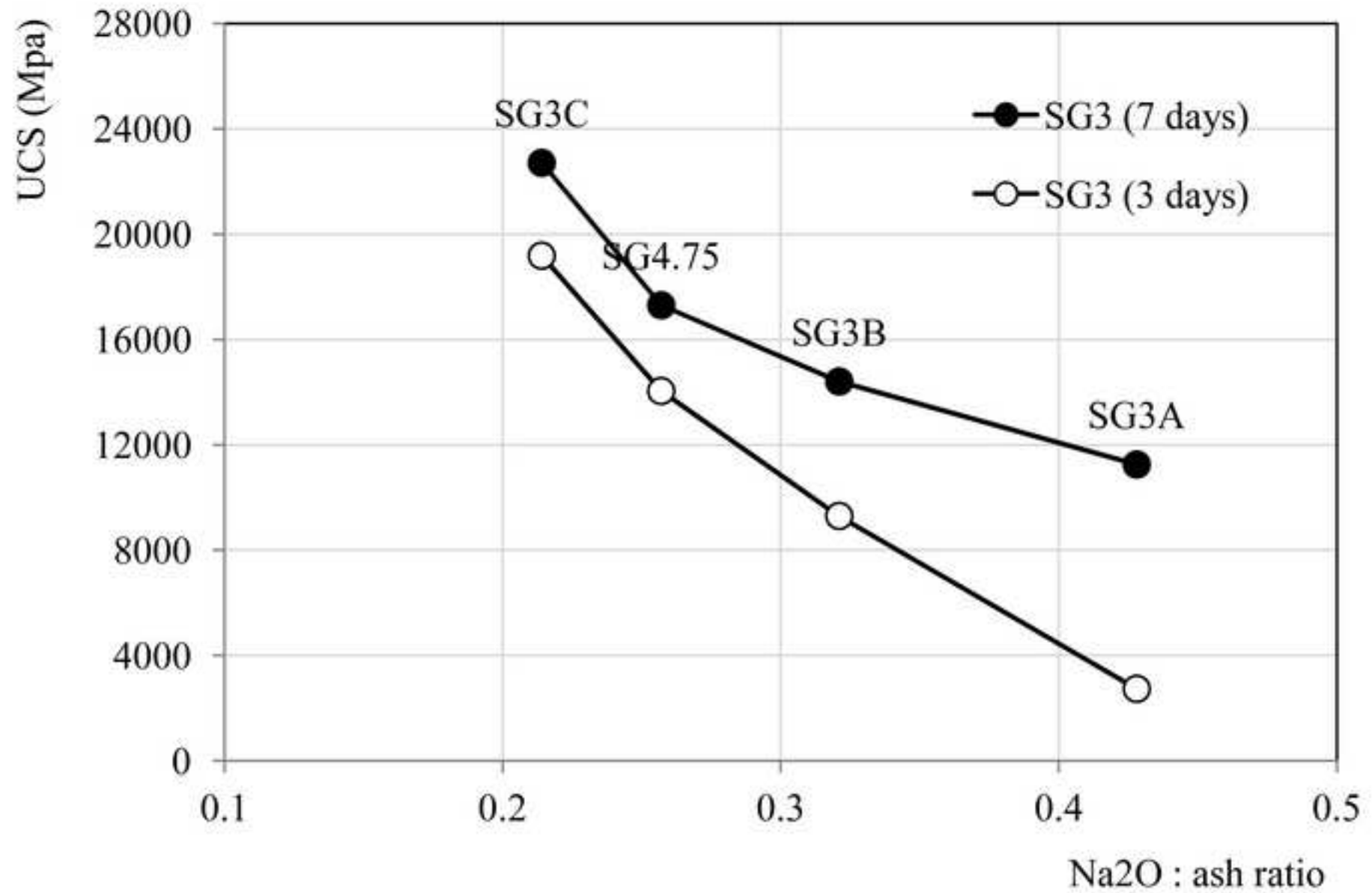


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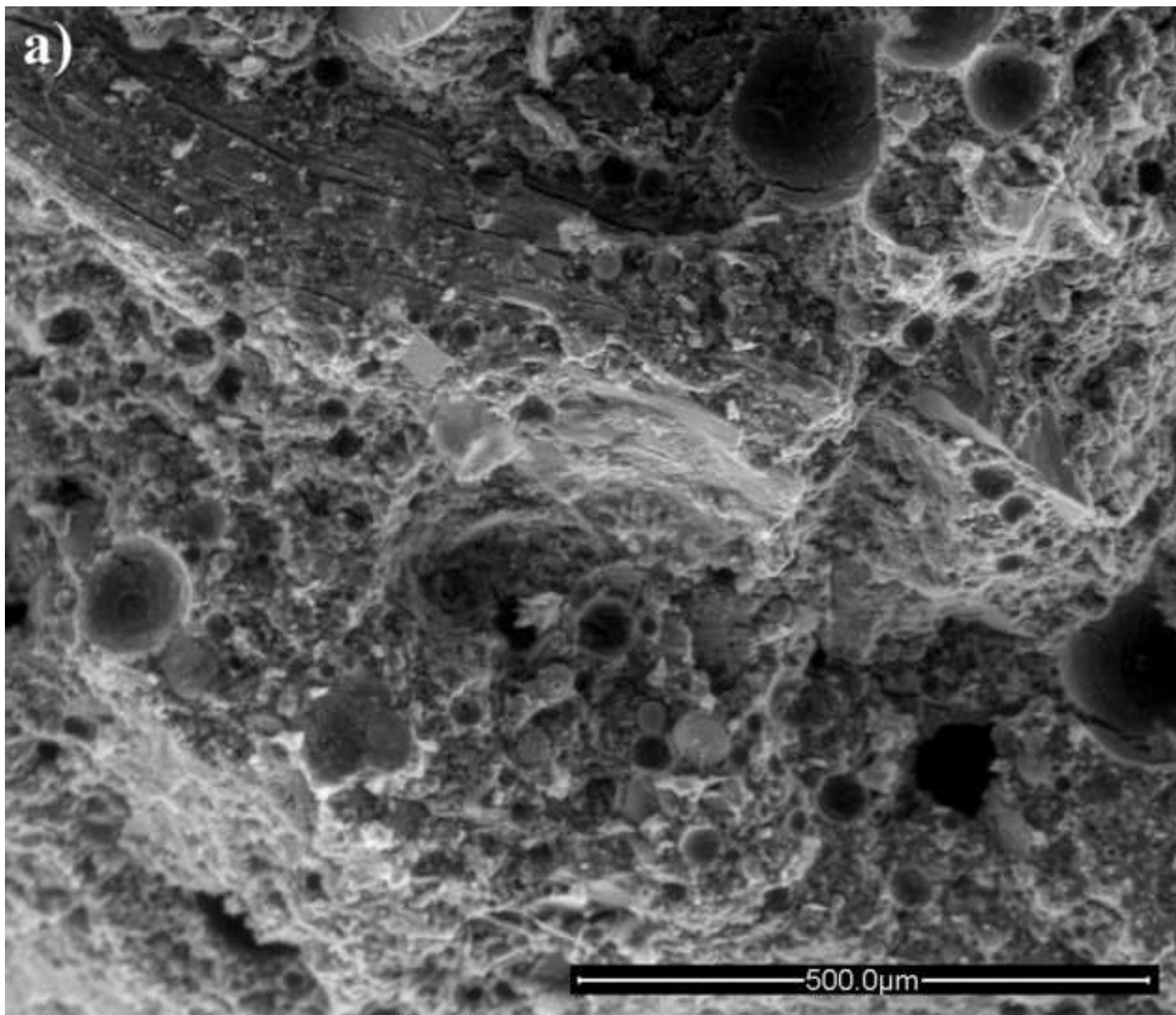


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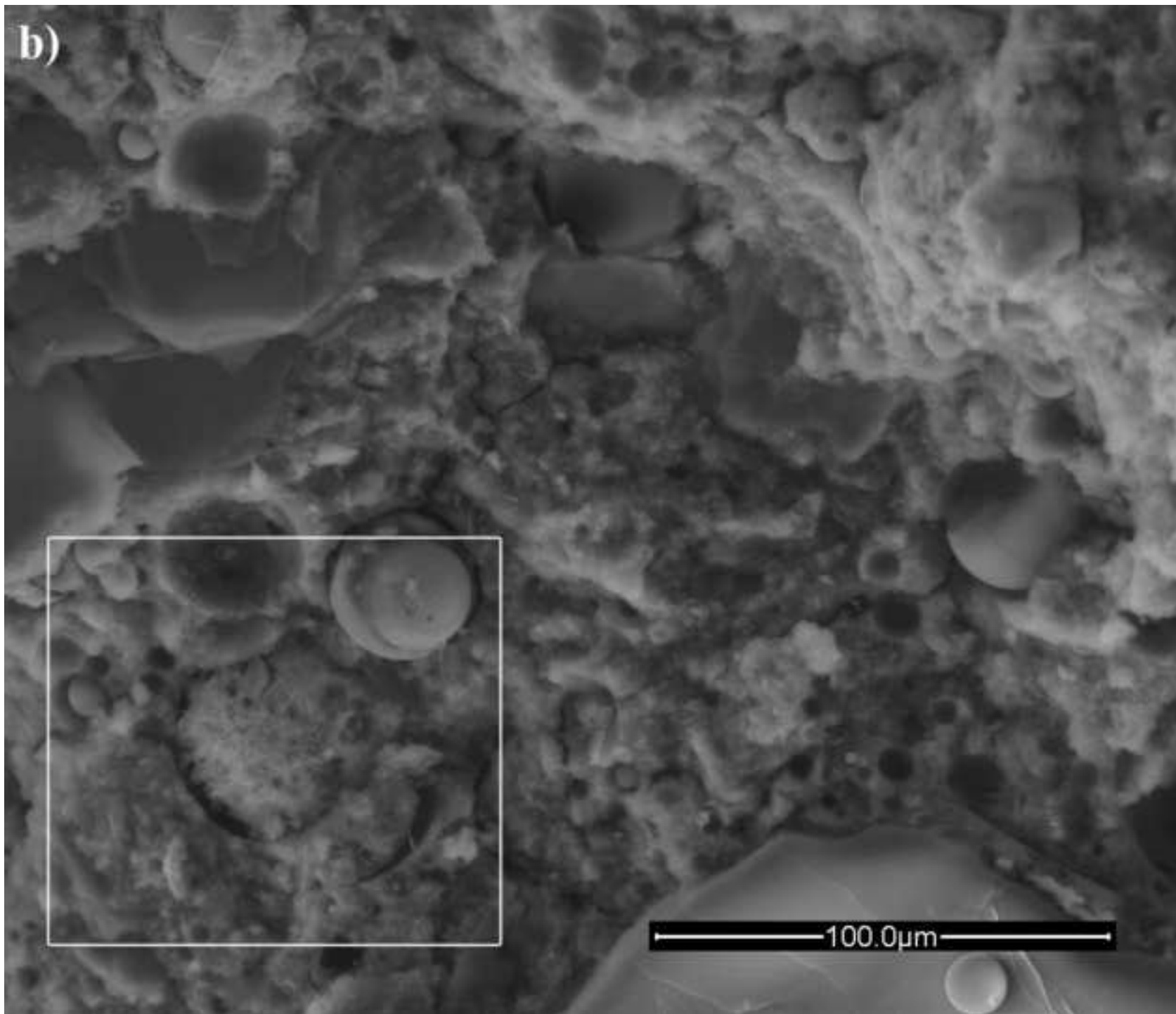


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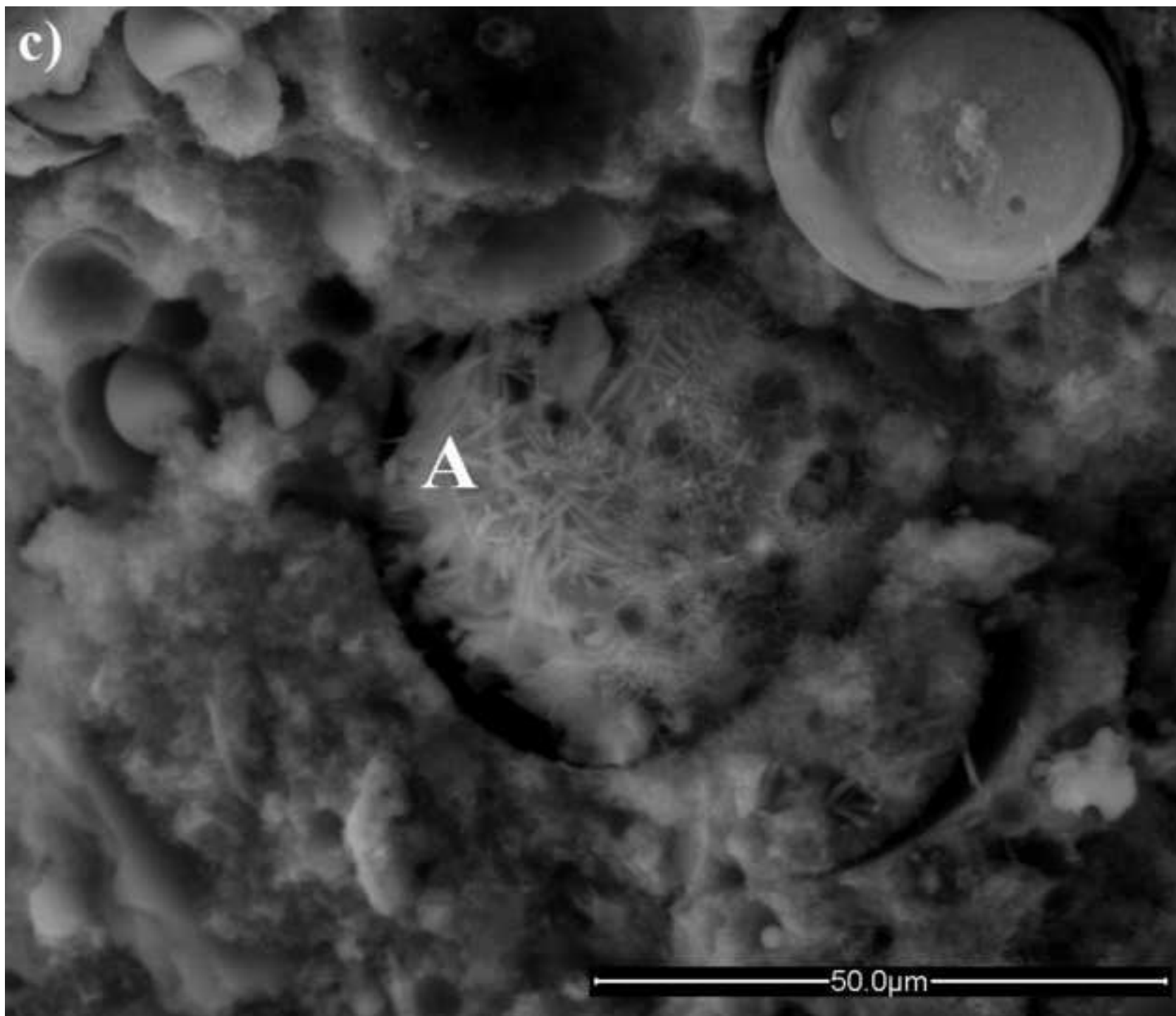


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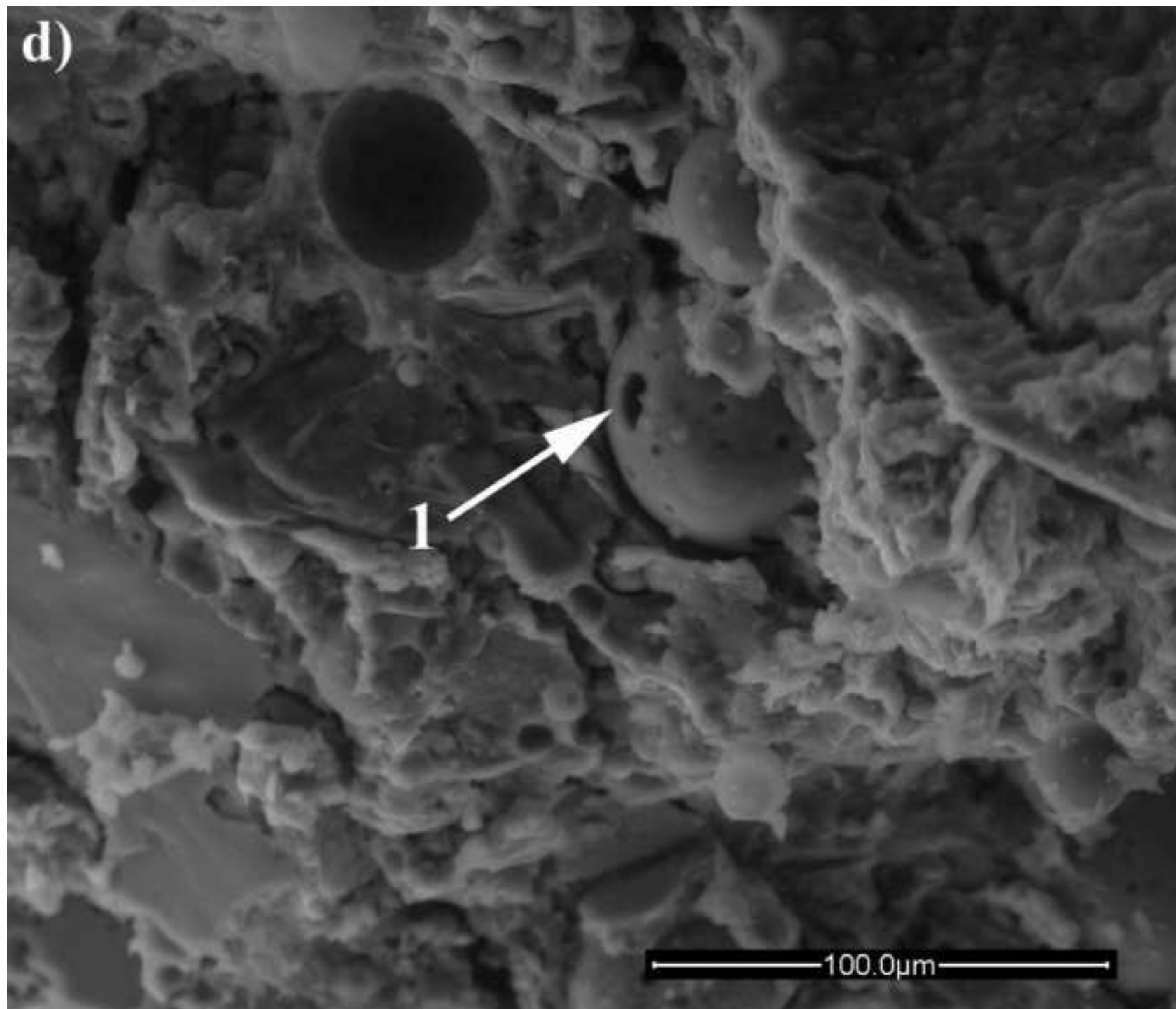


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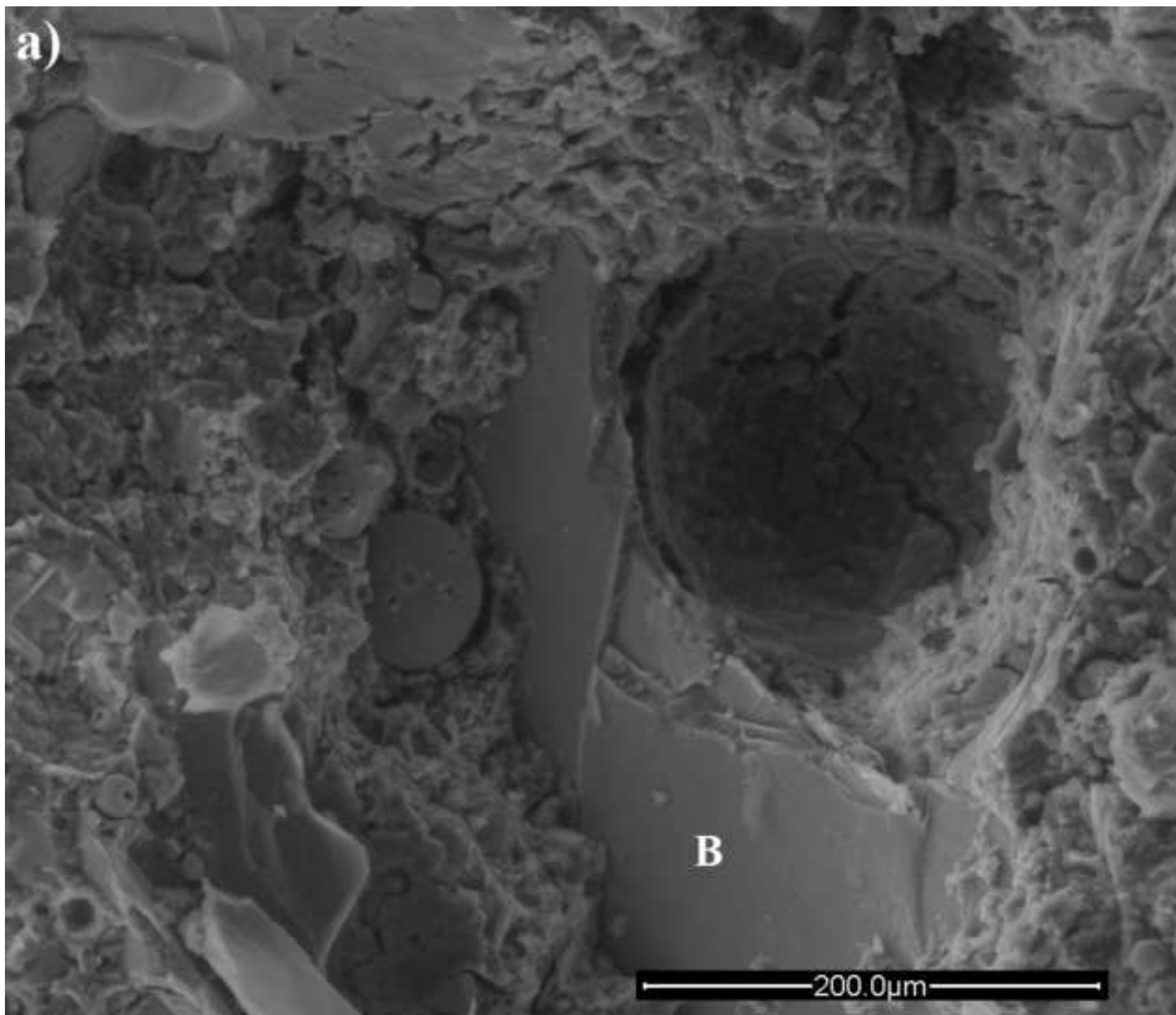


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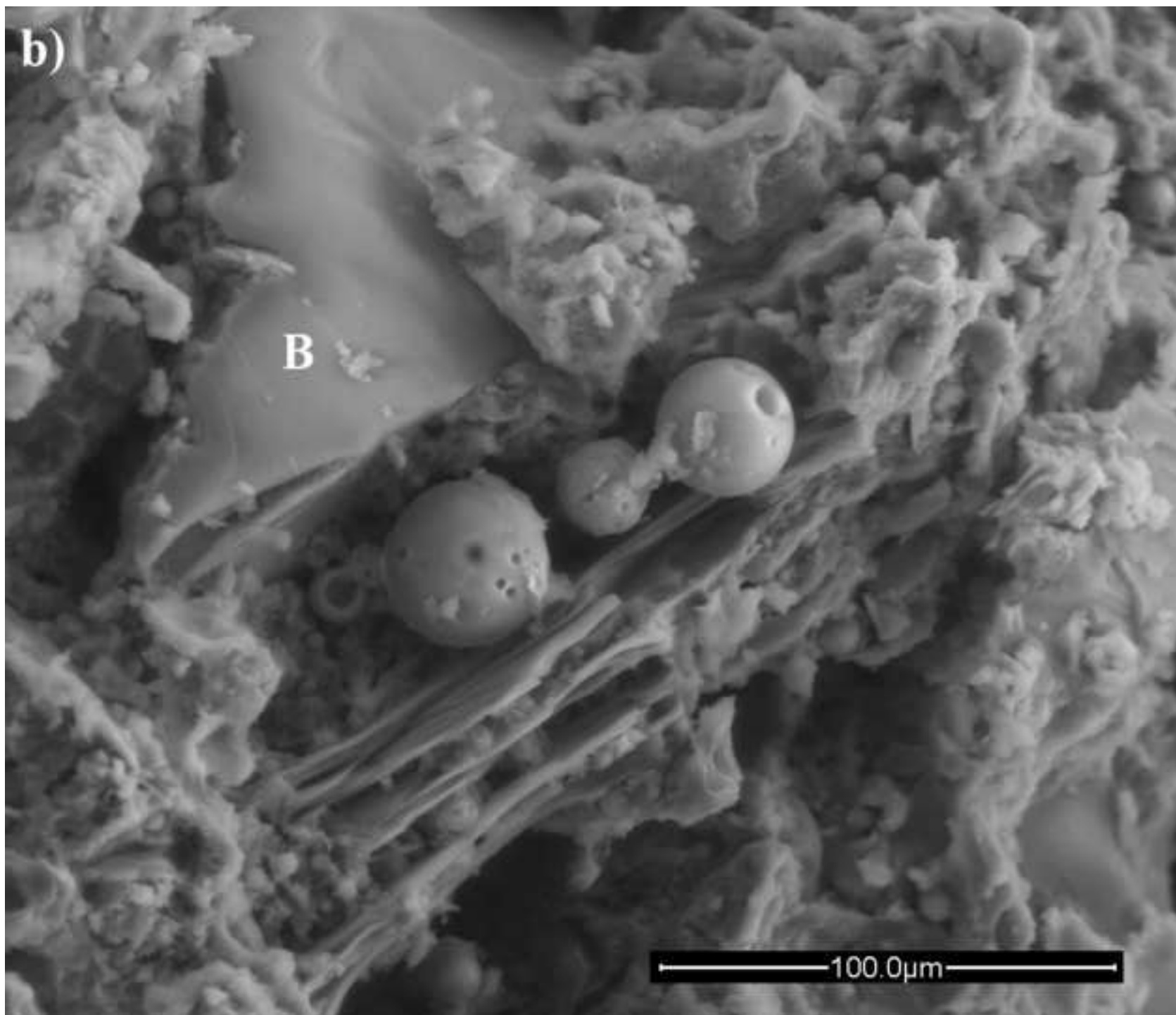


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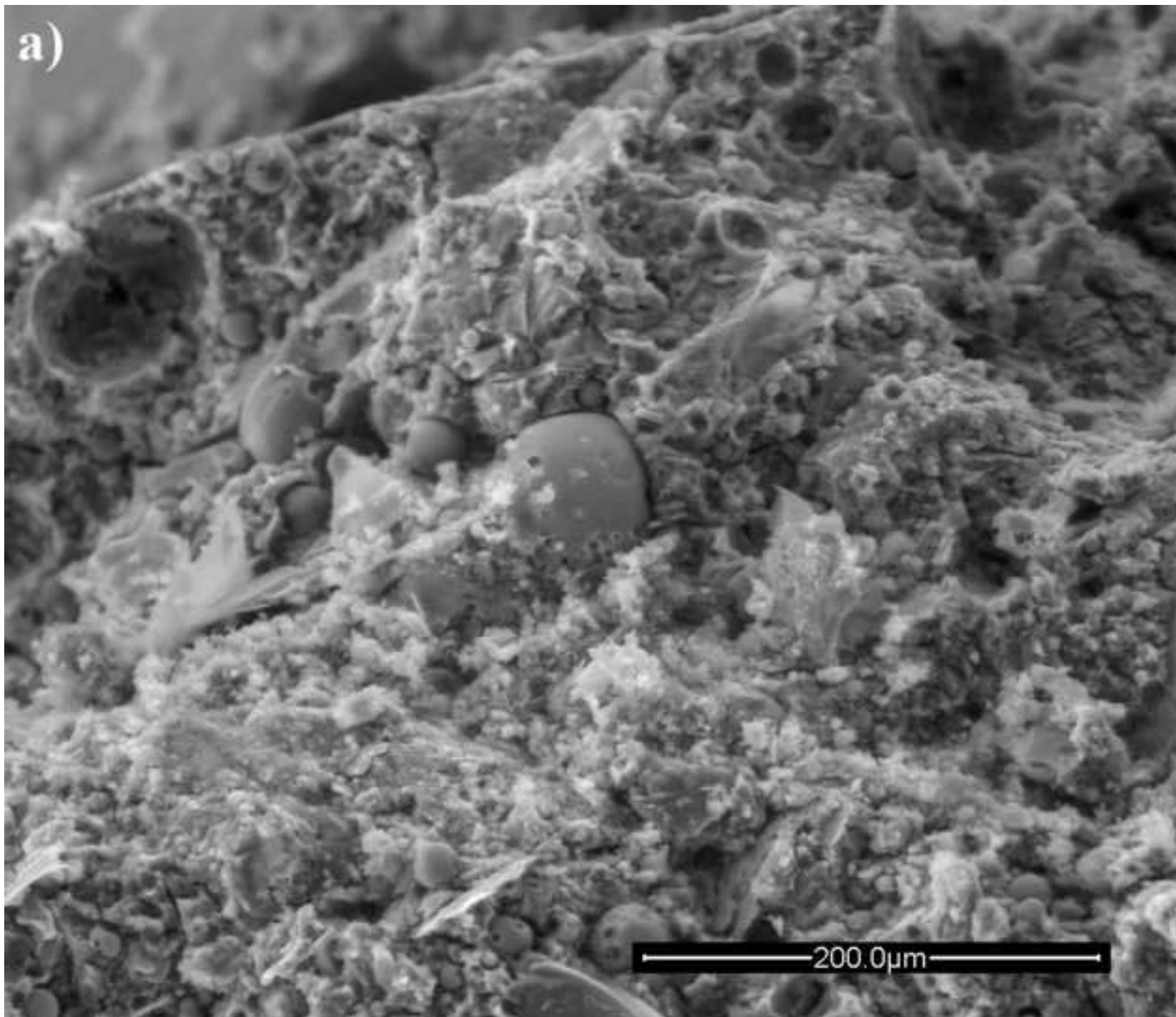


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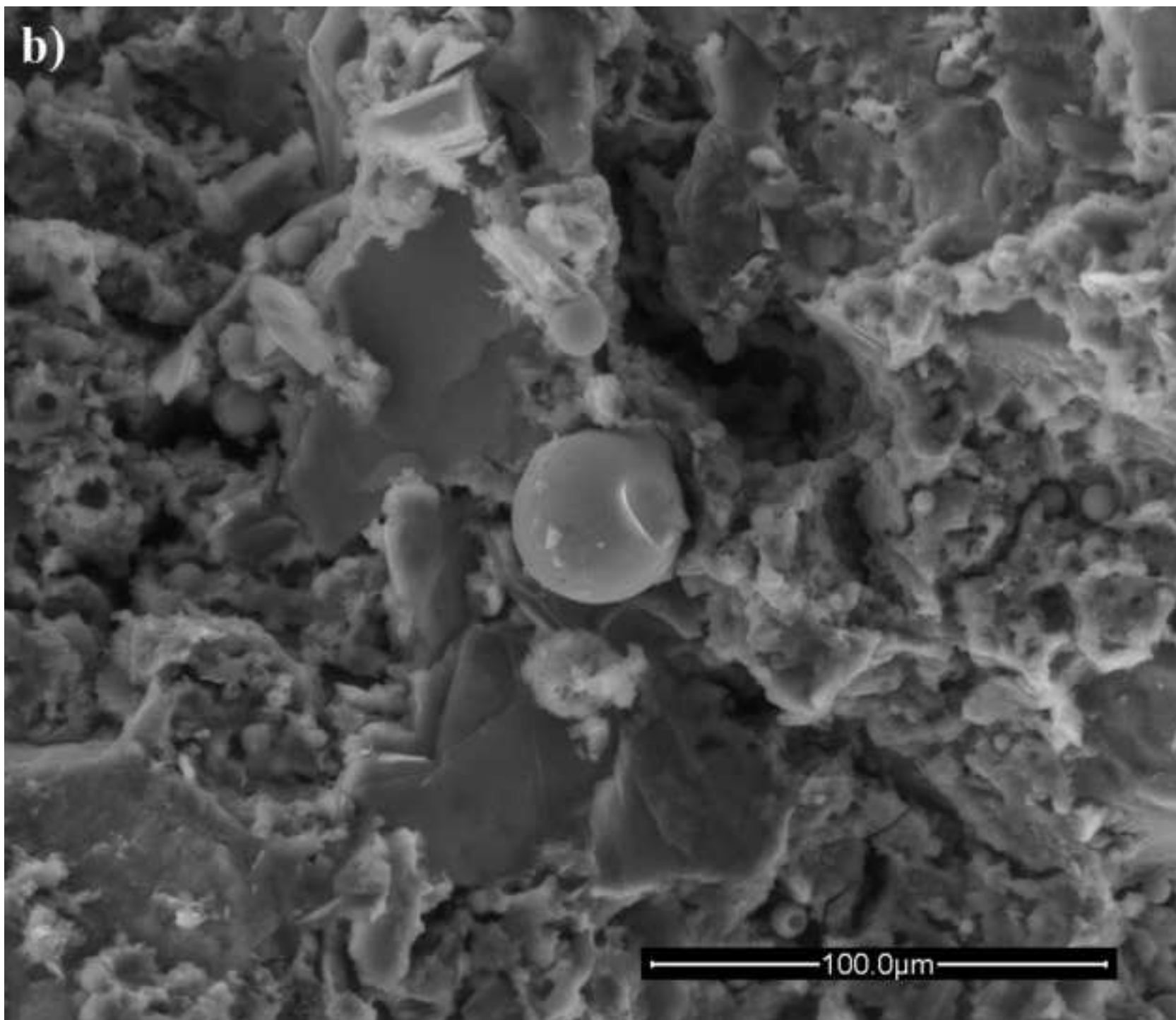


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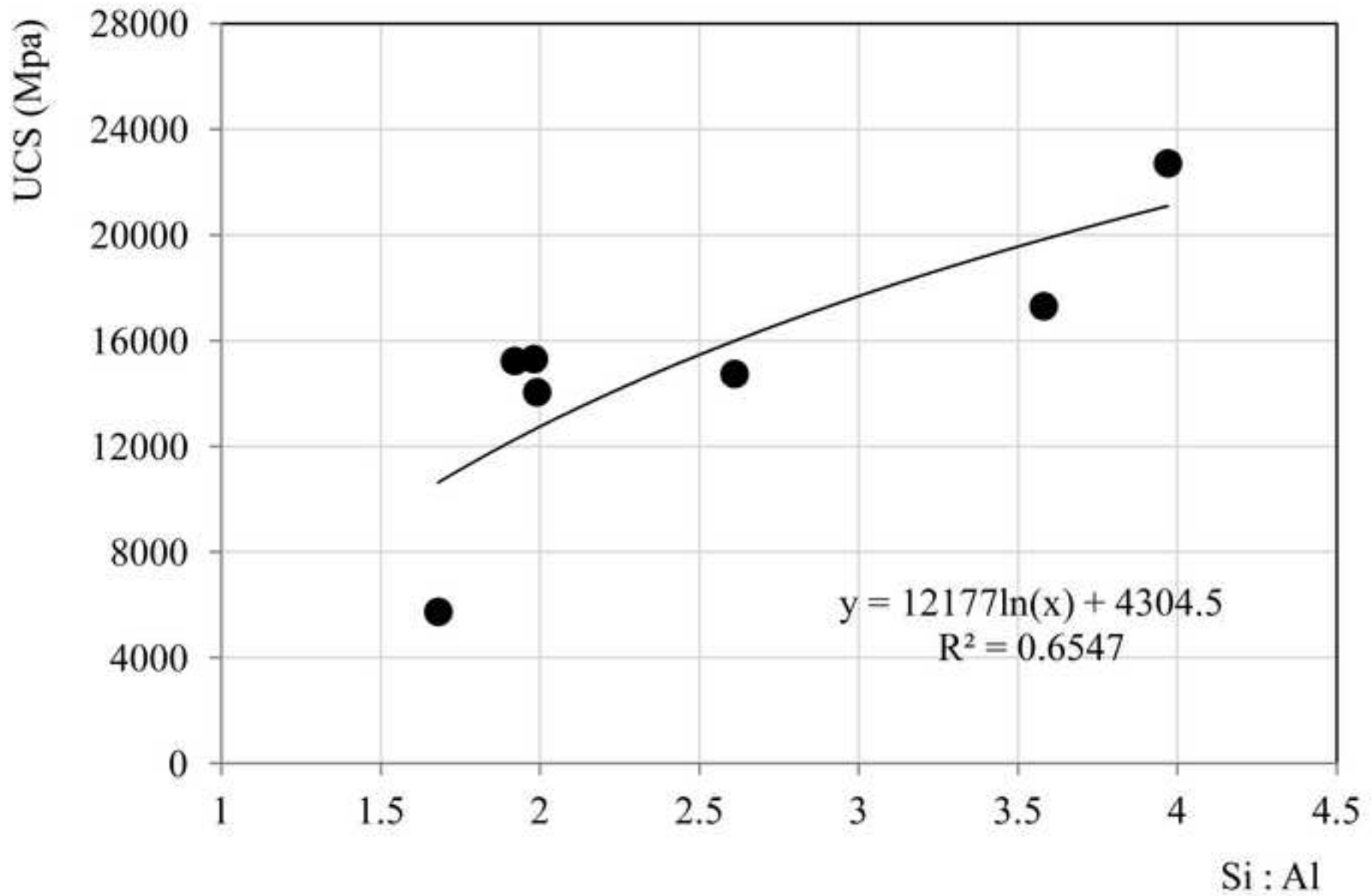


Figure11b

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