Effects of Hydrophobic Expanded Silicate Aggregates on Properties of Structural Lightweight Aggregate Concrete

Q. L. Yu[1]; D. J. Glas[2]; and H. J. H. Brouwers[3]

Abstract: This article addresses the performance of structural lightweight aggregate concretes and the relation of their performance to density class. Natural expanded silicate materials treated with a hydrophobic agent were used and their effects were systematically investigated. Three lightweight concretes with densities of about 1,000, 1,150, and 1,400 kg/m^3 (classes D1.0, D1.2, and D1.4) were designed by applying an optimized particle packing theory. The microstructure, mechanical properties, and durability of the developed concretes were determined and the relations of these properties with density were evaluated. The lightweight concretes showed excellent structural efficiency, with 28-day compressive strengths of about 23, 28, and 42 MPa, respectively. Microstructural analyses showed that the developed concretes had a rather compact microstructure, contributing to enhanced strength. Existing codes for calculating concrete *E*-modulus were compared, and the best predicting formula is proposed. Mix D1.4 showed relatively low drying shrinkage, which can be attributed to relatively low initial water use and the internal curing effect brought about by the applied lightweight aggregate. The developed mixes showed excellent durability, as indicated by very low water penetration after 72 h of exposure under a pressure of 0.5 MPa (5 bars) and very small mass loss after 56 cycles of a freeze–thaw test under both deionized water and NaCl conditions. **DOI: 10.1061/(ASCE)MT.1943-5533.0003198.** *© 2020 American Society of Civil Engineers.*

Author keywords: Structural lightweight aggregate concrete; Hydrophobic natural expanded silicates; Microstructure; Structural efficiency; *E*-modulus; Drying shrinkage; Durability.

Introduction

Lightweight aggregate concrete (LWAC) has been used in structural concrete for many years (Chandra and Berntsson 2002). Different lightweight aggregates (LWA), including natural materials such as pumice, oil palm shell, and so forth, and artificial materials such as expanded clay, expanded shale, sintered fly ash, and so forth, are widely used in lightweight aggregate concrete (Mo et al. 2017). Lightweight concrete is a very versatile material that offers a number of technical, economic, aesthetic, and environment advantages (Haque et al. 2004). Because of its many advantages, such as low density, good thermal insulation, and good fire resistance, LWAC has recently been widely developed and applied as both a structural and nonstructural material.

However, the porous characteristics of lightweight aggregates may facilitate the transport of deleterious substances, such as chloride, sulphate, acid rain, and so forth, into the concrete matrix (Spiesz et al. 2013; Bogas et al. 2016; Kurt et al. 2016). The water absorption of porous lightweight aggregates is always a challenge, because the lightweight aggregates continue to absorb water even after being immersed for 50 days (Moreno et al. 2014). Nevertheless, wetted porous aggregates can potentially act as a self-healing agent, and a relative recovery of cracks, up to 60%, has been observed due to the application of prewetted lightweight aggregates (Medjigbodo et al. 2018). In addition, applying saturated lightweight aggregates has been shown to contribute to reducing the autogenous shrinkage of ultra-high-performance concrete (Meng and Khayat 2017). Efforts have been devoted to developing lightweight aggregates with a relatively dense outer layer; however, this has a negative influence on the interfacial transitional zone between the cement paste and aggregates (Zhang and Gjørv 1992). Several studies have been performed to modify the surface properties of lightweight aggregates by various methods in order to make them hydrophobic or water impermeable (Gürsoy and Karaman 2016; Peng et al. 2017; Güneyisi et al. 2016). Güneyisi et al. (2016) reported that the water absorption of lightweight aggregates was significantly reduced after waterglass treatment, and the compressive strength of lightweight concrete containing waterglass surface-treated lightweight aggregates significantly increased. Nevertheless, there has still been limited effort to research the effects of hydrophobic lightweight aggregates on lightweight concrete.

Recent developments with regard to lightweight concrete structures have seen increased attention on durability aspects (Hwang and Hung 2005). ACI 318 stresses both the maximum water-to-cement ratio for highlighting the utilization of pozzolanic material and the minimum 28-day compressive strength for guaranteeing construction safety (ACI 2008). The use of supplementary cementitious materials (SCM) can help to modify the microstructure and to improve the latent mechanical properties and durability (Moreno et al. 2014). Mo et al. (2017) reported that the use of fly ash, silica fume, blast furnace slag, and metakaolin is, in general, a benefit to durability in terms of resistance to chemical deterioration from chloride permeability, chloride diffusion, and electrical resistivity because of microstructure modifications caused by the use of the SCMs. Nevertheless, negative effects of SCMs, such as high

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water demand caused by high fineness or high porosity and slow reactivity resulting in low strength development, have also been observed (Mo et al. 2017; Farahani et al. 2017). Bogas et al. (2016) reported reduced carbonation resistance when silica fume was used in lightweight aggregate concrete, possibly due to poor dispersion of the fine particles. The positive effects of using ternary binders—including, for instance, a combination of portland cement, fly ash, and blast furnace slag or other SCMs—on durability in order to overcome this issue has also been reported. For example, Real et al. (2015) observed that the chloride penetration resistance of lightweight concrete was improved by using a ternary binder of portland cement, fly ash, and silica fume.

In previous studies, mix design methodology based on acquiring an optimized packing of all the ingredients in lightweight aggregate concrete and relevant properties, including workability, strength, and durability, have been investigated (Spiesz et al. 2013; Yu et al. 2013; Yu et al. 2015). It has been shown that lightweight aggregate concrete can be designed with excellent properties under different density classes for different engineering applications. According to ACI 213 R-03 (ACI 2003), concrete with a density range between 1,120 and 1,920 kg/m^3 and a minimum 28-day compressive strength of 17 MPa can be termed structural lightweight concrete. However, as reviewed by Yu et al. (2015), the compressive strength of lightweight concretes within this specified density range has shown very large deviations for concrete of the same density, and the effect of density class has not been systematically addressed.

The present research focused on studying the properties of structural lightweight aggregate concrete. A surface treated hydrophobic lightweight aggregate, natural expanded silicate, was used, and its effect, including the effect of size fraction and amount, on crucial engineering properties was evaluated. Furthermore, an expanded clay lightweight aggregate was used. A quaternary binder consisting of blast furnace slag cement, fly ash, and limestone powder was used, and microstructure, durability, sustainability, density, and workability were considered. Fresh behavior, including fresh density and workability, of the developed lightweight concrete was assessed. The microstructure of the resulting concrete was analyzed using scanning electron microscopy (SEM) and image analysis. Mechanical properties, including compressive strength, splitting tensile strength, and elastic modulus, were determined and are discussed. The structural efficiency of the developed lightweight concrete was calculated and compared with literature. Durability-related properties, including drying shrinkage, water penetration under pressure, and freeze–thaw resistance were investigated and the effect of density was assessed.

Experiments

Materials

The cement used in this study was blast furnace cement CEM III/A 52.5 N (provided by ENCI, Maastricht, Netherlands). The lightweight aggregates used were produced from natural expanded silicate materials (NEP) (provided by Rotec, Mühlheim-Kärlich, Germany) and recycled expanded clay. The lightweight aggregate was surface treated with a hydrophobic agent that ensured that no water would be absorbed by the porous structure; this is further discussed subsequently in the section "Results and Discussion." Limestone powder was used as filler. Class F fly ash was used to replace cement. A polycarboxylate ether–based superplasticizer was used to adjust workability. The materials used are summarized in Table 1 and Fig. 1. The oxide compositions of the cement, limestone powder, and fly ash used were determined using X-ray fluorescence and are presented in Table 2.

Table 1. Summary of materials used

Materials	Specific gravity
CEM III/A 52.5 N	3.123
Fly ash	2.360
Limestone powder	2.650
NEP 0.04–0.09	0.750
NEP 0.09–0.3	0.700
NEP 0.25–0.5	0.700
NEP 0.5–1.0	0.600
NEP 1.0–2.0	0.550
NEP 2.0–4.0	0.500
EC 4.0–8.0	1.500
Superplasticizer	1.050

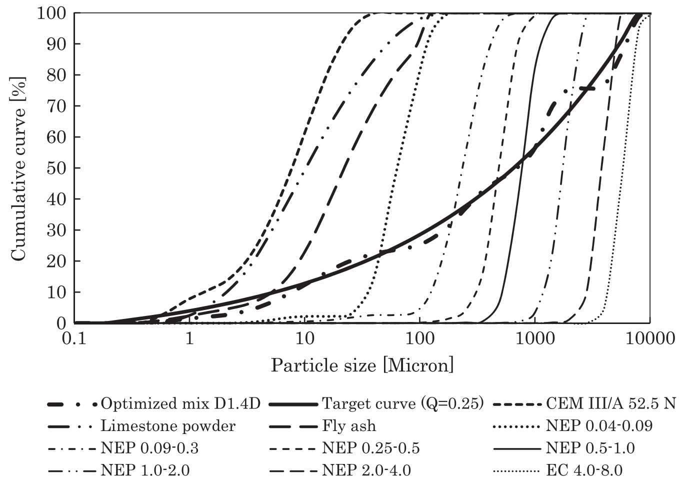

Fig. 1. Particle size distribution (PSD) of the raw ingredients used and the composed mixes.

Table 2. Chemical composition of powders used

Oxides (%)	Fly ash	CEM III/A 52.5 N	Limestone powder
SiO_2	54.62	27	0.8
Al_2O_3	24.42	9	0.2
CaO	4.44	51	54.0
MgO	1.43	0	1.0
Fe_2O_3	7.21	2	0.3
Na_2O	0.73	0.66	—
K_2O	1.75	—	0.3
SO_3	0.46	3.1	—
LOI	2.80	—	43.0

Mix Design

The mix proportions of the structural lightweight aggregate concrete were designed using a mix design tool that applies the optimized packing theory. A modified Andreasen and Andersen (A&A) curve acted as a target function for the subsequent granular optimization of the individual materials (Andreasen and Andersen 1930). The proportions of the individual materials in the mix design were adjusted until an optimum fit between the composed mix and the target curve was reached using an optimization algorithm based on the least-squares method (LSM). Detailed mix design

Table 3. Proportions of the designed mixes (kg/m^3)

Proportions	D1.0	D1.2	D1.4
CEM III/A 52.5 N	400	400	423
Limestone powder	33	30	32
Fly ash	113	78	83
NEP 0.04–0.09	26	49	12
NEP 0.09–0.3	45	84	119
NEP 0.5–1.0	69	124	41
NEP 1.0–2.0	110	110	88
NEP 2.0–4.0	98	0	0
EC 8	0	53	289
Water	240	195	169
Superplasticizer	0.007	0.021	0.018

methodology has been investigated and can be found elsewhere (Yu et al. 2013; Yu and Brouwers 2012; Hüsken and Brouwers 2008; Yu et al. 2014; Li et al. 2017). In the present study, attention was given to the influence of density on the structural behavior of lightweight aggregate concrete. Three mixes, with density classes of D1.4, D1.2, and D1.0 according to EN 196-1 (BSI 2005) were designed. A quaternary binder, consisting of blast furnace slag cement, class F fly ash, and limestone powder was studied and used in this research, and microstructure, mechanical properties, workability, durability, and sustainability were considered. Detailed information is presented in Glas (2017). The mix proportions are provided in Table 3, and the particle size distributions of the materials used and the composed mixes are given in Fig. 1, using mix D1.4 as an example.

Testing Methods

Mixing Procedure

In this study, the concrete matrix was prepared following the method described in Yu et al. (2015). The natural expanded silicate aggregates were surface-treated with a hydrophobic agent to ensure that no water would be absorbed by the porous structure (ACI 2003). Because the expanded clay (EC) aggregates used had a relatively high water absorption, the EC was presoaked for 1 h in water and then surface dried before the concrete mixing procedure began. Mixing was executed under laboratory conditions with dried and tempered aggregates and powder materials. Total mixing time was about 8 min. The room temperature during mixing and testing was about 21°C ± 1°C.

The LWAC mixes were cast in molds with dimensions of 150 × 150 × 150 mm or 100 × 100 × 400 mm for different properties tests at hardened state. The samples were demolded 24 h after casting and subsequently cured in a climate chamber with a relative humidity (RH) >90% at room temperature until testing age.

Fresh Behavior

After mixing, the fresh behavior of the concrete, including density and flowability, was determined following EN 12350-5 (CEN 2009a) and EN 12350-6 (CEN 2009b), respectively. For the slump flow test, an Abrams cone with an internal upper/lower diameter equal to 100/200 mm and a height equal to 300 mm was employed. Two diameters (d1 and d2) perpendicular to each other were recorded and their mean was recorded as the flow value of the LWAC.

Microstructural Analysis

The air content of the developed lightweight concrete was expected to be rather high because of the incorporated lightweight aggregates. It is not reliable to measure the air volume of LWAC in traditional ways [such as the pressure method according to EN 12350-7 (CEN 2009c), BS EN 12390-7 (CEN 2009f), or counting the particle density of all raw solid ingredients] because of the porous LWA and the relatively large deviations in the particle densities of the LWAs. Thus, another method was used in the present study, which was based on the analysis of cross sections of hardened specimens of the developed mixes. Pictures were taken in a conditioned environment in which a light source created an even light perpendicular to the surface of the cross sections, which provided shading of the pores. The accuracy of this method was a pore size of approximately 40 μm. Therefore, only the entrapped air bubbles in the cement paste and the air in the LWA could be detected, providing a basic idea as to how the air was distributed in the matrix, which primarily affects properties such as strength and durability.

The microstructure of the developed concrete was further characterized by scanning electron microscopy using an FEI instrument (FEI Quanta 600 FEG-SEM, Eindhoven, Netherlands) with a Schottky field emitter gun operating in high vacuum mode at an accelerating voltage of 10 kV.

Mechanical Properties

Mechanical properties, including compressive strength, splitting tensile strength, and modulus of elasticity, were investigated in this study. Compressive strength was tested using cubic samples (150 × 150 × 150 mm) at different curing ages of 1, 7, 28, and 91 days, following EN 12390-3 (CEN 2009d). Splitting strength was tested according to the EN 12390-6 (CEN 2009e). The modulus of elasticity was tested on prisms (100 × 100 × 400 mm) after 28 days of curing according to EN 12390 (CEN 2013).

Drying Shrinkage

The samples for the drying shrinkage test were cast in molds with dimensions of 40 × 40 × 160 mm and cured in sealed conditions at a temperature of 20°C. After 24 h of curing, the specimens were exposed in a cabinet with a temperature of 20°C and a relative humidity of 60% ± 5% per DIN 52-450 (DIN 1985); the initial length (L_0) was also measured at that time. Afterward, length (L_n) was measured periodically until an age of 54 days. Length change was calculated as follows:

$$L(\%) = \frac{L0 - Ln}{Li} \times 100\% \tag{1}$$

where L_i = effective initial length.

Durability

The produced concrete's permeability to water under pressure was tested at the age of 28 days according to EN 12390-8 (CEN 2009g). The samples (three 150-mm cubes for each prepared mix) were exposed to water under a pressure of 5 bars for 72 h and were subsequently split in order to measure the maximum depth of the obtained water penetration front. If leakage of water from the side wall of a cube was observed, the test on the cube in question was terminated.

Freeze–thaw resistance, expressed by the surface scaling factor Sn, was determined following EN 12390-9:2006 (CEN 2009h). In the present study, both deionized water and NaCl solution were used as exposure environments for the freeze–thaw resistance test; cubic samples (100 × 100 × 100 mm) were used for the experiments. The applied temperature profile followed the recommendations given in CEN (2009h). The level of the freezing medium on the surface of the concrete was adjusted regularly. In total, 56 freeze–thaw cycles were performed; the surface scaling was measured after 7, 14, 28, and 56 cycles.

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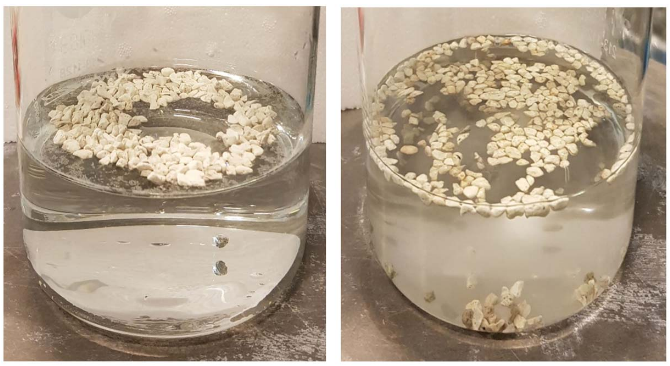
Results and Discussion

Fresh Behavior

The natural expanded silicate aggregates used in this study were treated with a hydrophobic coating [M. Roos and G. Runkel, "Granular pumice and method for producing granular pumice," US Patent Application No. 2013/0143044 A1 (2013)]. The treated aggregates showed an excellent water repellent effect, and the samples were able to float in water for one week without any water absorption; that is, they were completely hydrophobic. The hydrophobicity of the aggregates was checked before the application in this research by immersing them in water and isopropanol, respectively, as shown in Fig. 2. The aggregates floated on the water and did not absorb any water but were quickly wetted by the isopropanol and slowly sank to the bottom of the beaker. The isopropanol also gradually turned muddy, while the water remained clear. This unique property ensured that the widely known issue of water absorption by lightweight aggregates did not need to be taken into account when using this aggregate in concrete.



(a) (b)

Fig. 2. Hydrophobic aggregates immersed in (a) water; and (b) isopropanol.

Table 4. Fresh behavior of the designed concretes

Value	D1.0	D1.2	D1.4
Fresh density (kg/m^3)	1,189	1,324	1,504
Apparent density (kg/m^3)	1,226	1,324	1,544
Slump flow (mm)	435	260	780
Flow class	F3	F1	F6

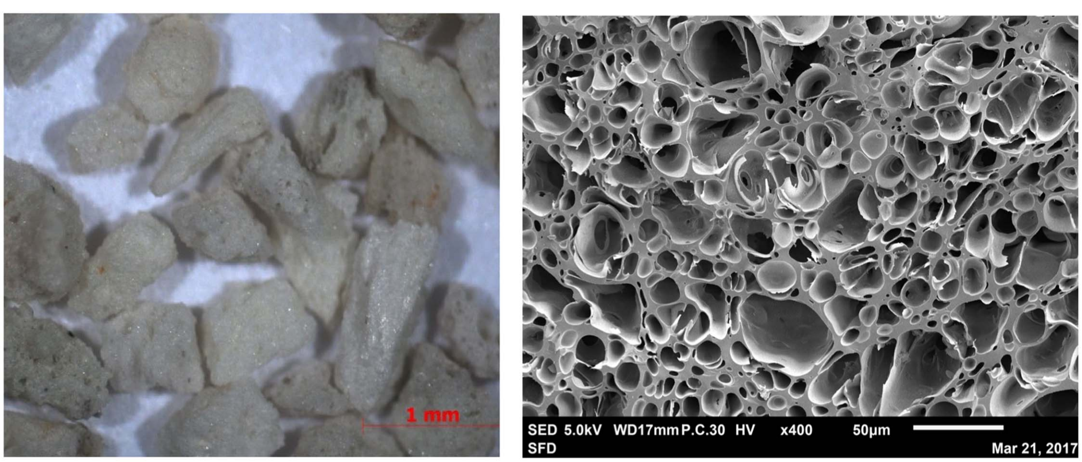
The fresh behavior results for the designed recipes are listed in Table 4. However, despite the hydrophobic treatment of the natural expanded silicate, it was observed that the aggregates still uptake certain paste into their open pores. This was confirmed by the measured fresh densities (Table 4), which were slightly higher than the designed densities (Table 3). This was caused by the capillary absorption of small pores (Li et al. 2008). The broken shapes of the particles had a negative influence on workability, because the particles had high specific surface areas due to their shapes and surface textures, as shown in Fig. 3. It can also be seen that water absorption was different for the mixes. This is in line with Bogas et al. (2012), who reported that the water absorption of lightweight aggregates in a concrete mixes is inversely proportional to the initial water content.

The flow class shown in Table 4 suggests that the developed mixes can meet the requirements of various engineering applications. It can be seen that the adopted NEP 0.04–0.09 had a rather negative effect on workability when comparing mix D1.0 with mix D1.2. In addition, the system was very sensitive to the applied superplasticizer (SP); a very slightly excessive SP dosage can result in segregation. It is rather difficult to adjust workability by simply adding water or superplasticizer to the mix; this also explains the poor workability of the resulting mix, D1.2. Kurt et al. (2016) also observed that workability is easier to handle as the density of lightweight concrete increases.

Microstructure

Fig. 4 shows the air pore distribution in the developed mixes. For all the mixes, the same compaction method was used for casting the concrete into the molds, and a poke stick was used to compact the concrete with 15 pokes. The largest air pore size in D1.0 reached 5 mm, while D1.2 and D1.4 had smaller pores, with a maximum pore size of 3 mm. The large pore sizes were possibly caused by the pore packing of the solids. The large air pore distribution in mix D1.0 potentially affects its mechanical and durability properties. This is further discussed in the following sections.

SEM analyses were used to characterize the microstructure of the developed concrete. The SEM pictures of the samples shown



(a) (b)

Fig. 3. (a) Natural expanded silicate aggregates; and (b) SEM image.

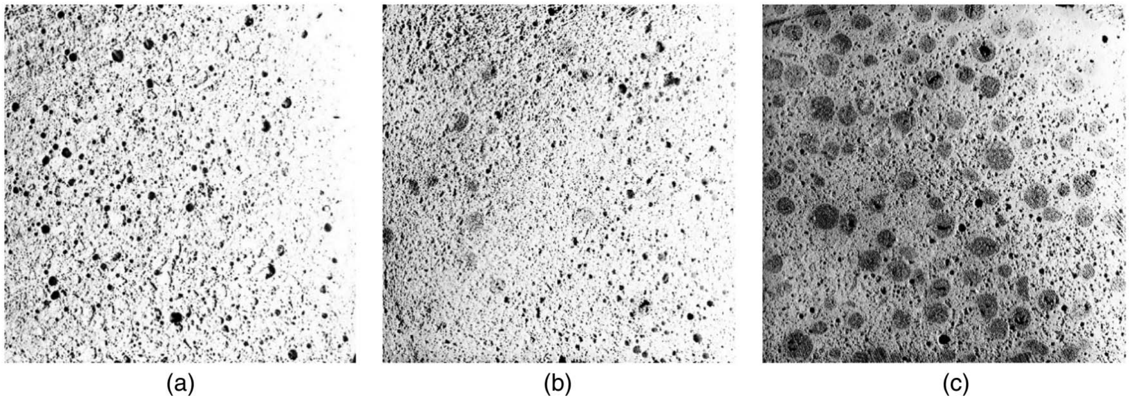


Fig. 4. Air pore distribution in the developed mixes: (a) D1.0; (b) D1.2; and (c) D1.4.

in Fig. 5 were taken after 9 months of curing. Mixes D1.2 and D1.4 had rather densely compacted structures and relatively good interfacial transition zones (ITZs); some apparent air pores were observed in mix D1.0, which was in line with the image analysis shown in Fig. 4. Zhang and Gjørv (1990) confirmed that the interfacial zone is more dense if the lightweight aggregate has a more porous outer layer. In multiphase composite such as concrete, the ITZ is the key area with regard to mechanical properties. Connected pores in the lightweight aggregates may absorb water from the sounding paste during mixing, which affects properties such as density and strength. No crystallized hexagonal portlandite plates were observed in the cement matrix; this can be attributed to the fly ash and ground granulated blast furnace slag (GGBS) utilized in the mix. This contributes to enhanced performance, such as higher mechanical strength, which will be discussed subsequently. In addition, no needle-like structures were seen in the SEM pictures; this can be explained by the relatively low amount of limestone powder in the system. Furthermore, SEM pictures taken using backscatter detector mode did not show any traces of alkali silica reaction in the matrix.

Mechanical Properties

Fig. 6(a) shows the strength development ($150 \times 150 \times 150$ mm) of the three mixes (D1.0, D1.2, and D1.4). After 7 days of curing, the compressive strength had reached already 90%–95% of the final 28-day strength; it was shown in a preliminary study that the pastes used in the concrete mixes still developed about 30% from 7 to 28 days. This indicates a ceiling strength for LWA combinations. This phenomenon was previously reported by Yu et al. (2013). Nevertheless, in this study, the concrete matrix still increased in strength by about 5%–10% between 7 and 28 days, while in Yu et al. (2013), LWAC mixes had already reached their final strength after 7 days. This can be explained by the higher crushing resistance of the LWA used in the present study; in addition, the irregular shape of the LWA used in this study contributed to a densified ITZ that also helped to enhance the strength.

Mix D1.0 reached a compressive strength of 23 MPa at the age of 28 days, while mixes D1.2 and D1.4 reached 28 MPa and 43 MPa, respectively. In order to have a density of around 1,000 kg/m^3, only natural expanded silicate aggregates were used in mix D1.0; these aggregates were less dense than the expanded clay aggregates. However, the natural expanded silicate aggregates, especially the larger particle sizes, had a lower crushing resistance than the expanded clay aggregates. Moreover, mix D1.0 required a higher water:binder (w:b) ratio in order to achieve an acceptable flowability. Despite the use of the same type of aggregates, the proportions differed from those used for mix D1.4, in which a significant amount of expanded clay aggregates was used. In addition, a lower amount of water was required, which contributed to a higher strength as well. The relationship between compressive strength and density shows the efficient design of lightweight concrete for structural applications; this relationship is usually interpreted using the term structural efficiency:

$$ste = \frac{\sigma_c}{\rho} \tag{2}$$

where ste = structural efficiency ($N \cdot m/kg$); σ_c = compressive strength at 28 days (N/mm^2); and ρ = apparent density of the sample (kg/m^3).

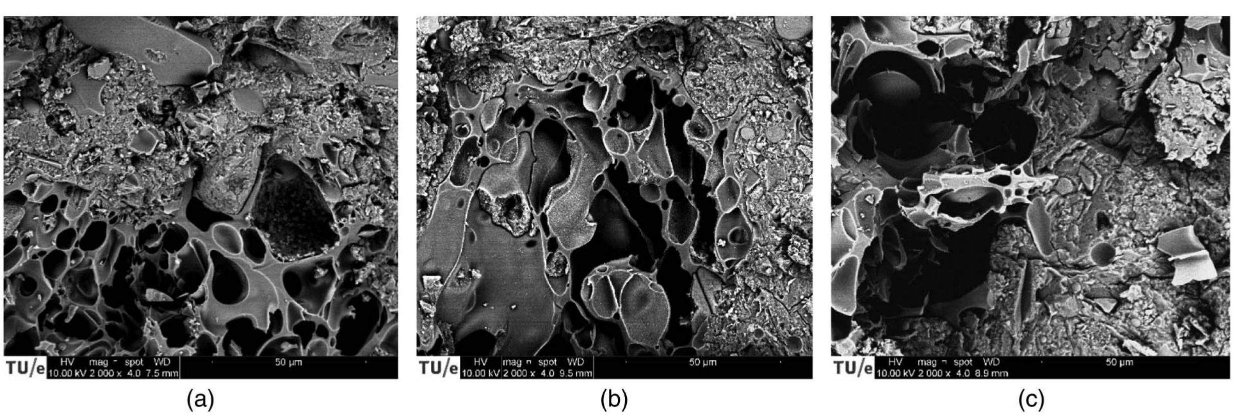


Fig. 5. SEM images of mixes: (a) D1.0; (b) D1.2; and (c) D1.4.

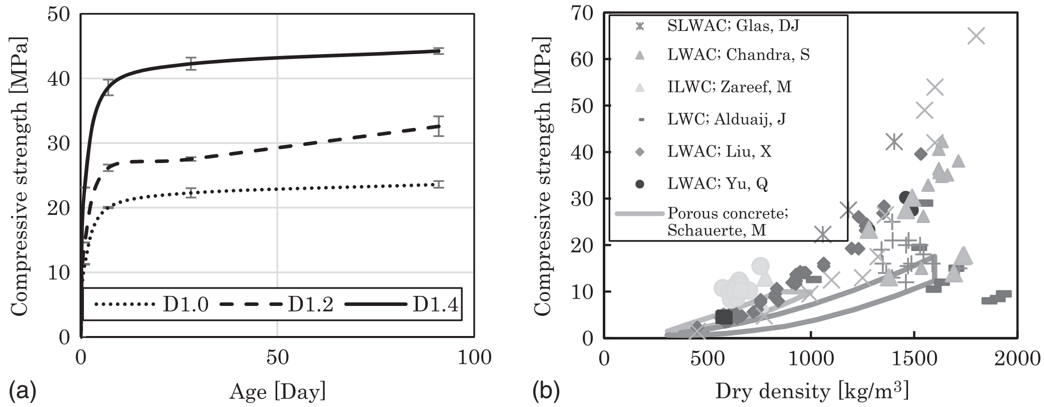


Fig. 6. Compressive strength: (a) development of the designed mixes; and (b) versus dry density for results taken from the available literature.

The calculated structural efficiencies of mixes D1.0, D1.2, and D1.4 were 18,760, 21,150, and 27,850 $\mathrm{N \cdot m/kg}$, respectively. It is evident that structural efficiency increased with the increase of the density class in the present study. In previous research, the structural efficiency of lightweight concrete using expanded glass as aggregates was studied (Yu et al. 2013), and 18,200, 20,260, and 18,456 $\mathrm{N \cdot m/kg}$ were observed for concretes with apparent densities of 1,280, 1,490, and 1,460 $\mathrm{kg/m^3}$, respectively. Choi et al. (2006) reported a structural efficiency ranging from 17,000 to 28,300 $\mathrm{N \cdot m/kg}$ for a semilightweight concrete with density ranging from 1,965 to 2,306 $\mathrm{kg/m^3}$. It is evident that the present lightweight concrete showed a clear improvement with regard to structural efficiency, especially at the higher density class. Structural efficiency was best for mix D1.4 in the present study; this may indicate that, for structural applications, the density class of D1.4 can be recommended. Fig. 6(b) shows the relationship between compressive strength and dry density for the developed concrete mixes, compared with values taken from the available literature. It can be clearly seen that the developed mixes had a better structural efficiency than the mixes reported in the available literature. This could be explained by the applied optimized packing design theory.

Fig. 7 presents the compressive strength versus the splitting tensile strength of the designed mixes in the present study, along with reported data from the available literature. In relation to the mixes reported in the literature, the splitting tensile strength of the developed mixes is higher. ACI 213 R-03 (ACI 2009) reports that the splitting tensile strength of LWAC ranges from 70% to 100% in comparison with normal weight concrete (NWC) with similar compressive strength. The higher splitting tensile strength can be explained by the curing method. In this study, the specimens were cured under moist condition with a RH over 90%, and the water absorbed by the expanded clay aggregates used during presoaking provided an internal curing effect; Hanson (1961) and Pfeifer (1968) have proven that this can lead to an improvement of approximately 20%. Another possible reason that the developed mixes achieved a high splitting tensile strength may have been due to the enhanced quality of the ITZ, relatively small maximum particle size, and optimized packing of the solid ingredients.

The modulus of elasticity of lightweight aggregate concrete depends on the modulus of elasticity of the matrix, the type of aggregates, the effective water-to-binder ratio, and the volume of the cement (Chandra and Berntsson 2002), but is primarily affected by the stiffness and volume of the aggregates (Zhang and Gjørv 1991). Fig. 8 shows the relationship between the 28-day compressive strength and the E-modulus of mixes using different aggregates

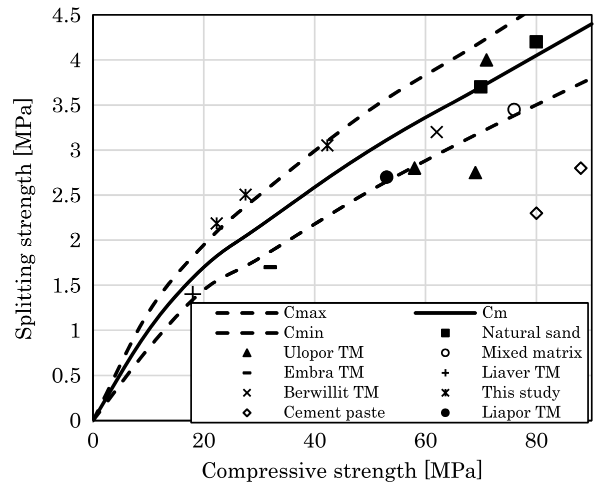


Fig. 7. Compressive strength versus splitting tensile strength.

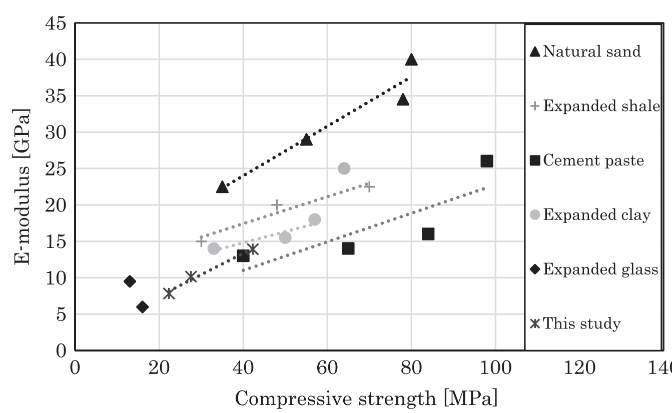


Fig. 8. Relationship between E-modulus and compressive strength.

(Chandra and Berntsson 2002). The E-modulus of the present mixes ranged from about 8 to 14 GPa. Zhang and Gjørv (1991) reviewed a number of E-modulus calculation methods, including Norwegian concrete code 3473 (NBR 1998) and ACI 318-83 (ACI 2008) and observed that the formula given by the Norwegian code predicts rather reliable values:

$$E_c = 9500(0.9\sigma_c)^{0.3}\left(\frac{\rho}{2400}\right)^{1.5} \quad (3)$$

where E_c = modulus of elasticity (MPa); σ_c = cube compressive strength (MPa); and ρ = apparent density (kg/m^3).

The Norwegian code also predicted the E-modulus fairly well for the present mixes, while other codes, such as Eurocode 2 or ACI code, slightly overestimated the elastic properties.

The aggregates primarily used in this study were natural expanded silicate with a low particle density in the range of 500–750 kg/m^3, which caused a relatively lower E-modulus (Chandra and Berntsson 2002). Nevertheless, this would contribute to a better resistance toward, for instance, earthquake effects, especially compared to normal weight concrete (Mo et al. 2017). It must be emphasized that other factors, including aggregates type, play an essential role as well (Dilli et al. 2015). Moreover, the mixes had a relatively high water-to-cement ratio (Table 3, especially mixes D1.0 and D1.2) and high air content (see the previous section). As seen in Fig. 8, lightweight concrete with a compressive strength between 20 and 40 MPa has an E-modulus of about 5–18 GPa using various types of aggregates, including expanded glass, expanded clay, and expanded shale (Chandra and Berntsson 2002). Using expanded clay and sintered fly ash, Zhang and Gjørv (1991) reported a relatively low E-modulus of 17.8–25.9 GPa in the density range of 1,600–1,900 kg/m^3. The developed lightweight concretes in the present study showed a similar E-modulus in the same strength range, although the density range in this study was 1,000–1,400 kg/m^3.

Drying Shrinkage

The drying shrinkage of concrete can be a considerable factor affecting the extent of cracking, effective tensile strength, prestress loss, warping, and so forth (ACI 2009). With relative humidity ranging from 40% to 100%, drying shrinkage is mostly caused by capillary tension occurring in the moisture existing in the pores in the cement paste (Chandra and Berntsson 2002). Fig. 9 shows the shrinkage development of the designed lightweight concretes, together with a number of LWACs and NWCs reported from the literature.

The developed mixes had shrinkage levels of 670, 870, and 900 microstrains. The hardened elements in concrete, including aggregates, play a role in restraining the drying shrinkage of cement paste (Choi 2017). Therefore, a lower E-modulus of concrete would result in a higher drying shrinkage due to reduced restraining capacity of the matrix. Note that the NWC and the Liapor 10 25% were vacuum cured for the first 7 days, leading to lower shrinkage levels. However, compared to other LWACs (e.g., NSB 035 and NSA 035) or to the study by ACI (1987), the present research shows clear potential. Considering the relatively low E-modulus, relatively high cement content, high porosity, lack of normal weight aggregates, and high water:cement (w:c) ratio, a higher shrinkage should be expected. Nevertheless, the developed mixes, especially mix D1.4, still performed better compared to other LWACs. This can be explained by the higher E-modulus and the fact that the expanded clay aggregates were presoaked 1 h before mixing. The prewetted lightweight aggregates were effectively used as internal curing agents, as confirmed by Lura (2005). In addition, curing conditions have a major influence on shrinkage. Beushausen and Bester (2016) reported that prolonged wet-cloth curing can effectively reduce shrinkage and increase resistance to cracking based on an evaluation of seven different curing regimes. Steam curing can also reduce shrinkage levels from 10% to 40% (ACI 2009). When considering curing conditions for concrete in precast elements, factors such as temperature and relative humidity can be adjusted in order to control reduced drying shrinkage.

Water Penetration under Pressure

Fig. 10 shows the results of the water penetration test on the developed mixes, and Fig. 11 shows cross sections of the mix samples, which were split after the experiments. Mix D1.2 had the highest permeability, with an average water penetration of 5.5 mm. D1.4 had the lowest average penetration depth of 2.1 mm; mix D1.0 had a penetration depth of 4.1 mm. Concrete with a less than 50-mm intrusion is regarded as impermeable (Reinhardt 2002); therefore, all the mixes developed in this study were impermeable. In comparison with data reported in the literature, these results were better than those for similar LWC matrices, indicating a wide range of applications for the developed mixes. Zhang and Gjørv (1990) reported that the higher the water penetrability of a given concrete, the higher the penetration of damaging species such as carbon dioxide, sulphate, and chloride ions into a concrete. Therefore, the depth of water penetration of a concrete can be used as an indicator of its durability. This indicates the excellent durability of the concretes developed in this study.

A low w:c ratio leads to a denser microstructure, which helps to reduce permeability. This explains the excellent permeability of mix D1.4. Furthermore, it is well known that the interfacial zone

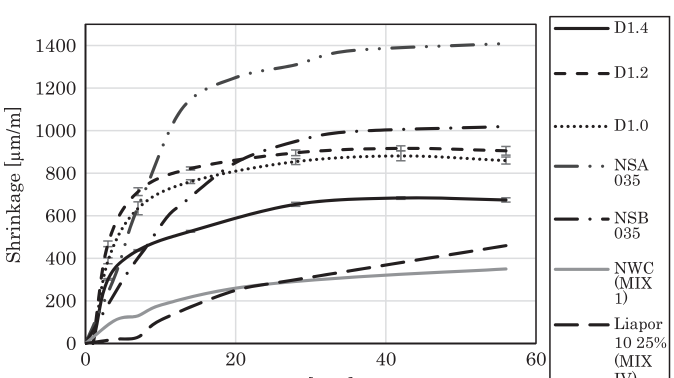


Fig. 9. Shrinkage of the developed mixes compared with fly ash aggregate LWAC (NSA 035 and NSB 035), NWC (mix 1), and NWC with 25% Liapor 10 (mix IV).

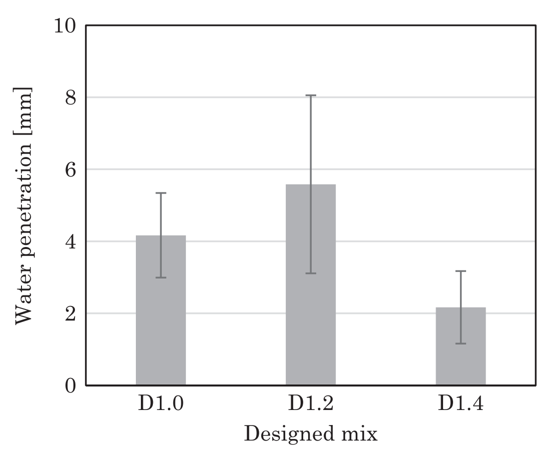


Fig. 10. Water penetration of the developed mixes.

Fig. 11. Cross sections after water penetration test (from left to right: D1.0; D1.2; D1.4).

between the cement paste and LWA plays a major role in the water permeability of an LWAC. In the case of mix D1.4, the 1-h presoak of the expanded clay aggregates potentially brought about an internal curing effect. Hence, the enhanced interfacial zone contributed to a reduced permeability. It has been reported that natural expanded silicate as a LWA leads to higher porosity (Dhir et al. 1989), but in the present study the results shown by mixes D1.0 and D1.2 were very good—especially, the results for mix D1.0, in which only natural expanded silicate was used as LWA. Mix D1.0, with the highest w:c ratio, showed only a very low water penetration of around 4 mm after 72 h of 5-bar water pressure.

In a previous study, Spiesz et al. (2013) developed three LWAC mixes with densities ranging from 1,280 to 1,490 kg/m^3. Two mixes, self-compacting lightweight composites, failed the water penetration depth under pressure by leaking from the side walls of the cubes, indicating high permeability, while the third mix, a vibrated cement-based lightweight composite, showed an average penetration depth of 6.4 mm. Compared with the mixes developed in this study, the powder contents and the w:c ratios were within the same ranges; however, the materials were different. Spiesz et al. (2013) used ordinary portland cement and a combination of normal weight sands and expanded glass; this study made use of a quaternary binder (consisting of blast furnace cement, fly ash, and limestone powder) and LWA consisting of natural expanded silicate and expanded clay. First, the blast furnace slag cement and pozzolanic fly ash used contributed to a densified microstructure (Biskri et al. 2017). Second, when considering binders of the same order of magnitude due to the same volume and w:c ratios, the applied LWAs play a decisive role. This can probably be explained by the natural expanded silicate aggregates used, which were surface treated with a hydrophobic agent, greatly enhancing the water permeability of the resulting concrete.

Freeze–Thaw Resistance

Fig. 12 shows the scaling development of the samples after 56 freeze–thaw cycles. For both the deionized water and the 3% NaCl samples, mix D1.0 was the most vulnerable. According to the Swedish Standard (2005), the strictest classification of the surface scaled material, by using deionized water, after 56 cycles should be lower than 100 g/m^2. For the use of deicing salt, the strictest classification is lower than 1,000 g/m^2, according to Boos and Giergiczny (2010). In both cases, mixes D1.2 and D1.4 fit into the strictest classes with regard to freeze–thaw resistance. Compared to the LWACs developed by Spiesz et al. (2013), which resulted in a scaling of 21–28 g/m^2 for mixes with densities ranging from 1,280 to 1,490 kg/m^3, the freeze–thaw resistance of the mixes in this study was significantly better. The measurements with the deicing salt resulted in more grouped results for D1.2 and D1.4 (600 and 700 g/m^2), while D1.0 had a significantly lower freeze–thaw resistance. This can be explained by its lower compressive strength and inadequate pore size distribution, as seen in Fig. 4.

According to EN 206 (CEN 2002), the freeze–thaw resistance of concrete is dependent on the following aspects: air void system,

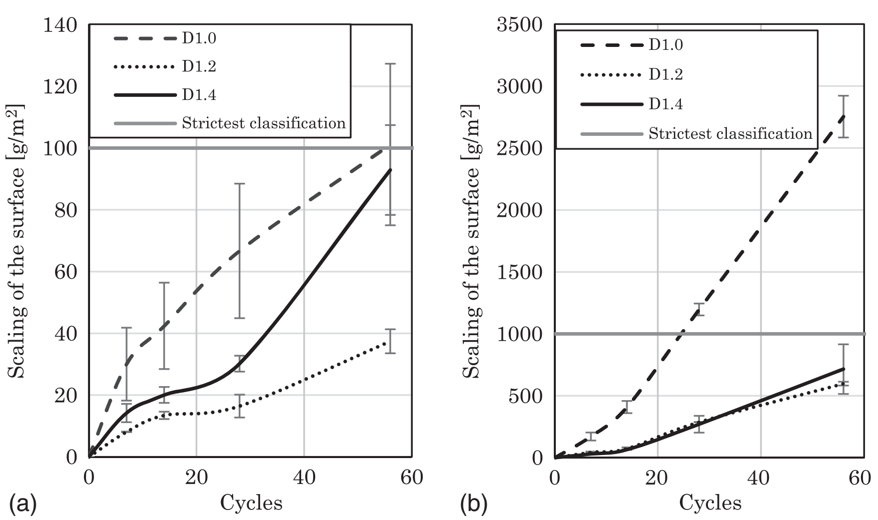


Fig. 12. Freeze–thaw resistance: (a) deionized water; and (b) 3% NaCl.

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cement content, w:c factor, and compressive strength. This is also applicable to the mixes developed in this study. When comparing the results for the water penetration depth under pressure and freeze–thaw scaling with data reported in the literature, a similar trend between permeability and freeze–thaw resistance can be observed. It is obvious that both factors are affected by the interconnectivity of the pore structure in the mix. Higher interconnectivity results in higher permeability, which leads to a higher freeze–thaw resistance.

Conclusions

This study investigated the physical and mechanical properties and durability of a new class of structural lightweight aggregate concrete, with extra attention focused on density class and cement structural efficiency. The effect of the hydrophobic natural expanded silicate aggregates used was thoroughly investigated. The excellent performance of the designed structural lightweight concrete indicates that it has broad potential for engineering applications, both for precast and in situ cast purposes. Based on the acquired results, the following conclusions can be drawn:

- By applying the optimized particle packing design mmmethodology, three mixes were developed with oven dry densities of about 1,000, 1,150, and 1,400 kg/m^3 (classes D1.0, D1.2 and D1.4, respectively). The developed LWACs showed excellent structural efficiency compared to similar LWCs, with 28-day compressive strengths of about 23, 28, and 42 MPa, respectively.
- The applied natural expanded silicate is recommended for structural lightweight concrete production due to its great density-to-strength ratio. These hydrophobic aggregates showed a high water repellent effect. However, the aggregate's negative influence on workability due to its irregular shapes, rough surface texture, and high specific surface area should still be considered during the mix design process.
- The very lightweight aggregates were distributed very homogenously through all the developed concretes. Scanning electron microscopy images showed that the developed concrete structure had a rather compact microstructure, which contributed to enhanced strength.
- The *E*-modulus of the developed concrete was fairly well-predicted by the Norwegian concrete code. The relatively low *E*-modulus of the lightweight concrete can help to increase resistance to certain damages, such as earthquake effects. Mix D1.4 showed relatively low drying shrinkage, which can be attributed to relatively low initial water use and the internal curing effect brought about by the applied lightweight aggregate.
- The developed mixes showed excellent durability. Mix D1.4 fit in the strictest levels of all environmental exposure classes, as indicated by a very low water penetration of 2.2 mm after 72 h exposure under a pressure of 5 bars and scaling of 90 and 700 g/m^2 using deionized water and NaCl solution, respectively, after 56 cycles of freeze–thaw testing.

Data Availability Statement

All data, models and code generated or used during the study appear in the published article.

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