Study on the production of core-shell structured lightweight aggregate by cold-bonding agglomeration process and its utilization in concrete

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Abstract

Lightweight aggregate concrete has been widely used in different construction applications due to its advantageous characteristics such as low density, low thermal conductivity and good durability properties. Compared to conventional concrete, lightweight aggregate concrete is made by replacing normal weight aggregate with lightweight aggregate, which is taken from natural resources or produced artificially. Artificial lightweight aggregate can be manufactured from natural materials or from industrial by-products via either sintering or cold bonding technique. The cold bonding method has the potential to reduce the energy consumption, lower the pollutant emission and requires low investment when compared with the sintering method. The agglomeration process via the pelletizer disc is the common-used method for the production of cold-bonded aggregate. Most of the research studies have focused on the importance of this method in recycling wastes into aggregate irrespective of the density of the aggregate produced, which is the most important property of lightweight aggregate.

The objective of the present research is to evaluate the use of an encapsulation process, which has been widely used in different industrial fields, in the production of low-density cold-bonded aggregate and to study the feasibility of using such kind of aggregate for the production of lightweight concrete. The first part of this research focusses on the development of production process of core-shell structured lightweight aggregates and discusses the influential factors on the manufacturing productivity and properties of the aggregate. Reducing the density of the shell structure is considered an option to produce low-density aggregate. In this concern, low-density powder mixture has been optimized to be used in the cover matrix to reduce the density of the manufactured aggregate as low as possible. In the second part of this research, the influences of different curing regimes on the microstructure and mechanical properties of the aggregate have been investigated to find out the most appropriate curing method. Furthermore, surface treatment for the core-shell aggregate has been employed to upgrade its properties by reducing its water absorption and enhancing its crushing strength. Finally, the mechanical and
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microstructural characteristics, thermal properties, durability and shrinkage of concrete made of core-shell structured lightweight aggregate have been investigated and compared with that made of expanded clay lightweight aggregate, the most popular type of sintered aggregate.

The results of this experimental study show that the encapsulation process can be successfully employed to produce a new kind of cold-bonded lightweight aggregate applicable for the production of structural and insulating lightweight concrete, which emulates the properties of expanded clay aggregate concrete, and thereby may have positive economic and environmental consequences.

Kurzfassung

Oberflächenbehandlung für die Kern-Schalen-Gesteinskörnung eingesetzt, um ihre Eigenschaften zu verbessern, wodurch die Wasseraufnahme reduziert und die Druckfestigkeit verbessert wurde. Schließlich wurden die mechanischen, mikrostrukturellen und thermischen Eigenschaften, die Dauerhaftigkeit und das Schwinden von Beton aus Gesteinskörnung mit Kern-Schale-Struktur untersucht und mit denen aus Blähtongesteinskörnung, der üblichen Sinterzuschlagart, verglichen.

Die Ergebnisse dieser experimentellen Studie zeigen, dass der Verkapselungsprozess erfolgreich eingesetzt werden kann, um eine neuartige kalt verbundene Gesteinskörnung herzustellen, die für die Produktion von tragendem und thermisch isolierendem Leichtbeton geeignet ist, der in den Eigenschaften vergleichbar ist zu Leichtbeton aus Blähtongesteinskörnung und dadurch positive wirtschaftliche und ökologische Auswirkungen haben kann.
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1. Introduction

1.1. General

Normal-weight concrete is one of the high demand and most widely used building material throughout the world. The popularity of normal-weight concrete (NWC) is attributable to its high compressive strength, high water and fire resistance, ease to be used and cast into a variety of shapes and sizes, as well as being cheaper and more available than other constructions materials [1]. Nevertheless, the high density and low thermal insulation performance of NWC is a crucial problem for several applications. It results in increasing the weight of the building structure, therefore, reducing its resistance against earthquake, since the earthquake effect is linearly dependent on the mass of the structure [2]. Moreover, the high density of NWC increases the dead load and thus, adversely affects the total cost of the structures especially in long-span bridges and tall buildings, where structural elements with large sections should be designed [3]. Therefore, the use of lightweight concrete helps to decrease the dead weight of the structures, which can substantially reduce the total cost of building as well as minimize the risk of earthquake damages [4].

Generally, lightweight concrete (LC) has many advantages in comparison to NWC; lower thermal conductivity which saves the high cost of thermal insulation required, superior sound and fire resistance, low transportation cost, minimal pressure on the formwork, and high implementation productivity [5,6]. Moreover, although the cost of cubic meter of LC may be more than that of NWC, its low density allows designers to decrease the cross-sections of the structural elements as well as the dimensions of the foundations, thereby reducing the total cost of building [7].

Historically, LC is known for over 3000 years. The Port of Cosa, the Pantheon Dome, and the Coliseum are the most outstanding LC structures which have been built during the early Roman Empire using LC made from natural volcanic materials [8]. In 1918, reinforced LC made of artificial lightweight aggregate had been used first in the construction of ships and barges. While the use of LC in the field of home construction was considerably developed in 1940. Different studies on the production of
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High-strength LC started in the early 1980s and ended in 1992 with the conclusion that the LC is feasible for special applications, where high compressive strength and high-durability performance are required [9].

1.2. Classification of lightweight concrete

The term lightweight concrete is used for concrete with a density between 800 and 1850 kg/m³, and between 800 and 2000 kg/m³ as specified in ACI 213R-87 [10] and EN 206-1 [11], respectively. LC can be produced through three ways: (1) by using porous aggregate with a specific density lower than 2 g/cm³. This type of LC is known as lightweight aggregate concrete; (2) by introducing air voids within the concrete using air entraining agent or pore-forming and gas-generating admixtures. This kind of LC is called as aerated, cellular, foamed or gas concrete; (3) by taking out the fine aggregate from the concrete mix and using only normal-weight coarse aggregate to generate large voids within the concrete structure and produce no-fines concrete [12].

The most popular type of LC is lightweight aggregate concrete (LWAC). It can be classified, based on the purpose of its utilization, into three types: (1) structural LWAC with a compressive strength > 17 MPa and density range of 1400 - 1800 kg/m³, which can be used for structural purposes; (2) low-density LWAC with a density range of 300 - 800 kg/m³ and compressive strength ranging from 1 to 7 MPa, which is normally used for thermal insulation purposes; (3) moderate strength LWAC, located between structural and low-density LWAC, with a strength range of 7 - 17 MPa. Figure 1.1 shows the classification of LWAC as specified in ACI 213R-87 [9].
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Between these types of LWAC, structural LC is the most versatile type due to the possibility of achieving an efficient balance between strength and density in combination with good thermal properties. Figure 1.2 illustrates an indication of the relationship between strength classes and the necessary dry density for LC made either by normal-weight or lightweight sand [13].

Figure 1.1. Classification of lightweight concrete according to ACI 213R-87 [10]

Figure 1.2. Relationship between strength and necessary dry density for LC [13]
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1.3. Types of lightweight aggregate

Some lightweight aggregates are naturally occurring; others are produced artificially from either natural raw materials or wastes/by products [14].

1.3.1. Natural lightweight aggregate

Most of popular natural LWA are of volcanic origins such as pumice, volcanic cinders, tuff and scoria. Volcanic rocks are produced by the release of gases during the solidification of lava. Natural LWA is made by crushing the volcanic rocks to achieve the optimum size and gradation. It generally has an irregular shape with a bulk density ranging from 300 to 1200 kg/m$^3$. As shown in Figure 1.1, this kind of LWA is suitable to produce LWAC with good mechanical properties and density ranging from 400 to 1800 kg/m$^3$.

1.3.2. Artificial lightweight aggregate

Due to the lack of availability of natural LWA in some countries as well as the limited performance of this kind of aggregate, local industrial by-products have been invested for the production of artificial LWA in which the properties of manufactured aggregate can be controlled [15]. Artificial LWA is produced either from natural raw materials such as clay, shale, slate, perlite and vermiculite or from waste and by-product materials like ground granulated blast furnace slag, waste glass powder, fly ash and quarry dust.

The most common artificial LWA produced from natural raw materials for non-structural purposes are expanded perlite and vermiculite. The world reserves of perlite are estimated to be around 700 million tons [16]. Perlite is a volcanic glass rock which contains 2 – 5 wt.- % water which has the distinguishing feature, that when it is heated, it expanded from four to twenty times its original volume forming expanded perlite aggregates with a bulk density of 60 - 80 kg/m$^3$ [17]. While vermiculite is a shiny flake of natural mineral formed by alteration of micaceous minerals such as biotite or phlogopite by weathering or hydrothermally. When the
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Vermiculite flakes are heated, they expand 8-20 times of their original size forming exfoliated vermiculite aggregate composed of very thin plates with air gaps in between having a bulk density of 65 - 160 kg/m$^3$ [18]. Due to their high porous microstructure, expanded perlite and exfoliated vermiculite aggregates are very weak and thus suitable only for nonstructural insulation purposes, as shown in Figure 1.1.

Some other artificial LWA produced from natural raw materials, through special manufacturing process, are expanded clay, shale and slate. The pore structure of these aggregates consist of uniformly distributed fine pores, and therefore, they are strong enough to be used in the production of structural LWAC [3]. The production of such kind of LWA begins with the quarrying and crushing the raw material into suitable size particles with irregular shapes, which are then fed to a rotary kiln for thermal treatments. In the rotary kiln, when the raw materials are heated to the point where a chemical reaction or physical change takes place, the particles swell up to several times of its original size forming a porous lightweight aggregate. This swelling occurs due to the generation of gas inside the particle, resulting from either the burning of organic compounds which exist in the chemical composition of the expandable raw materials or by using special expanding agents such as silicon carbide to induce the non-expandable materials to expand during the heating. When the expanding agent is heated, it decomposes into silicate and carbonate compounds. The release of gases at a high temperature causes expansion of the clay, leaving it with high porosity and low density, thus forming an expanded LWA [19]. Afterwards, the manufactured aggregates are cooled by water, then screened for size to get ready to put into practice.

Modern factories have been developed to produce spherical shape LWA, which are more favorable to enhance the consistency of fresh concrete. In this production line and after quarrying, the raw materials are crushed, ground and then agglomerated to form spherical fresh pellets with somehow a uniform distribution of fine pores. After that, the fresh pellets are introduced into a heat treatment process, as mentioned before, to produce rounded LWA. Leca and Liapor are the commercial names of the expanded clay aggregate produced in this way, which are widely used in Europe for structural and non-structural purposes.
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The excessively exploitation and depletion of natural resources as well as the high costs of waste disposal encourage the employment of different wastes and by-products as raw materials in the production of artificial LWA. Foamed blast furnace slag, sintered fly ash and expanded glass aggregates are the most important types of such aggregates. Foamed slag aggregate is made by either spraying the molten slag discharged from blast furnace or by rapidly extinguishing it with a controlled amount of water. In both methods, the generated steam puffs the slag and lets it solidify in a porous structure. Then the expanded blast furnace slag is crushed and sieved to obtain the required aggregates. While sintered fly ash aggregate is firstly produced by the agglomeration of fly ash powder into fresh spherical pellets using water and binders if necessary. The fresh pellets are then sintered at a temperature below the melting or softening temperature of the raw materials used. This sintering process hardens the green pellets through the coalescence between fly ash particles by fusing them together. The pore structure of sintered fly ash LWA results from the evaporation of the water used in the agglomeration and from the removal of carbon by combustion [20]. However, in the production process of expanded glass LWA, recycled glass powder, binding and expanding agent are firstly agglomerated to form spherical grains. The green grains are then sintered at a temperature of 800 - 900 °C to produce considerably low density LWA with excellent mechanical and thermal properties. Figure 1.3 shows the particles of expanded clay LWA, expanded glass LWA, perlite and vermiculite and their cross-sections under the optical microscopy.
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Despite the substantial use of sintered LWA for structural purposes, the high energy consumption as well as the emission of different pollutants emitted mainly by the rotary kiln are still dissatisfying from an economical and environmental point of view. Moreover, the sintering method is considered to be a highly sophisticated technique where many factors should be optimized to produce good-quality LWA. The sintering temperature is the most influential factor; it must be adjusted to be below the melting point of the raw materials. The properties of sintered LWA were also found to be affected by the grain size of raw material as well as the size of green pellets [21]. Therefore, research was carried out recently on the using of the cold bonding process, a relatively low-polluting and energy-efficient method, in the production of artificial LWA. The LWA manufactured by this method is known as cold-bonded LWA and fabricated by the agglomeration of moisturized powders into

![Figure 1.3. Microscopy images of expanded clay, expanded glass, perlite and vermiculite aggregate](image-url)
spherical fresh pellets. After that, the fresh pellets are cured, without thermal treatment, to gain an adequate strength to be used as an aggregate in practical applications. In spite of the advantages of cold bonding method over sintering method, the higher particle density of cold-bonded LWA, as well as its urgent need for an extended curing period to gain sufficient strength to be used practically, are the primary concern to expand its practical applications, as compared with sintered LWA.

1.4. Research objectives

Agglomeration process via pelletizer disc has been used in the production of cold-bonded aggregate in all related research topics. The main aim of those researches was to recycle various wastes and by-products materials into aggregates, regardless of their density or their feasibility in the production of lightweight aggregate concrete, which can substantially help to reduce the energy consumption of buildings. The production of low-density cold-bonded aggregate as well as its utilization in lightweight concrete have therefore been the main two objectives of this study. The originality of this work is to employ encapsulation technology, which is currently not applied in the field of cold-bonded aggregate production, to produce core-shell structured lightweight aggregate. The optimized core-shell aggregate should exhibit a particle and bulk density lower than that produced in literature through the agglomeration technique; meanwhile, it should meet the standard requirements of lightweight aggregate. The next objective of this work is to evaluate the characteristics of concrete made with core-shell lightweight aggregate in comparison with that made from expanded clay lightweight aggregate manufactured through sintering technique.

1.5. Research outline

This experimental research is presented in four scientific publications; three peer-reviewed journal papers and one conference paper. Information about the types of lightweight aggregate, production methods of artificial aggregate and the classification of lightweight concrete have been given in the introduction. Afterwards, state of
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knowledge includes an overview of the agglomeration process, and the main properties of both lightweight aggregate and lightweight aggregate concrete are presented. It explains, on the one hand, the mechanism of agglomeration and demonstrates the primary influential factors on its efficiency and, on the other hand, illustrates the main characteristic of lightweight aggregates and their effects on the behavior of lightweight aggregate concrete.

The main body of this thesis is presented in the peer-reviewed papers, as follow: In the first peer-reviewed paper, a comprehensive literature review is conducted and divided into two main parts. In the first part, a literature overview of the production process of cold-bonded aggregate is given. In this regard, factors influencing agglomeration efficiency and the properties of cold-bonded aggregates, such as pelletizer disc angle and speed, pelletization duration, water content and the types of raw materials and binders used, are deeply discussed. Moreover, different techniques for improving the performance of cold-bonded aggregate are also presented. The second part of this publication reviews the practical application of cold-bonded aggregate in concrete. In this aspect, the fresh, mechanical, microstructural and thermal properties, as well as the durability of cold-bonded aggregate concrete, are reported.

The second peer-reviewed paper focuses on the production process of core-shell structured lightweight aggregate using a pelletizer disc through an encapsulation approach. Being that both the kind of the manufactured aggregate and the production technique used in this study are new, the optimum angle and speed of the pelletizer disc as well as the binder content of the aggregate shell structure have been first determined. To achieve that, twenty-four types of core-shell aggregate have been manufactured and tested to evaluate their performance under the influence of the aforementioned parameters. After that, aiming to reduce the particle density of the aggregate produced as low as possible, six core-shell aggregate kinds have been produced by the incorporation of lightweight-mineral admixture in the cover matrix of the manufactured aggregates.

The utilization of core-shell structured lightweight aggregate in concrete as a replacement of normal-weight aggregate has been investigated in the conference
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In this concern, three concrete mixes have been prepared by replacing normal-weight aggregate with core-shell lightweight aggregate at replacement levels of 0, 50 and 100 vol.-%. Dry density, compressive strength and thermal conductivity of concretes were measured. The aim of this work is to give an indication about the feasibility of using such new kind of lightweight aggregate in concrete, as a preliminary step to an extensive study about the behavior of core-shell lightweight aggregate concrete, which has been implemented in the third peer-reviewed paper.

The third peer-reviewed paper is divided into two phases. The target of the first phase is to enhance the properties of the manufactured aggregates, by determining firstly the appropriate curing method and then by treating their surfaces with a dense thin film in order to improve the crushing strength and reduce the water absorption of the final product. In this investigation, the mechanical and microstructural characteristics of the aggregate were evaluated under the effects of three curing methods and two surface treatment techniques. Then, the second phase of this paper is carried out regarding the feasibility of using core-shell structured lightweight aggregate for the production of structural and insulating lightweight aggregate concrete. Mechanical characteristics, thermal properties and durability of six concrete mixes made of core-shell and expanded clay lightweight aggregate have been studied and compared.

Since shrinkage is considered to be an essential phenomenon in lightweight concrete, complementary experimental work has therefore been performed on the concrete mixtures designed in the third paper to measure and evaluate their shrinkage. Moreover, an additional investigation has been carried out to study the feasibility of using early age-core-shell aggregate in the production of lightweight concrete.

Finally, all the results and achievements presented in this work are collected and summarized at the end of this thesis, in addition to general conclusions and some recommendations for future research.
2. State of knowledge

2.1. Agglomeration technology

Agglomeration is a size-enlargement process of moisturized fines into larger granules or pellets. As the world is concerned about increasing the generation rates of waste, agglomeration is considered to be a highly efficient process for recycling a wide range of waste and by-product materials into high-value product [22]. Two granulation techniques are known; pressure and non-pressure. In the pressure technique, a mechanical compression is required to shape the raw materials into the desired form. The amount of moisture has to be accurately determined to avoid squeezing of the pellets. While the pelletizer disc is the widely used device to prepare granules via non-pressure agglomeration technique [23]. A photo of a typical pelletizer disc is shown in Figure 2.1. It consists mainly of the pan (A) which is equipped with: scraping blades (B) to remove the materials sticking on the bottom of the pan and let the pellets to move in different paths; spray nozzles (C) to moisturize the raw materials and screw (D) to adjust the pan angle.

![Figure 2.1. Photograph of pelletizer disc; A: the pan, B: scraping blades, C: spray nozzles, D: screw (©FEECO international, Green Bay, WI, USA)](image-url)
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In the pelletizer disc, the size enlargement is achieved by the collisions and coalescence of moisturized powders due to their rolling motion in the pan. The amount of sprayed water, as a bonding agent, is a crucial factor for the effectiveness of agglomeration. It cannot be determined without preliminary production trials, since the insufficient amount of water leads to weak cohesion forces between the fines and a high amount of water leads to the formation of muddy pellets instead of granules [24]. The mechanism of pelletization can be summarized as follows: The sprayed water forms a thin liquid film on the surface of the fine particles and holds them together due to the surface tension of the liquid bridge. Then, due to the rotational motion of moisturized particles in the pelletizer, the mechanical forced contact between grains enhances bonding between them, leading to the size enlargement and thus to the formation of spherical pellets [23]. As shown in Figure 2.2, based on the water amount used through the pelletization, three stages of pellet formation can be defined. In the first stage (Figure 2.2 a), the amount of water is just enough to form the liquid bridges between particles at the contact points, and then it becomes a little bit higher in the second stage to be able to fill some of the pores between these particles as shown in Figure 2.2 b. Afterward, in the third stage (Figure 2.2 c), water completely fills all the pores between the particles, enabling the highest tension force between them and therefore, achieving the best state of fresh pellet [25].

![Figure 2.2. Stages of pelletization [25]](image)

In addition to moisture content, and since the rotational motion of the particles in the pelletizer is generated by the centrifugal and gravitational forces, the pelletizer angle and speed definitely affect the pelletization efficiency and the properties of the final products. On the one hand, at a high rate of speed, the movement of particles is
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subjected to the dominance of centrifugal force, and thus, they stay stuck to the side-walls of pelletizer, while the gravitational force becomes dominant and hinders the pelletization when a low speed is used. Therefore, the optimum pelletizer speed is that required to avoid the dominance of either gravitational or centrifugal force on the particle’s movement to ensure good collision between them and thus enhance the pelletization efficiency and the quality of the final product. On the other hand, the pelletizer angle affects the movement paths of particles over the pan; the higher angle increases the movement paths of particles producing, therefore, larger pellets and vice versa. Accordingly, the angle and speed of the pelletizer and the interaction between them have a significant influence on the agglomeration process.

However, Figure 2.3 shows the typical movements and the growth-path of pellets in the pelletizer. In path 1, the fines are lifted to the top of the pan and fall back down to the lower portion; in this path, the growth mechanism is started. Then the small granules move to the path 2 and grow up gradually to shift due to the size-segregation effect to path 3 where the final pellets can be discharged from the pan.

![Figure 2.3. Grow-paths of pellets in the pelletization disc [26]](image)

After the agglomeration, the fresh pellet should be treated to gain the desired properties to be used as an aggregate in construction applications. As mentioned before, two post-treatment methods are known and used: sintering and cold bonding. In the sintering technique, the fresh pellets are heated to a temperature below the melting or softening temperature of raw materials used. During the sintering, the fresh pellets are first dried, and therefore, the surface tension forces between the fine
2. State of knowledge

Particles disappear before the beginning of sintering. Therefore, to counteract this problem and protect the dry pellets from being damaged during the sintering, binders or special additives should be used through agglomeration to ensure sufficient bonding forces during the time interval between the dry and sintered state [27]. This procedure increases the cost of sintered aggregate, which is already high due to the large energy consumption of this process. While the cold bonding method depends mainly on using binders in the agglomeration process and employing economic post-treatment methods compatible with the raw material used.

2.2. Encapsulation process

Encapsulation is an essential process in different industrial fields such as pharmaceutical, food, and fertilizer industries. It is used for several purposes such as enlarging small particles, improving the shape of irregular granules, protecting the encapsulated elements from the ambient effects and improving their performance [28]. The pelletizer disc provides a simple process in which the application of powder coatings can be employed to produce core-shell capsules. The cores are placed into the rotating disc and then sprayed with powders to apply coating layers until achieving the desired size and shape of the capsules. The growth of capsules and the binding mechanism of the encapsulation process is somehow similar to that of agglomeration, which has been discussed before [27].

2.3. Characteristics and test methods for lightweight aggregate

Lightweight aggregate constitutes about 70 - 80 % of the volume of lightweight aggregate concrete. Due to this large volume fraction, the quality of LWA has significant importance and is considered to be the major influential factor on the characteristics of LWAC. The main properties of natural LWA, such as density, porosity, strength and the chemical composition depend entirely on the properties of the source material, which can vary from place to place. While the quality of artificial
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LWA can be controlled during the manufacturing process [29]. In the following subsections, the main characteristics of LWA, their standard testing methods and their influences on the performance of fresh and hardened LWAC will be presented.

2.3.1. Particle density and water absorption

Particle density of aggregate is defined as the mass of a unit volume of material including the pores existing within the aggregate particles, excluding the voids between them. Therefore, for proportioning concrete mixtures, determining the density of the aggregate is required to know its occupied volume in concrete. The particle density of coarse aggregate is usually measured by the pycnometer method as specified in EN 1097-6, Annex C [30] or through the wire basket method according to ASTM C 127 [31]. In this research, the oven-dry and saturated surface dry particle density was measured by the pycnometer method in accordance with EN 1097-6, as shown in Figure 2.4, and calculated using equation 1 and equation 2.

![Figure 2.4. Determining the particle density according to EN 1097-6 [29]](image)

Saturated-surface dry particle density

\[
\rho_{SSd} = \rho_w \frac{M_1}{M_1 - (M_2 - M_3)} \quad \text{Equation 1}
\]

Oven-dry particle density

\[
\rho_{r.d} = \rho_w \frac{M_4}{M_4 - (M_2 - M_3)} \quad \text{Equation 2}
\]
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Where: $M_1$ is the mass of the saturated-surface dried aggregate sample in the air after immersion for 24 h (grams); $M_2$ is the mass of the pycnometer containing the saturated sample and water (grams) after immersion for 24 h; $M_3$ is the mass of pycnometer containing only water (grams) and $M_4$ is the mass of the oven-dried sample in air (grams).

Due to the porous structure of LWA, they tend to have high water absorption. Measuring the water absorption of LWA is important to determine the total amount of water required in the concrete mixtures. Equation 3 has been implemented to calculate the water absorption at 24 h ($WA_{24}$), as a percentage of its dry mass (wt.-%).

$$WA_{24} = \frac{100 \times (M_1 - M_4)}{M_4}$$

Equation 3

The rate of water absorption of LWA is relatively high in the first hour and affects the proportioning, handling and workability of concrete. It is therefore essential to investigate the water absorption of LWA over time especially when dry LWA is used in concrete. On the one hand, when the water absorption over time is known, a correlation between concrete consistency and the effective water content can be demonstrated as a function with time [32]. On the other hand, it indicates the time needed to compact the concrete without affecting its properties; if the concrete is compacted before the completion of water absorption by LWA, voids will be generated in the concrete due to the desiccation and adversely affects its strength [12]. The water absorption rate at several times ($WA_i$) can be calculated as a percentage of the dry mass (wt.-%), by equation 4.

$$WA_i = WA_{24} - \frac{(M_2 - M_i)}{M_4}$$

Equation 4

Where, $M_i$ is the mass of the pycnometer, aggregate sample and water in grams at the measuring time ($i$).

The fresh properties of LWAC are affected by the particle density and water absorption of LWA. The difference in specific gravity between the LWA and mortar increases the possibility of segregation in fresh mixtures; this can be avoided by the
use of viscosity-modifying admixture (stabilizer) which enhances the stability of fresh concrete [33]. While the high water absorption of LWA can cause a rapid slump loss of fresh concrete, this problem can be overcome by the utilization of saturated-surface dry LWA to protect fresh concrete from the reduction of water/cement ratio resulted from the high absorption of dry LWA [34].

Moreover, the use of dry LWA significantly affects the pumpability of the LWAC. The penetration of water into dried LWA caused by pumping pressure reduces the workability of concrete, making it stiffer, which increases the friction between the concrete and the inner walls of the pump line. Furthermore, the movement of water from paste into LWA reduces the volume of fresh concrete, thus affecting the mechanism of pumping [35].

On other side, the high water absorption of LWA has been found to have serious positive impacts on the compressive strength, microstructure and durability of LWAC due to the internal curing phenomenon, which will be discussed in detail later [36].

**2.3.2. Loose bulk density**

The loose bulk density or the unit weight of LWA is the mass of a unit volume of aggregate particles, including the voids between them as well as the pores within the particles. The filling ability of the aggregate is mainly influenced by its grading and shape. For the same grading and particle density, the loose bulk density of rounded LWA is less than that of angular LWA by about 80 kg/m$^3$ [29]. According to the requirements of ASTM C 330 and C331 [37,38], LWA should have a maximum dry-loose bulk density of 1120 and 880 kg/m$^3$ for the fine and coarse aggregate, respectively. Whereas, as specified in EN 13055-1, it should exhibit a loose bulk density $< 1200$ Kg/m$^3$ to be classified as LWA. In this research, the loose bulk density of the core-shell LWA was measured as shown in Figure 2.5 and calculated following EN 1097-3 [39], using equation 5.
2. State of knowledge

\[ \rho_b = \frac{M_2 - M_1}{V} \]

Equation 5

Where, \( \rho_b \) is the loose bulk density in kg/m\(^3\); \( M_2 \) is the mass of the container and test sample in kg; \( M_1 \) and \( V \) are the mass and volume of empty container in kg and m\(^3\), respectively.

2.3.3. Bulk crushing resistance, grading and surface texture

The mechanical performance of LWAC is controlled by the water-cement ratio of the mixture, the particle density, volume content and bulk crushing resistance of the LWA used [40]. LWA is often the weakest component of LWAC; it is weaker than the cement matrix and the interfacial transition zone. The crushing resistance of LWA is, therefore, the primary factor affecting the compressive strength of LWAC [41]. Crushing strength of aggregate particles can be measured by performing the crushing test either on single aggregate particle or on the aggregate particles in bulk state. Since the single pellet strength might be influenced significantly by the chosen particle, its shape and density, the crushing resistance test has been performed in this study on the aggregate in bulk state as stated in EN 13055-1 [42] in order to demonstrate the strength of the whole aggregate produced. Details of measurements procedures can be found in the mentioned standard.

The requirements and testing method of grading for LWA are similar to that of normal-weight aggregate. As in conventional concrete, grading of the fine and coarse fraction of the LWA is a major influential factor on the cost, fresh and hardened
2. State of knowledge

properties of LWAC [10]. The use of very coarse sand leads to unworkable concrete, while the use of very fine one increases the needed amount of water, thus the required amount of cement, which contributes to rising concrete cost. It has been reported that the use of well-graded coarse aggregate with a suitable portion of graded sand helps to produce workable and economical LWAC [3].

The natural and artificial LWA may have a regular, irregular, elongated, flaky or rounded shape as well as different surface textures based on its sources and production methods. The shape of LWA affects not only the workability and pumpability of fresh concrete but also the proportioning of a concrete mixture. It is well known that the concrete produced with rounded aggregate has higher workability than that made of irregular one. Therefore, to obtain the same workability of irregular-aggregate concrete, either the amount of cement paste or the dosage of the superplasticizer can be decreased in the case of rounded-aggregate concrete, which might affect the cost positively [43]. Moreover, the surface texture of LWA may also differ due to its type. Some LWA has a relatively smooth surface; another has a rough surface texture with small or large pores. The surface texture of LWA directly influences the contact zone between the aggregate and the cement mortar. The penetration of fresh cement paste into the surface pores of the aggregate develops a mechanical interlocking between them, thus improving the performance of concrete [44].

2.4. Characteristics of lightweight concrete

2.4.1. Mechanical properties

Since the density and compressive strength of concrete are highly interrelated, lightweight aggregate concrete has been classified in standards based on these two properties. As described before in section 1-2, ACI 213R-87 categorizes three types of LC based on the density, compressive strength and the purpose of the application. While EN 206-1 defines LC as concrete with an oven-dry density ≥ 800 kg/m³ and ≤ 2000 kg/m³ and classifies it by density into six classes, and by compressive strength into fourteen grades, as shown in Figure 1.2.
2. State of knowledge

Generally, for the same strength of concrete, and due to the weakness of LWA compared with NWA, the cement content of LWAC is higher than that of normal-weight concrete. LWAC has, therefore, a low water/cement ration and high-strength cement matrix compared with NWC. The compressive strength of LWAC is a combination of aggregate strength, mortar strength and adhesion strength between the LWA and the cement matrix. Since the transmission of loads in concrete depends on the rigidity of its components and being that the LWA is weaker and softer than the cement matrix, the transfer of load in LWAC is mostly happening through the cement matrix, and the concrete failure takes place through the LWA. While in conventional concrete, the load-transmission mode is through the aggregate skeleton, which is more rigid than the cement matrix [45]. Therefore, the mechanical properties of LWAC depend mainly on the stiffness of the LWA. Moreover, as in normal-weight concrete, superplasticizer and supplementary cementitious materials can be incorporated in LWAC mixtures to enhance the compressive strength of hardened concrete. In this experimental research, the density of concrete was determined as specified in EN 12390-7 and an automatic machine from Toni Technik GmbH was used to measure its compressive strength in accordance with EN 12390-3.

The modulus of elasticity of concrete is defined as the ratio between the stress and strain of concrete in the elastic stage before starting the permanent deformation, which is known as plastic strain. Similar to compressive strength, the modulus of elasticity of LWAC is a function of the modulus of elasticity of its ingredients. As the elastic modulus of LWA is lower than that of NWA, LWAC exhibits a lower modulus of elasticity than NWC. This means that the LWAC is more flexible and thus less stiff compared to NWC, which justifies its use in special applications where a high dynamic response is required and in some elements where a low stiffness is desirable from a structural point of view [10]. Moreover, the modulus of elasticity of LWAC depends on the elastic modulus and volume content of LWA and can be expressed, as specified in different standards, as a function of concrete density and compressive strength [29].

As mentioned before, LWAC generally has a low water/cement ratio and high cementitious materials content compared to NWC. Drying and autogenous shrinkage of LWAC resulted from the environmental drying and cement hydration might be
2. State of knowledge

therefore higher than that in NWC. The loss of concrete moisture to the environment as well as the self-desiccation of concrete cause the concrete to shrink, thus generate tensile stress which leads to cracking when it exceeds the tensile strength of concrete itself [46]. In order to compensate the moisture loss and thus overcoming the shrinkage and cracking in LWAC, the use of pre-soaked LWA as a water reservoir in the concrete mixture has been recommended. The use of saturated LWA not only does not affect the initial water/cement ratio and the workability of fresh concrete but also provides internal curing as the cement paste hydrates and dries due to the consumption of initial mix water. When the self-desiccation of the concrete takes place, the retained water within the LWA is released through the aggregate pores, in a process called desorption, and travels to fill the pores of the hardened paste, which reduces the tensile stresses caused by the drying shrinkage, thereby minimizing the possibility for cracking [47]. Therefore, the desorption behavior of LWA is a crucial factor for successful internal curing of LWAC. It depends mainly on the pore structure of the LWA; the larger the pore size, the higher is the desorption rate of LWA. Moreover, the effectiveness of internal curing has found to be related to the flow distance of the retained water into the surrounding paste, which also depends on the structure of the cement matrix itself [48]. Internal curing of LWAC caused by the incorporation of pre-saturated LWA allows the water reserved in the LWA to be transferred to the cement paste throughout the cross-section of the concrete. It is, therefore, more beneficial than the conventional external curing where the provided water penetrates the surface of concrete for only a small depth [49]. However, sometimes, the internal curing is not sufficient to solve the shrinkage problem and should be associated with external curing to achieve a significant reduction in both drying and autogenous shrinkage [20].
2. State of knowledge

2.4.2. Thermal conductivity

Thermal conductivity ($\lambda$) of a material is a measure of its ability to transfer heat passing through a unit thickness perpendicularly to a unit area for a temperature gradient of one degree; $\lambda$ is expressed in the unit of watt per meter kelvin [W/(m·K)]. Thermal conductivity of concrete is affected by the thermal properties of its ingredients. Being the aggregate comprises about 70 - 80 % of the concrete volume, and due to the porous structure of LWA compared to NWA, thermal properties of LWAC are superior to that of conventional concrete. Many researchers have reported that the thermal conductivity ($\lambda$) of LWAC is correlated to its dry density ($\rho$) [50]. Thermal conductivity of LWAC with a density range of 320 - 1600 kg/m$^3$ can be calculated, according to ACI 122R-02 [51], as a function of the logarithm of $\rho$ (kg/m$^3$) using equation 6.

$$\lambda = 0.072 \ e^{0.00125\rho}$$

Equation 6
3. References


3. References


3. References


[51] ACI Committee 122, Guide to thermal properties of concrete and masonry systems, ACI 122R-02.
4. Publications

4.1. The production and properties of cold-bonded aggregate and its applications in concrete: A review

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Review

The production and properties of cold-bonded aggregate and its applications in concrete: A review

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Abstract

The rapid increase in waste quantities, as well as the depletion of the natural resources in the near future are some of the major concern worldwide. In response to these problems, as well as to the growing demand for concrete in the construction field, the focus has been directed towards the production of artificial aggregates, as an alternative to natural ones. Artificial aggregates are produced either by sintering or cold bonding method. Compared with the sintering method and besides being an effective recycling solution for a wide variety of wastes and by-product materials, the cold bonding method is characterized by minimal energy consumption, low pollutant emissions and low investment requirements. The use of cold-bonded aggregate as a potential construction material in concrete production is summarized in this manuscript. The paper discusses factors influencing manufacturing productivity and the properties of cold-bonded aggregate, such as pelletizer disc angle and speed, pelletization duration, water content and the types of raw materials and binders used. The physical properties of cold-bonded aggregates made of different waste materials and binders are presented. The mechanical characteristics, thermal properties and durability of concrete made of cold-bonded aggregates are also reviewed. The literature in the field has established the potential of using cold-bonded aggregates as sustainable materials in the production of normal-weight concrete, as well as structural and nonstructural lightweight aggregate concrete.

Keywords:
Pelletization
Cold-bonded aggregate
Lightweight aggregate concrete
Mechanical properties
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Abbreviations:
BS, bulk crushing strength; Ca(OH)2, calcium hydroxide; CBA, cold-bonded aggregates; CBAC, cold-bonded aggregate concrete; CBCA, cold-bonded coarse aggregate; CEM, cement; CKD, cement kiln dust; CR, crumb rubber; C-S-H, calcium silicate hydrate; DDS, desulfurization device sludge; EPP, expanded perlite powder; FA, fly ash; GGBFS, ground granulated blast furnace slag; GQD, granite quarry dust; HP, hydrogen peroxide; ITZ, interfacial transition zone; LBD, loose bulk density; LWA, lightweight aggregate; LWAC, lightweight aggregate concrete; MS, marble sludge; MSB, medium swelling bentonite; MSWI, municipal solid waste incinerator fly ash; NA, normal aggregate; Na2SiO3, sodium silicate; Na2SO4, sodium sulfate; NAC, normal aggregate concrete; NAOH, sodium hydroxide; NS, nano-silica; NWCA, normal weight coarse aggregate; NWS, normal weight sand; OD, oven-dry; PCS, particle crushing strength; PD, particle density; PPF, polypropylene fiber; PS, particle crushing load; PSA, paper sludge ash; RH, relative humidity; RHA, rice husk ash; SCC, self-compacting concrete; SCM, supplementary cementitious material; SEM, scanning electron microscopy; SF, silica fume; SIA, sintered aggregate; SSD, saturated-surface dry; TPFV, ten percent fines value; WAB, water absorption; WAS, washing aggregate sludge; WTS, wastewater treatment sludge.

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1. Introduction

The construction industry is considered to be one of the most important indicators of a country's economic state [1]. Since concrete is the main and most widely used material in the civil engineering field, concrete production can be regarded as one of the biggest causes of both environmental pollution and excessive resource consumption [2]. Consequently, on one hand, particular attention has been paid to the incorporation of different by-products and waste materials, such as fly ash, granulated blast furnace slag and silica fume as cement replacements in order to decrease the amount of cement used, thus reducing the huge CO2 emissions stemming from cement production [3]. On the other hand, being that aggregate makes up about 70–80% of the total volume of concrete [4], the use of artificial aggregates manufactured from waste and by-product materials, as an alternative to natural aggregate, has attracted considerable research interest. This trend has many positive environmental consequences, including (1) the preservation of natural resources; (2) saving energy consumed by quarry processes; (3) converting waste into value-added products [5].

Generally, artificial aggregate is produced firstly through the agglomeration of powdered waste materials into fresh pellets with the desired size [6]. The fresh pellets are then hardened, either by autoclaving, sintering or cold bonding processes, to obtain the strength required for practical applications [7]. A detailed review of sintered fly ash lightweight aggregate and its utilization in concrete can be found in a publication by Nadesan et al. [8]. This paper aims to review knowledge about cold-bonded aggregates and their implementation in concrete. This review paper is outlined as follows: (1) the production process of the cold bonding method and its impact on the properties of the aggregate produced; (2) raw materials used in the manufacture of cold-bonded aggregates and their effects on the properties of the final product; (3) the physical properties of cold-bonded aggregates, as found in the literature; (4) the fresh, mechanical, microstructural and durability properties of cold-bonded aggregate concrete.

2. The production process of cold-bonded aggregates

Agglomeration is the first step in the production of cold-bonded aggregates (CBA) utilizing waste and by-product fine materials. This is the process of upgrading fines into larger particles, through either pressure or non-pressure agglomeration techniques [9]. Pressure agglomeration is used to shape the fines into the desired form through mechanical compression. While the pelletization process is a common and well-known method of non-pressure agglomeration, it enlarges the moistened fines into spherical pellets through their collision and coalescence, resulting from their rolling movement in the pelletizer disc [10]. The pelletizer disc must therefore be adjusted at a specific angle and speed to avoid the dominance of either gravitational or centrifugal forces on the movement of fines in the pelletizer. The adjustment of the angle and speed of the pelletizer ensures good collusion between the fine particles, resulting in a high production efficiency. Table 1 illustrates the production parameters used in the literature, including angle, speed, dimensions of the pelletizer and the pelletization duration; all of these being dependent on the raw materials used. The curing of fresh pellets is the second step in the production process of CBA, helping them to gain adequate strength for use as an aggregate in concrete applications.

3. Raw materials

Table 2 presents the raw materials, binders and additives used by researchers in the production of CBA. It is evident from this table that fly ash (FA) is the most commonly used material in CBA production [11–40]. The reason for this prevalence is the availability of this kind of waste material in huge quantities throughout the world, along with the urgent need to recycle it into a valuable product [41]. In addition to fly ash, different local industrial waste materials have also been employed in the production process of CBA, including wastewater treatment sludge (WTS) and desulfurization device sludge (DDS) [42], granite quarry dust (GQD) [43,44], ground granulated blast furnace slag (GGBFS) [45–47], rice
Special additives have been used by many researchers to meet specific performance requirements for the aggregates produced. Polypropylene fiber (PPF), crumb rubber (CR) and nano-silica (NS) have been utilized to enhance the strength of CBA\[53,57\]. Moreover, hydrogen peroxide (HP) as a foaming agent and expanded perlite powder (EPP) as a mineral admixture have been employed alternative binders. Alkaline activator, a composition of sodium silicate (Na$_2$SiO$_3$) and sodium hydroxide (NaOH), has therefore been incorporated into production processes to produce geopolymier CBA\[22,45,48,55,56\].

The binder is an essential element in the pelletization process, especially when a raw material with little or no cementitious properties is pelletized. As can be seen in Table 2, beside cement (CEM), which has been widely used in the literature, bentonite, glass powder, lime and clay binders have also been utilized as binders\[13,14,20,21,24,32,34,39,42,54\]. Moreover, the environmental impact of cement manufacturing has prompted researchers to employ alternative binders. Alkaline activator, a composition of sodium silicate (Na$_2$SiO$_3$) and sodium hydroxide (NaOH), has therefore been incorporated into production processes to produce geopolymier CBA\[22,45,48,55,56\].

Table 3 summarizes the physical and mechanical properties of the CBA produced in previous studies. It can be seen from this table that the size, particle density (PD), loose bulk density and water absorption of CBA produced in the literature are in the range of 4–20 mm, 0.88–2.12 g/cm$^3$, 510–1247 kg/m$^3$ and 2.5–77%, respectively. It is evident that most of the aggregate produced has exhibited an oven dry particle density <2 g/cm$^3$ or a loose bulk density <1200 kg/m$^3$; it can therefore be classified as lightweight aggregate, in accordance with EN 13055-1 \[62\]. The bulk crushing strength and ten percent fine value varied from 0.2 to 15.7 MPa and 0.25 to 5 ton, respectively, with individual particle strengths in the range of 30–2210 N and 0.4–22.8 MPa. The scattering results of particle strength, even within the same study, are attributed directly to the shape, size and density of the chosen particle \[64\], thereby, the bulk crushing strength test can be considered a reasonable estimate of the strength of the whole aggregate produced. Importantly, the properties of the CBA illustrated in Table 3 support arguments for the effectiveness of using the cold bonding method for producing lightweight aggregate, which may be used as a sustainable material in concrete production; this is discussed in detail below.

In this section, the difference between properties of CBA and sintered aggregate (SIA) produced from the same materials will be reported. Table 4 indicates that the CBA was observed to exhibit a somewhat higher bulk density, associated with higher absorption and lower crushing strength, as compared to SIA. However, the inferior performance of CBA can be justified by two main consider-
lations. One is related to the negative environmental impact of the sintering method due to the emission of a high amount of pollutants emitted from the burning of the raw materials in the rotary kiln. The other consideration concerns the high energy consumption of this method, where the raw materials are heated at a temperature below their melting point, which negatively affects the cost of the final product. These negative environmental and economic impacts of the sintering method substantially strengthen the advantages of employing the cold bonding technique in the production of artificial aggregates.

5. Factors affecting manufacturing efficiency and the properties of CBA

It is clear from Table 2 that the authors produced CBA using different raw materials, binders and water contents. They also used various pelletizer angles and speeds as well as different pelletization durations, as shown in Table 1. The subsequent section reviews the effects of these variables on production efficiency and the properties of the aggregate produced.
Table 3
Physical properties of cold-bonded aggregate (*RT: room temperature).

<table>
<thead>
<tr>
<th>Ref</th>
<th>Size (mm)</th>
<th>LBD (kg/m³)</th>
<th>SSD-PD (g/cm³)</th>
<th>OD-PD (g/cm³)</th>
<th>WAB (wt-%)</th>
<th>TPFV (ton)</th>
<th>PCS (MPa)</th>
<th>PS (N)</th>
<th>BS (MPa)</th>
<th>Method of curing</th>
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<td>510–650</td>
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<td>21.15</td>
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<td>19–23</td>
<td>2.5–3</td>
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<td>1.1–1.7</td>
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<td>16–21</td>
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<td>6.15–8.32</td>
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<td>1.56–2.15</td>
<td>4–20</td>
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<td>-</td>
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<td>0.8–2.2</td>
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<td>5–20</td>
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<td>-</td>
<td>-</td>
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<td>11–13.5</td>
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<td>-</td>
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<td>1.85</td>
<td>1.62</td>
<td>16.39</td>
<td>-</td>
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<tr>
<td>[76]</td>
<td>10–12.5</td>
<td>-</td>
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<td>14–20.5</td>
<td>1.92–2.8</td>
<td>-</td>
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<tr>
<td>[84]</td>
<td>4.75–12.5</td>
<td>995</td>
<td>1.98</td>
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<td>-</td>
<td>20.46</td>
<td>2.88</td>
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<td>-</td>
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<tr>
<td>[97]</td>
<td>4.76–12.7</td>
<td>857–972</td>
<td>1.65–1.76</td>
<td>1.23–1.44</td>
<td>20–35</td>
<td>-</td>
<td>6–8.57</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>[106]</td>
<td>0–8</td>
<td>920</td>
<td>-</td>
<td>-</td>
<td>57.8</td>
<td>-</td>
<td>0.96</td>
<td>-</td>
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</tr>
</tbody>
</table>


5.1. Effects of production parameters

Productivity is an essential indicator of the operational efficiency of the cold bonding process. This is the ratio between the mass of the aggregate produced with a size >4 mm and the total mass of basic powders incorporated in production [69]. Few researchers have studied the effects of production parameters on the productivity of the cold bonding process. Production efficiency has been observed to be highly dependent on the interaction between the angle and speed of the pelletizer, when no binder is used, with the speed and pelletization duration becoming the main influential factors when kaolinite is used as a binder [67]. This means that the influences of mechanical parameters on pelletization efficiency are also related to the materials used. Manikandan and Ramamurthy have reported that the use of low angles and speeds reduces the movement paths of particles in the pelletizer and decreases the collisions between them, thus leading to the formation of small-size aggregates with insufficient efficiency [34].

Besides aggregate size, other properties of CBA have been observed to be highly affected by production parameters. Colangelo and Cioffi have produced cold-bonded aggregate using different rotation speeds (35, 45 and 55 rpm) at a constant angle of 50\(^\circ\), finding that the highest density and the highest strength of CBA are achieved at a speed of 45 rpm and not at the maximum speed of 55 rpm. The interaction between this angle and speed contributes strongly to achieve best movement and collision of particles, thus improving the structure of the aggregate produced [52]. Tajra et al. used three speeds (20, 30, and 40 rpm) and two angles (35\(^\circ\) and 40\(^\circ\)) in their study. The authors observed that, at any fixed angle, the aggregate produced showed a clear increase in its particle density and bulk crushing strength with an increase in the speed of rotation. Thus, they concluded that the higher the rotation speed, the denser the aggregate structure [11]. These observations have been confirmed by Harikrishnan and Ramamurthy, where speeds of 20–40 rpm and angles of 40\(^\circ\)–70\(^\circ\) were used. They reported that rotation speed was the major influential factor on the strength and water absorption of the aggregate; due to the formation of more compacted aggregates, the higher the speed of rotation the lower the aggregate water absorption [68]. It was also demonstrated in [30] that increasing either the angle and speed or the speed and pelletization duration, lead to prolonging the pellet path and thus an increase in tumbling forces, thereby resulting in the formation of compacted aggregates with lower water absorption and higher strength. However, Baykal and Döven [32] have reported that the critical rotation speed of the pelletizer can be estimated as a function of the pan diameter and angle, using the following equation:

\[ n_c = \frac{42.3}{\sqrt{D}} \sqrt{\sin \alpha} \]

where, \( n_c \) is the critical speed (rpm), \( D \) is the diameter of the pan (m), and \( \alpha \) is the angle of the pan in degree.

5.2. Effects of raw materials and binders

Table 2 indicates the feasibility of various waste and by-product materials and different types of binder for the production of CBA. The characteristics of these raw materials and compatibility between them have a significant influence on the manufacturing efficiency and properties of the manufactured aggregate [66]. Gesoğlu et al. have reported that pelletization efficiency is affected mainly by the fineness of the raw materials. They observed that the pulverized FA, with a fineness of 570 m\(^2\)/kg, exhibited higher pelletization efficiency than FA which had a specific area of 287 m\(^2\)/kg [46]. A significant difference between the specific gravity of the raw materials used was found to harm pelletization efficiency. Chiou et al. have produced aggregate using sewage sludge ash and dried sewage sludge with a specific gravity of 2.64 and 1.2, respectively, finding that the pelletization efficiency deteriorated sharply from 90.6% to 37.7% when 30 wt-% of sewage sludge ash was replaced with sewage sludge [66]. Moreover, Geetha and Ramamurthy have studied the effects of different binders on the productivity of low calcium bottom ash-based aggregate [39], achieving pelletization efficiency of 98% with cement, lime and high swelling bentonite as binders, at a content of 14 wt-%. The same efficiency was achieved with 25 wt-% medium swelling bentonite (MSB) and 30 wt-% clay binder (kaolin and metakaolin). Many researchers have called for the use of additives to enhance pelletization efficiency and in this aspect, it has been established in the literature that the use of calcium hydroxide leads to a reduction of the duration of pelletization by accelerating agglomeration, thus improving manufacturing productivity [15,23,30,39].

The influence of the binder used, on the properties of CBA, has been investigated in several studies. Gomathi and Sivakumar have compared the performances of fly ash aggregates made with both cement and lime as binders. They observed that the oven dry particle density, loose bulk density and individual crushing strength of the aggregate containing cement were higher than that of aggregate produced with lime, by about 34% and 37%, respectively, along with lower water absorption by about 50% [20]. Similar effects were obtained by Cioffi et al., who have reported that aggregate produced with MSWI bottom fly ash (<125 \(\mu\)m) and cement as a binder, exhibits higher strength and lower water absorption than that produced with lime [69]. The reason for this is the formation of an additional hydration product, which in the case of cement as a binder, results from the reaction between calcium hydroxide, released from cement hydration itself, and the pozzolanic compounds of fly ash [30]. Moreover, the effect of binder content on the properties of CBA made of fly ash and quarry dust has been studied by Thomas and Harilal, who found that increasing cement content generates more hydration products, which seal the pores inside the pellets, hence leading to a denser and stronger aggregate [43]. However, Manikandan and Ramamurthy have found that the effect of bentonite content as a binder is related to the duration of pelletization. The authors reported that, at a pelletization duration of 8 min, increasing the bentonite content results in a marked improvement in TPFV and spherical shape of the aggregates. How-
ever, due to the expansive nature of bentonite, flaky CBA with less TPFV was produced when the duration was extended to 16 min [34].

The reactivity of raw materials used in the production process has also been found to be an influential factor in regards to the properties of CBA. Research in this field has compared the properties of CBA, produced with GGBFS, with that made of rice husk ash (RHA). It has been found that the GGBFS- CBA showed superior performance to that produced with rice husk ash [49]. This observation is consistent with the results presented in [46], where the CBA made of GGBFS and cement exhibited higher particle density and higher crushing resistance than that produced with cement and fly ash. This behavior is mainly attributable to raw material reactivity, represented by the content of reactive silicon dioxide, which reacts with the calcium hydroxide released from cement hydration to generate more hydration product, thereby resulting in the formation of denser and stronger CBA [26,46]. Moreover, Tajra et al. have reported that, due to the amorphous structure of expanded perlite powder and its high surface area compared to that of fly ash, the incorporation of expanded perlite powder as a fly ash replacement significantly improves the properties of CBA [11]. The use of a binder with high reactivity has also been reported to enhance the performance of CBA. Colangelo and Cioffi used two types of cement kiln dust as binders, differing mainly in their chemical composition, and found that the binder with a high amount of free lime and low calcite content improved the engineering properties of CBA [52].

Overall, it can be concluded that the physical and mechanical properties of the raw materials and binders used in production have a considerable impact on the manufacturing productivity and properties of CBA.

5.3. Effects of water content

In addition to the production parameters and the properties of the raw materials used, water content is a crucial factor for successful production of CBA. It also has direct effects on both the productivity and properties of CBA. Concurrently, the amount of water required in production is also affected by the properties of the raw materials used. It is evident that an inadequate amount of water prevents agglomeration, while excessive water leads to the formation of muddy balls [66]. Therefore, in most studies, the optimum amount of water has been determined through several production trials as a means of achieving high pelletization efficiency. Baykal and Döven have revealed that, in order to achieve size coherence for fresh pellets, enough water should be used to fill all intergranular voids without the presence of water film on the pellet surface [32]. Otherwise, entrapped air voids are generated inside the fresh pellets, thus weakening aggregate strength [46]. Harikrishnan and Ramamurthy [68] have reported that water content is the primary factor affecting the size of the aggregate produced. In their study, water contents of 15 and 35 wt-% were required to produce CBA with a size range of 5–8 mm and 10–20 mm, respectively.

6. Techniques for improving the performance of cold-bonded aggregate

Many researchers have attempted to improve the properties of CBA using different methods, like the incorporation of additives and alkaline activators in the production process or employing different curing methods and surface treatments for the aggregate produced. The subsequent section reviews the contribution of such efforts in enhancing the characteristics of CBA.

6.1. Incorporation of additives and alkaline activators

Various types of additives can be used in the manufacturing process of cold-bonded aggregate to improve productivity or to enhance the properties of the final product. Crumb rubber addition can be used to increase the crushing strength of the aggregate produced [57]. Tang and Brouwers have showed that the addition of 3 mm of polypropylene fiber (PPF) increases the crushing strength of CBA, due to its role as a reinforcement in the structure of the hardened pellet [53]. Sodium sulfate (Na2SO4) has also been successfully employed to accelerate the pozzolanic reaction of fly ash and to densify the structure of the aggregate produced, thereby reducing water absorption and enhancing the strength of CBA [16].

The additives mentioned above have been used in aggregate using cement as a binder. Alternatively, due to the excellent performance of cement-free matrices produced by alkalai activation, the use of an alkaline activator as a binder has been the focus of many researchers in producing alkali-activated CBA [70]. Low-calcium bottom ash has been activated by a combination of sodium silicate solution (Na2SiO3) and sodium hydroxide (NaOH), at a molarity of 8–12 mol/l, to produce geopolymer aggregates [55]. The authors observed that the bulk density and TPFV of the aggregate were 7–20% and 12–93% higher than that of cement-activated aggregate, respectively. A similar solution was also used as an alkaline activator to initiate the activation of fly ash, GGBS and rice husk ash [55]. Furthermore, the suitability of NaOH at a molarity of 10 mol/l in stimulating the activation of fly ash and bentonite, has been investigated by Gomathi and Sivakumar [71]. The authors have concluded that the use of an alkaline activator contributes effectively to improving the mechanical and microstructural properties of the CBA produced [22,72].

6.2. Surface treatment

It can be seen from Table 3 that the CBA produced possessed a high water absorption, ranging between 7 and 52 wt-%. Therefore, reducing the water absorption of CBA was of much concern for some researchers due to its negative effect on the performance of concrete [74]. Gesoğlu et al. have attempted to treat fly ash CBA by immersing it for 30 min, either in a solution of water glass or in silica fume slurry. The results demonstrated that water absorption dropped significantly from 27% to 3% in the case of water glass treatment and from 27% to 18% when the aggregate was treated by cement-silica fume slurry. It was also observed that the crushing strength of water glass treated aggregate was higher than that of untreated aggregate, by about 40–100% [26,27]. A possible reason for this was the formation of an additional C-S-H phase, made from the reaction between water glass and both the calcium oxide and calcium hydroxide present in the cementitious system [74]. Hwang and Tran have suggested a treatment method in which an alkaline solution (a combination of water glass and sodium hydroxide with a molar SiO2/Na2O of 2.5) is sprayed on the surface of the foamed CBA in a pelletizer disc. They found that this led to the unit weight and particle crushing strength of surface-treated aggregates being higher than that of non-treated aggregates, by about 4.5–6% and 15–27% respectively and with a lower water absorption by about 2–33%. Moreover, Colangelo et al. have proposed a double pelletization technique in which a second pelletization process is carried out on the aggregate produced, to cover it with a dense outer shell composed of 1:1 cement/coal fly ash. The authors found that the water absorption of the double-step CBA was about 12–18% lower than that of CBA made with only one pelletization step [51]. Recently, Tajra et al. applied surface treatment to the production of CBA, by spraying it with a cement-silica fume mixture, whilst it was rolling in the pelletizer disc. They concluded that the water absorption of
### Table 5
Compositions and properties of cold-bonded aggregate concrete.

<table>
<thead>
<tr>
<th>Ref</th>
<th>Cement (kg/m³)</th>
<th>SCM (kg/m³)</th>
<th>CBCA (kg/m³)</th>
<th>NWCA (kg/m³)</th>
<th>NWS (kg/m³)</th>
<th>w/b</th>
<th>Slump (mm)</th>
<th>Slump flow (mm)</th>
<th>Fresh density (kg/m³)</th>
<th>Dry density (kg/m³)</th>
<th>Compressive strength (MPa)</th>
<th>Flexural strength (MPa)</th>
<th>Split tensile strength (MPa)</th>
<th>Modulus of elasticity (GPa)</th>
</tr>
</thead>
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<td>[13]</td>
<td>350 –</td>
<td>1140 –</td>
<td>896 –</td>
<td>0.4</td>
<td>270 –</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>47.45</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>[18]</td>
<td>550 SF 55</td>
<td>592 –</td>
<td>636 –</td>
<td>0.26</td>
<td>150 –</td>
<td>1991</td>
<td>1860</td>
<td>42.3</td>
<td>–</td>
<td>3.7</td>
<td>19.6</td>
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<td></td>
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<tr>
<td>[31]</td>
<td>385 –</td>
<td>350 548</td>
<td>736</td>
<td>0.5–0.54</td>
<td>130–170</td>
<td>–</td>
<td>–</td>
<td>1990–2170</td>
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<td>0–812 –</td>
<td>0.4</td>
<td>700–730</td>
<td>1827–2345</td>
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<td>10–40</td>
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<td>792–797</td>
<td>0.32</td>
<td>–</td>
<td>700–750</td>
<td>2183–2315</td>
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<td>61.9–69.74</td>
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<tr>
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<td>508–598</td>
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<td>1937–1977</td>
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<tr>
<td>[52]</td>
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<td>–</td>
<td>1750–1981</td>
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<tr>
<td>[53]</td>
<td>310 FA 179</td>
<td>234–483</td>
<td>558–816</td>
<td>0.4</td>
<td>–</td>
<td>490–758</td>
<td>2220–2340</td>
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<td>47–64</td>
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<td>0.49</td>
<td>–</td>
<td>–</td>
<td>1682–1825</td>
<td>–</td>
<td>12.8–25.8</td>
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<td>–</td>
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<td></td>
</tr>
<tr>
<td>[75]</td>
<td>405 SF 45</td>
<td>575–600</td>
<td>593</td>
<td>0.36</td>
<td>–</td>
<td>420–450</td>
<td>1115–1540</td>
<td>17.9–29.1</td>
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<tr>
<td>[97]</td>
<td>450–600 –</td>
<td>297–634</td>
<td>626–1096</td>
<td>0.3–0.5</td>
<td>–</td>
<td>213–482</td>
<td>–</td>
<td>20–60</td>
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<tr>
<td>[98]</td>
<td>400 –</td>
<td>541–725</td>
<td>684–936</td>
<td>0.4</td>
<td>–</td>
<td>–</td>
<td>1682–1825</td>
<td>–</td>
<td>12.8–25.8</td>
<td>–</td>
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</tbody>
</table>

SCM: supplementary cementitious material; FA: fly ash; SF: silica fume.

CBCA: cold-bonded coarse aggregate; NWCA: normal-weight coarse aggregate; NWS: normal-weight sand.

CBA treated in this way decreased by about 27%, while the particle density, loose bulk density and bulk crushing strength improved by about 13%, 11% and 14%, respectively, as compared with untreated aggregate [75].

Accordingly, the treatment methods mentioned above can be considered to be proper techniques, as they have the advantage of being easy to implement and also because they effectively upgrade the physical and mechanical properties of the aggregate produced. However, from an economic perspective, further studies need to be conducted so as to reveal the effects of these techniques on the cost of the final product and to thus develop a cost-effective, reasonable approach.

6.3. Curing methods

Table 3 illustrates the curing methods applied in the literature. As can be seen in this table, curing at a relative humidity of 70% (RH 70%) and at a temperature of 20 °C is the most frequently used method of curing CBA. From amongst these studies, a number of research designs have exposed the impact of different curing regimes on the mechanical and microstructural properties of CBA, aiming to determine the method most compatible with the raw materials in use. Manikandan and Ramamurthy, for instance, have discussed the effects of water curing, autoclaving (at a pressure of 1 MPa) and steam curing (at 70 °C) for 5–10 h on the properties of class C-fly ash aggregate. They concluded that the water curing method is more effective than the other methods for enhancing the hydration of fly ash and thus improving CBA performance [76]. However, in a research study by Tajra et al., water curing was ranked second for enhancing the properties of core-shell CBA, after curing at a relative humidity of 99% (RH 99%); curing at RH 65% was ranked third. They reported that curing at RH 99% speeds up the reaction in the cover matrix of CBA more than both water curing in water and at RH 65%, thereby resulting in a higher crushing strength by about 17 and 58%, respectively [75]. Since a high curing temperature increases the extent of geopolymerization [77,78], Geetha and Ramamurthy have found that the geopolymer CBA cured at a temperature of 50–80 °C shows a higher TPFV than when cured at an ambient temperature of 30–32 °C, by about 11–32% [55]. Ferone et al. [42] have also found that the crushing strength of CBA, made with lime as a binder and cured at a temperature of 40 °C, is higher than that cured at room temperature by about 6–13%, with a higher hydration reaction being observed.

The analysis of the literature presented above provides evidence that the characteristics of CBA can be improved significantly, by determining the optimum curing process appropriate to the raw materials used and the structure of the aggregate produced.

7. Application of cold-bonded aggregates in concrete

Table 5 lists the mix proportions and fresh and hardened properties of cold-bonded aggregate concrete (CBAC) designed by different researchers. The subsequent sections discuss the different performance aspects of CBAC, including its fresh properties, mechanical and thermal properties, durability and microstructure.

7.1. Fresh properties

The content of water, cement, air, coarse and fine aggregates in a concrete mixture, as well as the shape, density and water absorption of the aggregate, are the primary factors affecting the fresh properties of concrete [79]. In order to enhance the workability of CBAC and to avoid the early slump loss caused by the high water absorption of CBA, many researchers have recommended using CBA in saturated-surface dry condition [80,81].

A significant improvement in concrete workability has been observed when normal aggregate (NA) is totally replaced by CBA [15]. This is mainly attributable to the spherical shape of CBA, as compared to the angular nature of NA [13]. Therefore, to achieve the same CBAC workability, the normal aggregate concrete (NAC) mixture should be modified, by either increasing the cement paste volume or the amount of superplasticizer, although this leads to increases in cost [82,83]. Kockal and Ozturan have found that, to achieve the aimed slump, the required dosage of superplasticizer decreased by about 8%, when NA was replaced with CBA [28].

Moreover, the workability of CBAC has been observed to be dependent on the CBA content. It has been found that the CBAC slump improves significantly with an increase in CBA volume content [33,84]. Increasing the volume content of CBA, which has a rounded shape and smooth surface, reduces the friction between the aggregate particles and the cement paste, thus leading to a significant improvement in concrete flowability [85].

The applicability of CBA in the production of self-compacting concrete (SCC) has been investigated by many researchers. Slump flow, T50, V-funnel and L-box tests help to assess the flowability, viscosity and passing ability of SCC to classify it in accordance with the norms of the European Federation of Specialist Construction Chemical and Concrete System (EFNARC) [86]. Gesoğlu et al. [47] have reported that the replacement of 100 vol-% NA with CBA contributes strongly to improving the fresh properties of SCC, with the amount of superplasticizer required decreasing from 8 to 4.2 kg/m³, the slump flow increasing from 700 to 750 mm and T50 and V-funnel decreasing from 3.43 to 1 s and from 17.22 to 5.13 s, respectively. This tendency is in line with observations made in many studies [38,40,53]. Hwang and Tran [45] have identified the positive effects of the surface treatment of CBA in improving the workability of SCC. The smoother surface of treated CBA, compared with that of untreated CBA, decreases the internal friction between the aggregate and the cement paste, thereby easing concrete flowability [87].

The discussion above clearly shows that the fresh properties of CBAC are superior to that of NAC and furthermore, these can be improved by the use of surface-treated CBA and saturated-surface dry CBA. Therefore, the use of CBA in concrete can be cost-effective, by reducing the amount of superplasticizer required to achieve the desirable workability.

Since the fresh density of concrete is related to the density of its ingredients, the incorporation of CBA has been observed to affect the fresh density of concrete. Tang and Brouwers [53] have observed that the fresh density of NAC decreases by about 5% and 8% when NA is replaced with CBA, at replacement levels of 30 vol-% and 60 vol-%, respectively. However, due to the low bulk density of the CBA (769 kg/m³) produced in [48], the fresh density of CBAC was found to be 17% less than that of NAC. This is consistent with the results presented in [12], where the unit weight of fresh mortar decreased by about 5%, 9%, 13% and 18% when natural sand was replaced with fine CBA at replacement levels of 25, 50, 75 and 100 vol-%, respectively. Fig. 2 presents the influence of CBA replacement on the fresh density of concrete. The literature in this aspect indicates that CBAC has a lower fresh density than NAC. It should be pointed out here, that even though most CBA produced was classified as lightweight aggregate, the fresh density of CBAC does not meet the requirement of lightweight aggregate concrete in accordance with the American Concrete Institute (ACI 213R-03) [88]. The main reason for this is the use of a high cement content and natural sand in the mix design of CBAC [48].

However, as shown in Table 5, CBAC with a dry density ranging from 1115 to 1981 kg/m³ has been produced in some studies. Being that the dry density of CBAC <2000 kg/m³, CBA is applicable for the production of lightweight aggregate concrete, as specified in EN 206-1 [89]. This may offer important economic and environ-
mental benefits, by reducing the weight of structures and improving their thermal performance [73].

7.2. Compressive strength

Table 5 shows that the compressive strength of CBAC produced in the available studies has ranged from 7 to 70 MPa. It has been established in the literature that lightweight aggregate is the weakest component in the microstructure of LWAC [90,91]. The volume content of the aggregate in concrete is one of the primary factors influencing its compressive strength [57,92]. Experimental studies have shown that the compressive strength of CBAC decreases with an increase in the content of CBA [15,17,22,53]. Gesoğlu et al. [40] have observed that the compressive strength of CBAC was 25% less than that of NAC when coarse NA was totally replaced with coarse CBA, with this percentage becoming 38% when only the fine fraction of NA was replaced with fine CBA. Increasing the CBA content from 50 to 100% has also been observed to decrease the compressive strength of CBAC by about 25% and 23%, as found by Tang et al. and Tajra et al., respectively [50,93]. However, the effect of CBA content on the compressive strength of CBAC has found to be related to the strength of the cement matrix itself [37]. CBAC with a cement content >350 kg/m$^3$ has showed a clear decrease in compressive strength with increasing CBA content, while the compressive strength of CBAC containing a cement content of 300–350 kg/m$^3$ has been observed to be independent of CBA content [84]. This phenomenon can be explained by concrete failure, which takes place through either CBA, the cement matrix or by the transition zone between them [71].

On the other hand, the compressive strength of concrete produced with lightweight aggregate depends mainly on the quality of the aggregate used [94]. The strength of CBAC has also been found to be affected by the properties of CBA; its water absorption, particle density and crushing strength [95]. Many authors have reported that the higher the crushing strength of CBA, the higher the compressive strength of the concrete produced [43,46,48,51]. Some researchers have observed that the use of high-density CBA increases the density of the CBAC produced and therefore enhances its compressive strength [28,96]. Moreover, the porosity and water absorption of CBA have been found to affect the transition zone between the CBA and the cement matrix, thereby affecting the mechanical properties of CBAC [5]; this issue is discussed below.

Fig. 3 presents the influence of CBA, as a NA replacement, on the compressive strength of concrete. The results indicate that the compressive strength of CBAC is inferior to that of NAC, thus suggesting that CBAC strength can be improved by enhancing the properties of both the cement matrix and the CBA. Kockal and Ozturun have reported that reducing the water/binder ratio contributes strongly to enhancing the strength of CBAC, especially in concrete with a CBA content of up to 18% [97]. The addition of steel fiber, up to 1 vol-%, leads to an increase in the compressive strength of CBAC by about 5–10% [98]. Some authors have suggested that the use of saturated surface-dry CBA in concrete enhances its compressive strength; whereas dry aggregate absorbs a high amount of water from the cement matrix, resulting in an insufficient amount for cement hydration, thereby decreasing concrete strength [81]. The incorporation of surface-treated CBA was found to have a positive effect on the compressive strength of CBAC [26,27]. An increase in the compressive strength of CBAC, by about 11% and 13%, was observed when surface-treated CBA was used in [45] and [75], respectively.

However, as can be seen in Table 5, the unit weight and compressive strength of CBAC produced in the literature are in the range of 1115–2345 kg/m$^3$ and 7.2–70 MPa, respectively. These results show the feasibility of using cold-bonded aggregate for the production of normal-weight concrete and structural and non-structural lightweight aggregate concrete [88,99].

7.3. Modulus of elasticity

The modulus of elasticity of concrete made of LWA has been reported to be affected by the volume content and structure of LWA, as well as by the elasticity modulus of the cement matrix [100,101]. Similar to the compressive strength trend, the modulus of elasticity of CBAC has been observed to decrease with an increase in the content of CBA [102]. It decreases by about 13%, from 28.2 GPa to 24.6 GPa, when the volume fraction of CBA is increased from 45% to 60% [98].

The modulus of elasticity was also found to increase with an increase in CBA strength and a decrease in its water absorption, stemming from the surface treatment process [26]. CBAC produced with surface-treated aggregate shows a modulus of elasticity higher than that produced with untreated CBA, by about 6–20% [75]. These observations have been confirmed by Ke et al., who
have reported that the lower the water absorption of aggregate, the better the modulus of elasticity of the concrete produced [91]. At the same time, the modulus of elasticity of CBAC, containing up to 18 vol-% CBA, has been observed to be independent of aggregate quality; in this case, the cement content and w/cm ratio become the controlling factors for the modulus of elasticity [97].

Tomas and Harilal have reported that the strength of the cement matrix has a considerable effect on the modulus of elasticity of CBAC. They have showed that the elasticity modulus of CBAC increases by about 46–74% when cement content is increased from 250 to 450 kg/m³ [43]. It has also been observed that increasing the water/cement ratio decreases the modulus of elasticity of CBAC, while the incorporation of silica fume in concrete mixtures enhances it by about 6–20% [27,37].

7.4. Flexural and split-tensile strength

The 28-day flexural strength of CBAC produced by various researchers is illustrated in Table 5 and ranges from 2.26 to 11.34 MPa. CBAC flexural strength has been observed to decrease with an increase in CBA content. Tang and Brouwers have found that the replacement of NA with CBA at replacement levels of 30 and 60 vol-% causes a reduction in flexural strength by about 24–42%, based on the crushing strength of the CBA used [53]. Teržič et al. have observed that the flexural strength of CBAC with 100% CBA was lower than that of NA by about 40–47% [22], with the percentage loss in flexural strength of CBAC being 29%, according to Kumar et al. [31]. This indicates that the flexural strength of CBAC is hardly affected by the quality of the CBA used. Hwang and Tran have investigated the flexural strength of CBAC made with two types of CBA differing in their crushing strength, and found that the flexural strength of CBAC made with high-strength CBA was higher than that made of low-strength CBA, by about 5.2–22.9% [45].

However, the loss in flexural strength due to an increase in the volume content of the aggregate can be compensated for by incorporating steel fibers in the CBAC mixture. Concrete made with 60 vol-% CBA and 0.35 vol-% steel fiber attains similar flexural strength to CBAC made with 45 vol-% CBA [98]. This is attributable to the bond between steel fiber and the cement matrix, which improves the bending carrying capacity of concrete and thus its flexural strength [103].

Table 5 shows that the split-tensile strength of CBAC ranges from 1.65 to 5.1 MPa. It is evident from this table that the ratio of splitting tensile/compressive strength of CBAC is in the range of 4–15%; for NAC it has been observed to be between 8% and 14% [2]. Similarly to compressive and flexural strengths, since the strength of CBA is lower than that of NA, the splitting strength of concrete has been reported to be negatively affected by the substitution of NA with CBA [57]. Gesoğlu et al. have reported that the tensile strength of CBAC made with 100% CBA was 44% less than that of NAC [40]. However, it has been established that this phenomenon can be mitigated by improving the properties of the cement matrix itself, by decreasing the water-binder ratio and through the use of silica fume and steel fiber in concrete mixtures [37,98].

7.5. Thermal conductivity

The thermal conductivity of concrete depends mainly upon the thermal properties of its components; especially the aggregates, which take up about 70–80% of its total volume. The low thermal conductivity of the aggregate used results in improvement of the thermal insulation properties of concrete [4]. The thermal conductivity of LWAC has also been shown to be proportional to its density [104,105]. It is worth pointing out that the available studies on the thermal conductivity of CBAC appear to be limited. However, the effects of replacing NA with CBA on the thermal properties of CBAC have been studied by Frankovic et al. and Tajra et al. [93,106]. Frankovic et al. have found that CBAC made with 100% CBA has a dry density of 1490 kg/m³ and a thermal conductivity of 0.73 W/m·K, which are 35% and 46% less than that of NAC, respectively [106]. This finding was later confirmed by Tajra et al. [93], who have reported that the thermal conductivity of NAC decreases by about 28% and 43% when NA is replaced with 50% and 100 vol-% CBA, respectively. These observations can be attributed to the highly porous structure and low density of CBA, compared to that of NA, which cause a decrease in both the density and thermal conductivity of concrete. Tajra et al. have also reported that the use of core-shell CBA allows the production of CBAC with a thermal conductivity of 0.8364 W/m·K and
0.3169 W/m K, when using normal sand and lightweight clay sand as fine aggregates, respectively [75].

Being that the thermal conductivity of CBAC produced in the available literature <0.75 W/m K, cold-bonded aggregate has sufficient potential to be used for the production of insulating concrete [107].

8. Durability properties of cold-bonded aggregate concrete

One of the most popular tests for durability evaluation is through capillary water absorption, which describes water transport into concrete, via the pores and against gravity. Capillarity water absorption tends to decrease with total porosity and the pore size of concrete [19]. Since the porosity of CBA is higher than that of NA, the sorptivity of CBAC is higher than that of NAC. Gesoğlu et al. have observed that the sorptivity index increased from 0.07 to 0.155 mm/min<sup>0.5</sup> when only coarse NA was replaced with coarse CBA, while it increased to 0.201 after the replacement of all the NA (coarse and fine fraction) with CBA [40]. The sorptivity index of mortar was also found to increase by about 44, 86, 88 and 110% when normal sand was substituted with cold-bonded sand, at replacement levels of 25, 50, 75 and 100% respectively [12].

However, some researchers have showed that the sorptivity of CBAC can be improved either by enhancing the microstructure of the cement matrix, or by the CBA surface treatment. Joseph and Ramamurthy have showed that the use of fly ash as a partial replacement of normal sand helped to produce CBAC with a sorptivity index lower than that of their control mix, by about 48% and 60% for concrete mixes with a cement content of 450 and 250 kg/m<sup>3</sup>, respectively [33]. Favourable results have also been achieved when the surface treatment process of CBA is applied. Tajra et al. have found that the CBAC produced with surface-treated CBA exhibits a lower water capillarity coefficient than that made of untreated aggregate, by about 29%. This behavior has been attributed to the formation of a dense film on the aggregate surface, which reduces the absorption of the aggregate itself and thus the sorptivity index of the concrete produced [75].

Water penetration into CBAC under pressure has also been investigated, to characterize its durability. Water penetration depth has been observed to be dependent on the quality and the volume content of CBA. It is evident from the literature that the water penetration depth in artificial lightweight aggregate concrete is higher than that of NAC [101,108]. This is attributed to the porous structure of CBA, which facilitates the penetration and transportation of water into and through the aggregate, thus increasing the water penetration depth in CBAC [109]. Gesoğlu et al. have reported that the water penetration depth of 56-day-old CBAC increased from 11 mm to 26 mm when 100% NA was replaced with CBA [40]. Tang and Brouwers [53] have announced an increase in penetration depth, from 11 mm to 12.5 and 13.8 mm, by 30 and 60 vol-% NA replacement with CBA, respectively. Moreover, the water absorption of CBAC, as a percentage of oven-dried mass (in accordance with ASTM C 642 [110]), has been evaluated in a study by Joseph and Ramamurthy [33]. The authors showed that with the use of fly ash CBA at contents of 50 and 60 vol-% of the total aggregate volume, CBAC with a water absorption range of 3.4–7.4 wt-% can be produced.

Accordingly, as the water penetration depth of CBAC <30 mm and its water absorption <10 wt-%, it can be classified as an impermeable concrete [79].

Moreover, some researchers have evaluated the durability of CBAC by evaluating its resistance against chloride ion penetration, in accordance with ASTM C1202, in terms of the total charge passed [111]. It has been observed that the rapid chloride permeability of CBAC exhibits an increasing trend with an increasing CBA content and a declining trend with an increase in concrete age [18,19,25]. The adverse effects of CBA on the chloride permeability of CBAC are therefore minimized by extending the testing age from 28 to 56 days [40]. However, at an age of 90 days, a chloride permeability with a total passing charge of 780 C can be achieved, as showed in [18,25]. While, as presented in [40], the passing charge was approximately in the range of 930–1400 C. These results demonstrate the feasibility of using CBA to produce concrete with low and moderate chloride permeability behavior, as specified in ASTM C1202.

The freeze and thaw resistance of CBAC, as a durability indicator, has also been investigated by many researchers. Although the performance of CBAC under the freeze-thaw condition is inferior to that of NAC, it shows a satisfactory durability factor of 85–90 [18]. Researchers have figured out that, due to the expansion of water inside aggregates under freeze cycles, the use of CBA in saturated condition negatively affects the freeze-thaw resistance of LWAC [53]. It has also been observed that the addition of silica fume contributes to densifying the microstructure of the cement matrix of LWAC, thus enhancing concrete durability against freeze and thaw cycles [25].

9. Microstructure of cold-bonded aggregate concrete

As in normal concrete, the microstructure of CBAC consists of cold-bonded aggregate, cement matrix and an interfacial transition zone (ITZ) between them. Generally, the quality of the ITZ, which influences the mechanical properties and the durability of the concrete, is highly affected by the type and properties of the aggregate used. Due to the large solid differences between NA and cement particles, a water-rich area with poor cement content is formed on the surface of the aggregate. This “wall effect” leads to a weak ITZ between the NA and the cement matrix [112]. However, in the case of LWA, the penetration of cement paste into the surface pores of LWA develops a mechanical interlocking between the LWA and the cement matrix, thus resulting in a good-quality ITZ [113]. It has also been reported that a chemical interaction between the CBA and the cement matrix can take place. This interaction stems from the pozzolanic reaction between the calcium hydroxide released from cement hydration and the raw materials used in CBA [114,115].

![Fig. 4. Interfacial transition zone between the cold-bonded aggregate and the cement matrix](image)
A typical ITZ microstructure of cold-bonded fly ash lightweight aggregate concrete is shown in the SEM image in Fig. 4. It was observed that the CBA was tightly and continuously bonded to the cement matrix and that its boundary was therefore relatively indistinguishable [25,75]. The effects of dry and saturated-surface dry LWA on the quality of the ITZ have been studied. The internal curing provided by the water reserved within the saturated LWA enhances the hydration of the surrounding cement paste, thus densifying the ITZ and improving its quality [116,117]. Moreover, the internal curing has been found to be one of the solutions to avoid cracking in concrete caused by the drying shrinkage. After the self-desiccation of concrete, the stored water within the aggregate is released to fill the pores of the hardened mortar, thus reducing the tensile stresses induced by drying shrinkage to be less than the tensile strength of the concrete itself, which contributes effectively to enhancing the cracking resistance of concrete [118]. While, due to the high water absorption of LWA, using it in concrete under dry condition has been found to affect the ITZ. It has been reported that the higher the water absorption of LWA, the higher the amount of water lost from the area surrounding it, thus generating a high content of unhydrated cement in this zone [119]. However, Alexander et al. have reported that, due to the high water sorption of the porous aggregate, the ITZ is characterized by a lower water to binder ratio and thus less porous in the presence of dry aggregate compared to that in the case of saturated-surface dry aggregate [120].

10. Summary and conclusions

This paper has reviewed various factors affecting the production efficiency of the cold bonding method and the properties of the cold-bonded aggregates produced. Different techniques for improving the quality of CBA have been discussed. Moreover, the different properties and performance of concrete made of cold-bonded aggregate have been presented and discussed. In reference to the details above, the following conclusions can be drawn:

- The absence of dominance between gravitational and centrifugal forces on the movement of raw materials in the pelletizer disc, is the basis for successful production and a guarantee for the production of good-quality cold-bonded aggregate.
- The variations in production parameters, the angles and speeds of the pelletizer used in the literature, are primarily attributed to the use of different raw materials and binders. Preliminary production trials should therefore be undertaken carefully, with the first step being the determination of the optimum angle and speed of the pelletizer disk, so that they suit the raw materials used. A reasonable productivity and good-quality cold-bonded aggregate can thus be produced.
- The particle and loose bulk densities of cold-bonded aggregate produced in the literature are in the range of 0.88–2.3 g/cm³ and 510–1460 kg/m³, respectively. It can therefore be classified as lightweight aggregates, according to EN 13055-1 [62].
- The properties of cold-bonded aggregates can be improved either by treating their surface, or by incorporating suitable additives and alkaline activators into the production process.
- Since cold-bonded aggregates are spherical in shape, the fresh properties of cold-bonded aggregate concretes are superior to that of conventional concrete.
- This review shows the ability of cold-bonded aggregate to produce concrete with a compressive strength between 7 and 70 MPa, a modulus of elasticity from 6 to 61.75 GPa and a density varying between 1115 and 2345 kg/m³.
- The mechanical performance of the CBAC mentioned above, strongly supports the feasibility of using cold-bonded aggregate for the production of normal weight concrete and both structural and nonstructural lightweight aggregate concrete.
- Lightweight aggregate concrete, with a thermal conductivity <0.75 W/m-K, can also be produced by the utilization of cold-bonded aggregates.
- Due to mechanical and chemical interlocking between the cold bonded aggregate and the cement matrix, a good-quality ITZ can be achieved.
- Though the durability properties of cold-bonded aggregate concrete are inferior to that of conventional concrete, impermeable CBAC with a water penetration depth <30 mm and water absorption <10% can be produced.

Declaration of Competing Interest

The authors declare no conflict of interest.

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References

4. Publications

4.2. Performance assessment of core-shell structured lightweight aggregate produced by cold bonding pelletization process

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Performance assessment of core-shell structured lightweight aggregate produced by cold bonding pelletization process

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HIGHLIGHTS

- Core-shell lightweight aggregates were produced by cold bonding pelletization process.
- Influence of production parameters on the aggregate properties was investigated.
- Effects of incorporation of expanded perlite powder into the shell matrix were assessed.
- Lightweight aggregate with particle density ranges from 0.88 to 1.14 g/cm³ were obtained.
- Performance of fly ash and expanded perlite powder as mineral admixtures was studied.

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ABSTRACT

The density of cold-bonded lightweight aggregates is significantly higher than that of sintered lightweight aggregates. Since the sintering technique consumes an enormous amount of energy and emits a huge amount of pollutants, the implementation of a cold-bonded method, in manufacturing low-density lightweight aggregates, is very important from an economic and environmental perspective. In this study, a cold bonding granulation technique was employed to produce low-density lightweight aggregate by the encapsulation of expanded perlite particles in shell structures. A variety of tests were conducted to evaluate the physical and mechanical properties of the aggregate produced. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to study the microstructure and the phase composition of the aggregate. Furthermore, X-ray micro-computed tomography (CT) was performed to investigate the pore system of the aggregate specimens. The obtained results showed that by adopting the angle and speed of pelletizer disc, a core-shell structured lightweight aggregates with particle density of 0.88–1.14 g/cm³ and bulk crushing strength of 2.04–2.66 MPa can be produced.

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1. Introduction

Recently, lightweight aggregates (LWAs) have attracted great interest and large industrial demand in a wide range of construction products. They have lower density and thermal conductivity than conventional aggregates, thus offering important economic and environmental benefits [1,2]. LWAs can be natural or artificial. The usage of natural aggregates has become a contentious issue due to their over use in addition to the lack of natural sources in most areas [3,4]. Consequently, the utilization of artificial LWAs in various construction products has been growing dramatically. Artificial LWAs can be produced via sintering or cold bonding technique. The sintering method has two main disadvantages: large energy consumption and the emission of huge amounts of pollutants [5]. On the contrary, the cold bonding method has the potential to satisfying from both economic and environmental perspectives, due to its potentially lower energy consumption if the requirements for density and strength can be improved.

Many studies in recent years have concentrated on the manufacturing of cold-bonded LWAs. Such research efforts have focused on recycling different by-products or waste materials as LWAs, regardless of the density of the aggregates produced. Gesoğlu et al. [6] have produced a cold-bonded aggregate with a specific gravity of 2.14 g/cm³ by using ground granulated blast furnace slag (GGBFS) in the manufacturing process. Kumar et al. [7] have utilized fly ash and cement at different cement/fly ash ratios, with the aggregate having specific gravity ranges between 1.72 and 1.97 g/cm³, depending on the cement content. Chi et al. [8] have reported that fly ash and cement can also be used to produce...
LWAs, with an oven dry specific gravity between 1.23 and 1.44 g/cm³, and a corresponding particle strength of 6.04–8.57 MPa. Thomas and Harilal [9] have developed a cold-bonded quarry dust coarse aggregate with a specific gravity of 1.9–2.5 g/cm³, which is similar to that of normal weight aggregate. Colangelo and Cioffi [10] have employed cement kiln dust, GBFS, and marble sludge in their manufacturing process. The aggregate fabricated had a dry density of 1.7–1.98 g/cm³.

One of the main disadvantages of cold-bonded LWAs is its high density compared to most of sintered LWAs available in the market, such as expanded clay and expanded glass. Only little attention has been paid to this vital issue, which has restricted the practical applications of cold-bonded LWAs. More recently, Hwang and Tran [11] have applied hydrogen peroxide as a foaming agent, in order to generate more pores inside the aggregate for the purpose of reducing its density. They have reported that the lowest oven dry specific gravity of 1.27 g/cm³ was achieved when a composition of 80 wt-% fly ash and 20 wt-% GBFS was used, with a foaming agent concentration of 7 wt-%. Though this approach decreased the particle density of cold-bonded LWAs, it was still higher than that of sintered LWAs. A novel cold-bonded production method is therefore required, in order to satisfy three main criteria: low particle density, good mechanical properties and an effective solution for recycling waste materials.

The main objectives of this research can be outlined as follows: (1) optimizing a cold-bonded method for producing artificial LWAs, with low density and good mechanical properties. In this study, a lightweight aggregates with core-shell structure were produced by the encapsulation of expanded perlite particles within a cover matrix composed of cement and fly ash; (2) investigation of the effects of different parameters (cement content, speed and angle of pelletizer disc) on the aggregate properties, in order to determine the optimum values of these parameters; (3) utilization of expanded perlite powder (EPP) as a way to reduce the particle density of the manufactured aggregate, to less than 1 g/cm³. To achieve this aim, EPP was incorporated in the shell structure as a partial replacement of fly ash. A variety of tests were conducted to evaluate the effect of EPP content on the mechanical and microstructural properties of the manufactured aggregate; and (4) detection and comparison of the pozzolanic activity of fly ash and EPP as mineral admixtures in cement paste, to fully understand the positive impact of EPP on the aggregate characteristics.

2. Methodology

Lightweight aggregates were produced by encapsulating expanded perlite particles within a shell structure as shown in Fig. 1. The encapsulation process was performed using a pelletizer disc, 40 cm in diameter and 10 cm in depth (Fig. 3). In this study, two groups of LWAs were manufactured. In the first group, expanded perlite particles (EP), with a size ranging from 2 to 4 mm, were encapsulated in a cover matrix composed of cement and fly ash. A total of 24 samples with three variable parameters were produced: the cement content of the cover matrix, speed and angle of the pelletizer disc. The performance of the aggregate was evaluated according to standard tests, in order to ascertain the optimal values of these parameters. Next, in the second group, expanded perlite powder (EPP) with a size <125 μm was incorporated into the manufacturing process. It was used in the cover matrix as a partial replacement of fly ash. Here, five aggregate samples were prepared, differing only in their EPP content, with the other parameters kept fixed at their estimated values. A variety of tests were conducted to evaluate the effect of EPP on the properties of the aggregates.

3. Experimental investigation

3.1. Materials

In this research, ordinary Portland cement (OPC, CEM I 42.5R), provided by CEMEX (Germany), and class F fly ash EFA-Füller HP, obtained from BauMineral (Germany), were used. Both the expanded perlite and expanded perlite powder were supplied by KLEIN (Germany). The loose bulk densities of EP and EPP were measured according to EN 1097-3, being 80 and 112 kg/m³, respectively. The physical properties and chemical compositions of the materials used are listed in Table 1. The specific surface area and specific density were determined using Brunauer-Emmett-Teller (BET) analysis and Helium pycnometry, respectively. Fig. 2 shows the particle size distribution of cement, FA and EPP, as measured by laser granulometry.

3.2. Production of LWAs

A total of 24 LWAX-A-S were produced by encapsulating EP particles within a cover matrix composed of cement and FA into the pelletization disc, where X refers to the cement content of the cover matrix (wt-%), and A and S refer to the angle (°) and rotation speed (rpm) of the disc, respectively. After several manufacturing trials and in consideration of production efficiency, two angles (35° and 40°), and three speeds (20, 30, and 40 rpm) were selected. Four different cement to fly ash ratios were used in the mixture of the cover matrix; 5:95, 10:90, 15:85, and 20:80. The amounts of both the EP particles, as a core structure, and the powder mixture as a shell structure, were based on the production of aggregates with a size range of 4–8 mm and a shape as close to spherical as possible. As such, for each production process, 25 g of EP particles were first placed into the pelletizer disc. Thereafter, they were simultaneously sprayed with water and fed with 1000 g of dry mixture. The water content was about 120 ± 10 g (12 ± 1 wt-% of the mixture). The water was sprayed carefully during the encapsulation process, in parallel with the feeding of the powder. The total pelletization time was about 15 min. At the end of the manufacturing procedure, the fresh pellets were carefully discharged from the device and directly placed and kept in sealed bags at a temperature of 21 ± 1 °C for 24 h. Next, the samples were cured under water until testing day. Afterwards, the hardened LWAs were sieved and particles with a size range of 4–8 mm were selected for the tests. The manufacturing process as well as the fresh pellets are shown in Fig. 3.

3.3. Influence of production parameters on aggregate properties

Data obtained in previous studies [5,9,12] has indicated that the angle and speed of the pelletizer disc, as well as the binder content, substantially affect the properties of the manufactured aggregate.
Therefore, the influence of these parameters on manufacturing productivity and the characteristics of the aggregates produced were studied carefully. The cement content, speed and angle which give a reasonable productivity and highest strength/density ratio will be considered as optimal values. Here, productivity was assessed by determining encapsulation efficiency; calculated as the ratio between the mass of pellets produced having a size of more than 4 mm and the total mass of the materials used in the production process. Furthermore, the aggregate performance was evaluated in terms of particle density, water absorption, loose bulk density and bulk crushing strength. Oven dry particle density and water absorption were estimated using the pycnometer method, according to EN 1097-3 [14], while, 28-day compressive strength was measured as specified in EN 1097-6, Annex C [13]. Loose bulk density was measured as specified in EN 1097-3 [14], while, 28-day compressive strength of the LWAs was investigated by measuring the bulk crushing resistance, in accordance with EN 13055 Annex C – procedure 1 [15]. Before performing the strength test, the aggregate sample was first dried in an oven at 105 °C until a constant weight was achieved and then cooled down to room temperature. Next, the aggregate was compacted into a steel cylinder and loaded, as shown in Fig. 4. Three samples of the same material were tested, and the mean value was taken into consideration. The crushing resistance was calculated using the following equation:

\[ C_a = \frac{L + F}{A} N/mm^2 \]

where \( C_a \): the bulk crushing resistance (MPa), \( L \): the weight of the piston (N), \( F \): the force recorded at 20 mm of compression for 100 s (N), and \( A \): the area of the piston (mm\(^2\)).

Fig. 5 illustrates the physical and mechanical properties of the manufactured aggregate. It is evident from the results that aggregate performance was significantly affected by the cement content, and the speed and angle of the pelletizer disc.

The effect of these parameters on pelletization efficiency is shown in Fig. 5a. It is clear that efficiency was improved by increasing cement content, reaching as high as 95% when a cement content of 20 wt-%, angle of 40° and speed of 40 rpm were used. However, at the same angle and speed but with a cement content of 5%, efficiency decreased to about 83%. This improvement can be directly ascribed to the role of cement as a binder; increasing the cement content helps to effectively hold the fly ash particles together because of being stickier. Moreover, the results show that encapsulation efficiency can also be enhanced by increasing the speed of rotation. This can be tentatively attributed to the formation of a denser cover matrix around EP particles, as a result of increasing the speed of rotation. As such, more powder materials were consumed and thus productivity was enhanced.

The variations in the particle density and 24-hour water absorption, caused by changes in the manufacturing parameters, are shown in Fig. 5b and c, respectively. It can be seen that the aggregate produced using a high speed possessed higher particle density and lower water absorption, compared to aggregate produced using a low speed. This confirms the assertion that increasing the speed of rotation promotes densification of the cover matrix.

The water absorption rate, of only the LWA₂₀⁻₅⁻, was measured at several time intervals; 5, 15, 30, and 60 min. As can be seen in Fig. 5e, an insignificant difference in water absorption was observed at 5 and 60 min. Moreover, in comparing Fig. 5c and e, it can be seen that water absorption at 5 min was equal to about 80% of that at 24 h. This phenomenon can have a harmful effect on the workability of fresh concrete. Therefore, to avoid the problem, some researchers have recommended presoaking aggregate with water before using it in concrete [16,17].

The results of bulk density tests in Fig. 5d show the same trend as those of particle density. Noticeable changes in loose bulk density occurred due to variations in speed and cement content, with an increase occurring together with increases in cement content and rotation speed.

Fig. 5f presents the 28 days bulk crushing strength for all aggregate samples. As can be seen, cement content is the main factor that affected crushing resistance. The speed of rotation was of secondary significance, while the effect of the angle was negligible.
For example, at a fixed angle of 35° and at a speed of 30 rpm, increasing the cement content from 5 to 20 wt-% strongly enhanced the crushing strength, from 0.34 to 2.04 MPa. This can be explained by the formation of calcium silicate hydrate (C-S-H) from the cement hydration itself, which increases with increasing the cement content. In addition, the hydration of OPC produces about 15–25 wt-% of calcium hydroxide (CH) [18]. Therefore, by increasing the cement content, the released amount of CH is also increased. This additional amount of CH is consumed by the pozzolanic compounds of FA to form pozzolanic C-S-H [19], which also contributes to enhancing the cover matrix strength, thus improving the bulk crushing resistance of the LWAs. It can also be observed from Fig. 5f that crushing strength was affected by the speed of rotation. For instance, at an angle of 35° and cement content of 20 wt-%, when the rotation speed increased from 20 to 40 rpm, the aggregate resistance improved from 1.93 to 2.07 MPa.
From the previous discussion, it can be concluded that the aforementioned parameters have differential influences on the characteristics of produced aggregates. Therefore, to determine the optimal values of those parameters, both density and crushing strength were mainly considered. In this regard, the specific strength factor was calculated as a ratio of crushing strength to particle density. Fig. 6 presents the specific strength factor for the aggregate specimens. It demonstrates that, despite the use of different speeds and angles, specific strength factors tend to converge as the cement content increases. This means that the impact of the mechanical parameters (angle and speed) on both density and crushing strength declines with an increase in cement content. This impact can be neglected at a cement content of 20 wt-%, as can be clearly seen in Fig. 6. Because of this evaluation, the LWA20-35-30 manufactured by using a cement content of 20 wt-%, angle of 35° and speed of 30 rpm exhibited the highest specific crushing strength factor of about 1.79 MPa/cm³, with corresponding particle density, crushing strength, and loose bulk density of 1.14 g/cm³, 2.04 MPa, and 636 kg/m³ respectively.

The major disadvantage of using 20 wt-% cement content is the high cost of cement, which affects the cost of the manufactured aggregates. Nevertheless, the influence of cement cost on the total cost of cold-bonded LWAs may be explained through a comprehensive-economic analysis. Different aspects should be considered in this analysis: manufacturing technique, production efficiency, practical use and quality of aggregate produced, recycling waste materials, energy consumption etc.

3.4. Utilization of EPP in the manufacturing process of LWAs

Optical microscopy was used to identify the shape and the inner structures of the aggregate specimen. Fig. 7a shows the irregular shape and high surface porosity of EP particle, which was used as a core structure. A hardened specimen with an almost spherical shape can be seen in Fig. 7b. Furthermore, the contact zone between the core and the shell structure seems to be dense and without cracks or big pores as shown in Fig. 7c. This may be attributed to the high surface porosity of the perlite particle, which secures a good interlock with the cover layer. The cross-section image (Fig. 7d) indicates that the cover matrix occupies about 80% of the total pellet volume. As such, there are two options, which would help to achieve the primary objective of this work; the production of LWAs with a particle density ≤1 g/cm³. The first way would be to decrease the volumetric ratio between the shell and the core, resulting in a decreased thickness of the cover matrix, which would definitely cause a substantial loss in crushing resistance and also adversely affect the productivity. The second way would be to reduce the density of the cover matrix by incorporating EPP as a partial replacement of fly ash. As is discussed in more detail below, the advantage of EPP is not only its very low weight, compared to fly ash, but that it might also have a pozzolanic activity similar to, if not higher than, fly ash.

Consequently, with the aim of lowering the particle density of manufactured aggregates according to the second method above, expanded perlite powder was incorporated into the shell mixture as a partial replacement of fly ash. In this respect, five LWA samples were produced at replacement levels of 10%, 20%, 30%, 40%, and 50% (by volume) as shown in Table 2. A volumetric replacement was used to maintain the total volume of the powder mixture constant, thus producing aggregate with a same size of 4–8 mm as well as same cement content per the unit volume of the cover matrix. In this step, the cement content of the cover matrix, angle and speed of pelletizer disc being set to the previously estimated values of 20 wt-%, 35° and 30 rpm, respectively.

4. Tests for LWAs produced with EPP

In addition to the tests carried out earlier (particle density, water absorption, loose bulk density and bulk crushing strength), the following investigations were performed to evaluate the effect of EPP on the properties of manufactured aggregate.

4.1. Micro-CT imaging to investigate general pore characteristics

For cementitious materials, the pores inside the samples are a critical factor in determining the performance of the material. There are several methods for investigating the pore characteristics of a specimen: mercury intrusion porosimetry (MIP), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and computed tomography (CT). Amongst these, micro-CT images can be effectively used to describe internal structure without damaging the specimen of a material. For cementitious materials, micro-CT imaging has been utilized to characterize material components, such as pores and aggregates [20,21]. A component within the order of micrometers can be identified using micro-CT imaging.

Micro-CT imaging is shown in Fig. 8. In Fig. 8a, the image on the left is an original 8-bit micro-CT image, which is represented with values between 0 and 255 (a total of 256). To classify a specific component from the original image as shown in Fig. 8b, an appropriate threshold value needs to be selected. Here, a multi-threshold method based on a modified Ostu algorithm [22] was adopted to classify the aggregate solid (gray in Fig. 8b), pores (black), and background (white). The original and segmented CT images in Fig. 8 are composed of 500 × 500 pixels with a 29.7 μm pixel size; this denotes that the pores larger than 29.7 μm can be detected using the micro-CT images used in this study. Fig. 8c shows the 3D image of a single aggregate particle and its inner structure. As can be seen in this figure, the pores located in the middle of the aggregate as well as the aggregate shell were effectively described and detected. Here, three samples of each aggregate type were measured using micro-CT, and their porosities were investigated to examine material characteristics.

4.2. X-ray diffraction and scanning electron microscopy

X-ray diffractometry (XRD) is one of the most distinguished analytical methods in cement-based material characterization. In this investigation, XRD measurement was carried out to study changes in the phase development of the aggregate produced.
Samples with an age of 28 days were firstly cut, and the perlite core was totally removed as shown in Fig. 9a. Next, the cover matrix was finely ground and used for XRD analysis. Furthermore, to observe the extent of EPP influence on the microstructure of the aggregate produced, scanning electron microscopy was performed on a cross section of the particle, which was prepared as in Fig. 9b.

### 4.3. Thermal conductivity

A transient plane source method, meeting the requirements of ISO 22007-2 [23], was used for measuring the thermal conductivity of LWAs via the Hot Disk device. In this method, the Hot Disk sensor is used as a source of heat, which diffuses into the sample, and as a dynamic temperature sensor which records the temperature increase over time. To perform the measurements and avoid heat losses during the test, an expanded polystyrene mold with dimensions of 20×20×12 cm³ was prepared and used to hold the testing sample. Before starting the test, the aggregate samples were dried in an oven at 105 °C until a constant mass was achieved and then cooled down to room temperature at moisture free conditions.

<table>
<thead>
<tr>
<th>Aggregate type</th>
<th>Replacement level (vol.-%)</th>
</tr>
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<tbody>
<tr>
<td>LWA0*</td>
<td>0</td>
</tr>
<tr>
<td>LWA10</td>
<td>10</td>
</tr>
<tr>
<td>LWA20</td>
<td>20</td>
</tr>
<tr>
<td>LWA30</td>
<td>30</td>
</tr>
<tr>
<td>LWA40</td>
<td>40</td>
</tr>
<tr>
<td>LWA50</td>
<td>50</td>
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</tbody>
</table>

* LWA0 refers to LWA20-35-30.

![Fig. 7. Microscopy images (a) EP particle, (b) hardened LWA, (c) cross-section of LWA, (d) interfacial transition zone.](image)

![Fig. 8. Micro-CT imaging of a sample aggregate: (a) original micro-CT, (b) segmented micro-CT, (c) 3D image of the aggregate particle (Note: in Fig. 8b, the black represents pores within the aggregate, the gray is the solid part, and the white is the background.)](image)
Thereafter, the polystyrene mold was filled with aggregate in two layers, with the sensor being centrally positioned in between. The Hot Disc device as well as the test process are shown in Fig. 10.

5. Results and discussions

The effects of EPP as a partial replacement of fly ash on the properties of manufactured aggregate and production efficiency are presented in Fig. 11. The sample at a replacement level of 0% refers to LWA20-35-30 used as a control specimen. The properties of LWA20-35-30 have been presented again in Fig. 11 to facilitate comparisons with samples containing different EPP contents. Firstly, the influence of EPP on productive efficiency has been illustrated in Fig. 11a. Compared to LWA0, the rate of productivity reduced marginally up to a replacement level of 40%. It decreased from 85% to 80% at this level. However, at a level of 50%, the efficiency deteriorated sharply to reach 65%. The reason for that could be due to the high amount of EPP particles, which need more cement content to be collected with fly ash particles to form a complete shell structure. Secondly, as has been shown in the following discussion, the overall performance of the aggregate was considerably affected by the use of EPP.

5.1. Density and thermal conductivity of the LWAs

Fig. 11b and c show the oven-dry particle density and loose bulk density of LWAs produced with different dosages of EPP. An inverse relation between particle density and EPP content can be observed. As can be seen in Fig. 11b, the aggregate density significantly dropped from 1.14 to 0.88 g/cm³ when the EPP content was increased from 0 to 50%. A similar trend can be clearly recognized for the loose bulk density (Fig. 11c). An increase in EPP content from 0% to 50% resulted in a reduction in bulk density from 636 kg/m³ to 540 kg/m³. However, the results in Fig. 11d indicate a strong direct link between water absorption and EPP content. LWA0 with a 0% EPP content showed the lowest water absorption of 32.8%, while, increasing the level of replacement to 30% (LWA30), resulted in a high increase in water absorption (about 32% more than LWA0). The reason for this lies in the very high porosity of EPP, which dramatically affected the water absorption of the aggregate manufactured.

Fig. 11e illustrates the changes in aggregate thermal conductivity. Since the perlite is an insulating material, the thermal insulation properties of the manufactured aggregates were enhanced when EPP was incorporated into the cover matrix. LWA50 showed a 37% reduction in thermal conductivity compared to LWA0. The low thermal conductivity can be attributed to two main factors. The first is the use of expanded perlite as a core structure, which has a low thermal conductivity of about 0.05 W/(m·K). The second is the implementation of expanded perlite powder in the shell structure, which enhanced its thermal properties.

5.2. Porosity of the LWAs

The porosity results of the samples obtained from the micro-CT images are presented in Fig. 11f and g. In these figures, the effect of EPP on the porosity of each aggregate can be examined. Pores larger than 29.7 μm, which significantly affect the material properties, are only considered due to the image resolution. The pores of core and solid matrix are discriminated using the imaging segmentation method [19]. As shown in Fig. 11f, the entire porosity of the aggregate increased as the content of EPP increased. In particular, the porosity of the cover matrix increased as more EPP was included in the aggregate, while that of the core part stayed within a constant range; this denotes that EPP content affects the pore characteristics of the cover matrix of the artificial aggregate. In addition, these porosity characteristics strongly affect the physical proper-
ties of cementitious materials since the pore characteristics are a dominant factor in determining material properties, such as strength and thermal conductivity.

5.3. Bulk crushing strength

The role of EPP in enhancing the 28-day crushing strength of the aggregate is shown in Fig. 11h. It is obvious from the results that, up to a replacement level of 40%, crushing resistance was enhanced continuously with an increase in the content of EPP. Interestingly, the partial replacement of fly ash with EPP decreased the particle density of the aggregate manufactured and significantly improved its crushing strength. The decrease in particle density is attributed to the increase in the EPP fraction, which has a low density, and decrease in the fly ash fraction, which has a higher density than EPP. While, the increase in crushing strength can be attributed to the high surface area as well as the pozzolanic reactivity of EPP, which has been reported in previous studies [24,25]. Yu et al. [24] have concluded that natural perlite powder is a good active mineral admixture for concrete due to its significant pozzolanic effect. In addition, Vosoughi et al. [25] have studied the effects of using EPP as a cement replacement in concrete. They reported a compressive strength increase of about 20% at 20% replacement, while a replacement level of 30% did not affect concrete strength. The results in Fig. 11a and h indicate that both, production efficiency and crushing strength were significantly decreased at 50% EPP content. By increasing the replacement level of EPP more cement is needed to achieve efficient pelletization. The structure of the manufactured aggregate was negatively affected by the deterioration of production efficiency and thus the crushing strength was decreased.

Therefore, the effect of EPP, as a partial replacement of fly ash, on the aggregate crushing strength suggests that the pozzolanic activity of EPP could be higher than that of fly ash. This interpretation is based on several reasons: the large specific surface area of EPP which is much higher than that of fly ash, and the chemical composition of EPP being composed mainly of aluminum oxide (Al₂O₃) and a high content of silicon dioxide (SiO₂) as illustrated in Table 1. In the investigations following below, the pozzolanic activity of fly ash and EPP have been examined and compared in detail.

The physical and mechanical properties of core-shell structured LWAs are summarized in Table 3, and compared with the properties of cold-bonded LWAs produced in previous studies.
5.4. Microstructure and mineralogy of the LWAs

Scanning electron microscopy was used to examine the influence of EPP on the microstructure of 28-day-old aggregate. The SEM measurements were performed on two different specimens: LWA0 as a control sample with 0% EPP and LWA40 with the use of EPP at replacement level of 40 vol-%. The SEM images of LWA0 and LWA40 are shown in Fig. 12a and b respectively. In general, the aggregate produced is composed of three structures; the honeycomb core structure of expanded perlite, a porous shell structure and a transition zone between the core and shell structure. Fig. 13 compares the shell microstructure of LWA0 and LWA40. Non-reacted fly ash particles were still evident in the case of LWA0 as shown in Fig. 13a. Moreover, ettringite and calcium hydroxide crystals (CH) formed on the surface of the fly ash particles can be clearly distinguished. Therefore, it can be inferred that a low rate of pozzolanic reaction may have occurred in the cover matrix of LWA0. On the other hand, in the case of LWA40, Fig. 13b shows a clear microstructural change stemming from the replacement of fly ash with EPP. It can be noticed that the fly ash particles almost disappeared and large amounts of hydration products were generated. This implies that the pozzolanic reaction was significantly accelerated in the cover matrix of LWA40. Furthermore, Fig. 14 evaluates the interfacial transition zone between the core and shell structures. A weak transition zone, as a result of the poor formation of hydration products, can be observed in the case of LWA0 (Fig. 14a). In contrast, as shown in Fig. 14b, the growth of pozzolanic reaction products on the surface of the core structure contributed effectively to densifying the transition zone of LWA40. These SEM observations confirm that the incorporation of EPP as a partial replacement of fly ash helped to improve the microstructure of the aggregate produced. This may explain the contribution of EPP in enhancing the 28-day crushing strength, despite its role in decreasing the particle density.

### Table 3

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</thead>
<tbody>
<tr>
<td>Raw material</td>
<td>Fly ash, cement, and EPP</td>
<td>Cement and GGBFS</td>
<td>Fly ash and cement</td>
<td>Fly ash, cement, and quarry dust</td>
<td>Cement kiln dust, G BFS, and marble sludge</td>
<td>Fly ash, cement, and GGBFS</td>
<td>Fly ash and cement</td>
</tr>
<tr>
<td>Particle density (g/cm$^3$)</td>
<td>0.88–1.14</td>
<td>510–636</td>
<td>2.14</td>
<td>1.23–1.44</td>
<td>1.25–2.0</td>
<td>1.70–1.98</td>
<td>1.26–1.47</td>
</tr>
<tr>
<td>Bulk density (kg/m$^3$)</td>
<td>510–636</td>
<td>2.14</td>
<td>857–972</td>
<td>950–1100</td>
<td>1.25–2.0</td>
<td>1.70–1.98</td>
<td>765.5–875.7</td>
</tr>
<tr>
<td>Water absorption (wt-%)</td>
<td>33–52</td>
<td>4.7</td>
<td>20.8–34.4</td>
<td>16–21</td>
<td>6.15–8.31</td>
<td>0.6–7.4</td>
<td>18–25.6</td>
</tr>
<tr>
<td>Crushing strength (MPa)</td>
<td>2.04–2.66</td>
<td>19.8</td>
<td>16.3</td>
<td>0.6–7.4</td>
<td>4.73–5.98</td>
<td>4.73–5.98</td>
<td>3.7</td>
</tr>
</tbody>
</table>

* Bulk crushing strength according to EN 13055 Annex C – procedure 1.
** Particle crushing strength.

Fig. 12. SEM images of the cross-section of LWA; (a) LWA0 and (b) LWA40.

Fig. 13. SEM images of the shell structure; (a) LWA0 and (b) LWA40.
The XRD patterns of aggregate samples, LWA0 and LWA40, have been provided comparatively in Fig. 15. The strength of the cement system is associated with the formation of C-S-H phases, which are located around two theta angles of 29° and 32° [27]. It is clear from the measurements that LWA40 shows a higher amount of C-S-H as compared to LWA0. In the cement-fly ash-EPP system (LWA40), due to the very high specific surface area of EPP, the pozzolanic reaction was accelerated and took place between calcium hydroxide released from cement hydration and EPP to produce pozzolanic C-S-H. On the other hand, in the cement-fly ash system (LWA0), a slower pozzolanic reaction occurred and less C-S-H phases were produced. This means that with the EPP incorporation, C-S-H formation was enhanced, as shown by XRD, which is in agreement with the crushing strength results.

5.5. The role of EPP as a mineral admixture

Based on the results of the aforementioned investigations, it can be clearly observed that the EPP is more effective than fly ash in improving the characteristics of manufactured aggregates. Nevertheless, further tests, including compressive strength, strength activity index, hydration heat flow, and XRD were performed to explain and compare the effectiveness of fly ash and EPP. In this regard, these materials were incorporated as mineral admixtures in pure cement pastes. A total of seven cement paste mixtures were produced, using different partial substitutions of cement, as shown in Table 4. A water/powder ratio of 0.5 was used in all the mixtures. The cement paste was mixed and prepared according to ASTM C305 [28]. The fresh paste was poured into 40 x 40 x 160 mm³ prism molds and covered with a polyethylene sheet to avoid evaporation. The specimens were demolded after 24 h and cured under water until the testing date. The results of 28-day compressive strength tests have been summarized in Table 4. Compared to M1 as a control mix (using only cement), replacing cement by FA in M2 and M3 resulted in a marked decrease in compressive strength. It decreased by about 11% and 14% at a replacement level of 6 wt-% and 12 wt-% respectively. While, M5 containing 12 wt-% EPP, exhibited a compressive strength similar to M1, whereas, the

Table 4

<table>
<thead>
<tr>
<th>Mix NO.</th>
<th>Type of cement paste</th>
<th>Cement (wt-%)</th>
<th>FA (wt-%)</th>
<th>EPP (wt-%)</th>
<th>Compressive strength (MPa)</th>
<th>Activity index %</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>C100F0E0</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>54.2</td>
<td>–</td>
</tr>
<tr>
<td>M2</td>
<td>C94F6E0</td>
<td>94</td>
<td>6</td>
<td>0</td>
<td>49.0</td>
<td>90</td>
</tr>
<tr>
<td>M3</td>
<td>C88F12E0</td>
<td>88</td>
<td>12</td>
<td>0</td>
<td>47.7</td>
<td>88</td>
</tr>
<tr>
<td>M4</td>
<td>C94F0E6</td>
<td>94</td>
<td>0</td>
<td>6</td>
<td>53.1</td>
<td>98</td>
</tr>
<tr>
<td>M5</td>
<td>C88F0E12</td>
<td>88</td>
<td>0</td>
<td>12</td>
<td>54.4</td>
<td>100</td>
</tr>
<tr>
<td>M6</td>
<td>C94F3E3</td>
<td>94</td>
<td>3</td>
<td>3</td>
<td>52.0</td>
<td>96</td>
</tr>
<tr>
<td>M7</td>
<td>C88F6E6</td>
<td>88</td>
<td>6</td>
<td>6</td>
<td>51.9</td>
<td>96</td>
</tr>
</tbody>
</table>
strength hardly decreased in M4 where 6 wt-% of cement was replaced with EPP. In contrast, the strength activity index of the pastes was calculated according to ASTM C311 [29], as a compressive strength ratio between the test mixtures (M2 to M7) and the reference sample (M1). It is obvious from the strength index that increasing the EPP content was often accompanied by an increase in the activity index. Consequently, it can be concluded that, at the given replacement levels, the expanded perlite powder revealed a higher reactivity than fly ash. This is mainly attributable to the large specific surface area of EPP, as compared to that of fly ash. This pozzolanic behavior of perlite as a mineral admixture is consistent with the results of Erdogan et al. [30], who has reported that 25–50 wt-% of OPC can be replaced with natural perlite without a significant negative impact on the compressive strength of cement paste.

The hydration heat development of the mixes 1, 3, 5 and 7 were further examined using isothermal calorimetry. In this test, 10 g of each mix and 5 g of water were mixed for 45 s and placed in an isothermal calorimeter (MC-CAL/100P) at 20 °C for 7 days. The heat flow diagrams of the hydrating paste mixtures are illustrated in Fig. 16. Compared to M1 as a reference, M3 with 12 wt-% replacement of cement with FA, showed the lowest heat flow. In addition, its main peak occurred a little bit later than the reference peak. In contrast, the main peak of M5, with 12 wt-% of EPP, appeared about one hour earlier and exhibited approximately the same heat flow as M1. The heat flow behaviors of M3, M5 and M7 showed that an increase in the EPP content is associated with an earlier main peak. Moreover, as shown in Fig. 16, the cumulative hydration heat curves of M3, M5 and M7 illustrate an inverse relationship between the total hydration heat and FA content. The total hydration heat tends to decrease as the fly ash content increases. Accordingly, it can be argued that the pozzolanic reaction is accelerated by increasing the content of perlite powder, while the opposite occurs with fly ash.

To predict the pozzolanic reactivity of EPP compared to that of fly ash, the amorphous characteristics of EPP and fly ash were observed by XRD as shown in Fig. 17. The XRD pattern of EPP exhibits generally amorphous characteristics. The existence of quartz indicates that the EPP has a siliceous nature amorphous phase. The difference between the XRD patterns of EPP and fly ash is the presence of crystalline quartz in fly ash. Accordingly, the amorphous structure of EPP as well as its high surface area explain its important role in enhancing the properties of aggregate produced.

![Fig. 16. Calorimetry curves of cement paste: (a) the rate of hydration heat development, (b) Cumulative hydration heat.](image)

![Fig. 17. X-ray diffraction patterns of EPP and fly ash (Q: quartz, Mu: mullite, Pe: periclase).](image)
6. Conclusions

In this experimental study, a new cold bonding method was developed to manufacture core-shell structured lightweight aggregates. Based on the obtained results, the following conclusions can be drawn:

- Cement content of 20 wt-%, angle of 35°, and speed of 30 rpm were beneficial to produce lightweight aggregates with highest specific strength factor (crushing strength/density).
- The particle and loose bulk density of aggregate produced ranged between 0.88 and 1.14 g/cm³ and 510–636 kg/m³, respectively. While, the bulk crushing strength was in a range of 2.04–2.66 MPa.
- The incorporation of expanded perlite powder (EPP) as a fly ash replacement, up to 40%, strongly improved the properties of aggregates produced.
- The high surface area of EPP as well as its amorphous structure denoted by XRD, explain its role as a pozzolanic material in increasing the crushing strength and densifying the transition zone as observed by the SEM.
- The results of cement paste, hydration heat flow and compressive strength, emphasized the high effectiveness of EPP compared to fly ash.
- The manufacturing technique presented in this study may help to increase the practical use of cold-bonded LWAs, thus reducing both energy consumption and pollution caused by the sintering method. At the same time, this new method may encourage the employment of a wide range of waste materials in the production of lightweight aggregates.

Further work focusing on the utilization of core-shell structured lightweight aggregate for the production of lightweight aggregate concrete, aiming to demonstrate the practical applications of the aggregate produced, has already been planned and initiated.

Conflict of Interest

None.

Acknowledgments

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References

4.3. An experimental study of using core-shell structured lightweight aggregate in producing lightweight concrete

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Authors: Feras Tajra, Mohamed Abd Elrahman, Dietmar Stephan
An experimental study of using core-shell structured lightweight aggregate in producing lightweight concrete

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Abstract

In this study, a cold bonding method was employed to produce low-density core-shell lightweight aggregates (CSLWAs) by the encapsulation of expanded perlite particles in a shell structure. The characteristics of the aggregate produced were investigated through particle density, water absorption, loose bulk density, and bulk crushing strength. In addition, the CSLWAs were used as normal-weight coarse aggregate replacement to produce lightweight aggregate concrete (LWAC). The properties of normal-weight concrete (NWC) and LWAC were evaluated in terms of consistency, dry density, water absorption, 28-day compressive strength, and thermal conductivity. This experimental work presents a new method for producing cold-bonded LWAs that satisfy both, a low particle density and good mechanical properties. The findings of this study also demonstrate the feasibility of using such aggregates for the production of structural lightweight aggregate concrete.

Keywords: cold-bonded lightweight aggregate; expanded perlite; production efficiency; bulk crushing strength; pozzolanic activity

1. Introduction

Recently, lightweight aggregate concrete (LWAC) attracts great interest and large industrial demand in a wide range of construction projects [1-2]. LWAC offers many technical, economic, and environmental advantages due to its lower density and thermal conductivity while compared to normal-weight concrete (NWC) [3]. LWAC is made by replacing normal-weight aggregates with lightweight aggregates (LWAs) which can be natural or artificial. Artificial LWAs can be produced via sintering technique or cold bonding method. Due to the high energy consumption of sintering method, a lot of attention has been paid to the development of cold-bonded LWAs. Many studies in recent years have focused on recycling different by-products or waste materials as LWAs to produce LWAC [4-8]. In this research, the first objective is to produce a low-density core shell lightweight aggregate (CSLWAs) using cold bonding method by the encapsulation of expanded perlite particles (EP) within a cover matrix composed of cement and fly ash. Thereafter, expanded perlite powder (EPP) was incorporated into the manufacturing process to reduce the particle density of the aggregate produced as low as possible. In this regard, EPP was used as a partial replacement of fly ash to reduce the density of the shell matrix, thus reducing the particle density of the aggregates. Moreover, studying the feasibility of using such type of aggregate in producing lightweight aggregate concrete is the second objective of this experimental work.
2. Experiments

2.1. Production of CSLWAs

CSLWAs were manufactured by encapsulating of expanded perlite particles (EP) within a cover matrix composed of 20 wt.-% ordinary Portland cement (CEM I 42.5R) and 80 wt.-% class F fly ash. The physical properties and chemical compositions of the cement and fly ash are listed in Table 1. The CSLWAs were prepared using a pelletizer disc, 40 cm in diameter and 10 cm in depth. Based on the production efficiency, the rotating angle and speed of pelletizer disc were 35° and 30rpm, respectively. The schematic illustration of aggregate production is presented in Fig. 1. In order to produce a nearly spherical aggregate with a size range of 4 - 8 mm, 25 g of perlite particles were sprayed with 1000g powder and 120 ± 10 g water. By using 1-2 mm perlite particles as cores, CSLWAs with size fraction of 2-4 mm have been produced. After production, the fresh pellets were put in sealed plastic bags for 24 h, then, they were cured under water until testing day. The microscopy images in Fig. 2 presents the shape and the inner structures of the aggregate specimen.

Table 1. Chemical and physical properties of cement, FA, and EPP [wt.-%].

<table>
<thead>
<tr>
<th>Material</th>
<th>CaO</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>MgO</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>Loss on ignition</th>
<th>Specific density</th>
<th>Surface area (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 42.5R</td>
<td>64.2</td>
<td>18.8</td>
<td>5.1</td>
<td>3.3</td>
<td>1.8</td>
<td>0.1</td>
<td>0.9</td>
<td>3.05</td>
<td>3.11</td>
<td>1.440</td>
</tr>
<tr>
<td>FA</td>
<td>4.4</td>
<td>55.9</td>
<td>22.6</td>
<td>6.7</td>
<td>1.9</td>
<td>0.8</td>
<td>1.9</td>
<td>2.50</td>
<td>2.34</td>
<td>1.620</td>
</tr>
<tr>
<td>EPP</td>
<td>0.9</td>
<td>76.9</td>
<td>11.3</td>
<td>0.9</td>
<td>0.1</td>
<td>3.6</td>
<td>3.6</td>
<td>1.02</td>
<td>2.38</td>
<td>6.290</td>
</tr>
</tbody>
</table>

Fig. 1. Production process: (a) pelletizer disc; (b) perlite particles before encapsulation; (c) fresh pellets

Fig. 2. Microscopy images: (a) hardened specimen; (b) cross-section of aggregate sample

Thereafter, in order to produce low-density CSLWAs, the aggregate were produced by incorporating expanded perlite powder (EPP) into the shell matrix. In this regard, EPP with a size <125 µm was used as a partial replacement of fly ash. 40 vol.-% of fly ash was replaced with EPP, as the maximum replacement ratio which demonstrated an insignificant effect on the productivity. The properties of EPP are presented in Table 1. It is clear from the table that
EPP has much higher surface area compared to fly ash, which affect the characteristics of the manufactured aggregates significantly.

2.2. Production of normal and lightweight aggregate concrete

In this research, three different concrete mixes were prepared and tested; one with normal-weight aggregates as a control mix and two with the manufactured CSLWAs. Natural quartzite aggregates (NA) with a density of 2600 kg/m³, water absorption of 0.5 %, and size ranges of 0 - 2, 2 - 4, and 4 - 8 mm were used for the production of normal-weight concrete (NWC). For LWAC, CSLWAs, made with EPP, with a size of 2 – 4 mm and 4 – 8 mm were utilized. Superplasticizer with a density of 1.08 g/cm³ was used to achieve reasonable consistency. CEM I 42.5R and silica fume, as a cement replacement at 10 wt.-%, were used as binders. Water / binder ratio of 0.36 was maintained constant for all mixes. Due to the high water absorption of the CSLWAs, they were immersed before mixing in a specific amount of water, which is equal to their water absorption. This procedure (pre-soaking) is recommended by some researchers [9-10], in order to avoid the effect of aggregate water absorption on the properties of fresh concrete. The volume fractions of the aggregate components were kept constant for all mixes, being 26, 17 and 57 vol.-% for the sizes of 0 - 2, 2 - 4 and 4 - 8 mm, respectively. In order to produce LWAC, the normal weight coarse aggregates were replaced volumetrically with CSLWAs at replacement levels of 50 and 100 vol.-%. Table 2 presents the mix proportions of the concrete mixtures. The fresh concrete was casted into 100 mm cubic steel molds in accordance with EN 12390-1 [11]. The specimens were covered with a polyethylene sheet to avoid evaporation, then they were demolded after 24 hours and cured at 20 ± 1 °C and relative humidity of 99% until the testing day.

Table 2. Mix proportions of concretes (kg/m³)

<table>
<thead>
<tr>
<th>Mix No.</th>
<th>NWC*</th>
<th>LWAC50**</th>
<th>LWAC100***</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix Type.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>w/b</td>
<td>0.36</td>
<td>0.36</td>
<td>0.36</td>
</tr>
<tr>
<td>Silica fume</td>
<td>45</td>
<td>45</td>
<td>45</td>
</tr>
<tr>
<td>Cement</td>
<td>405</td>
<td>405</td>
<td>405</td>
</tr>
<tr>
<td>Water</td>
<td>162</td>
<td>162</td>
<td>162</td>
</tr>
<tr>
<td>Superplasticizer</td>
<td>4.4</td>
<td>4.4</td>
<td>4.4</td>
</tr>
<tr>
<td>Normal sand 0 – 2 mm</td>
<td>939</td>
<td>939</td>
<td>939</td>
</tr>
<tr>
<td>Normal aggregate 2 – 4 mm</td>
<td>278</td>
<td>139</td>
<td>0</td>
</tr>
<tr>
<td>Normal aggregate 4 – 8 mm</td>
<td>425</td>
<td>213</td>
<td>0</td>
</tr>
<tr>
<td>CSLWAs 2 – 4 mm (SSD)</td>
<td>0</td>
<td>79</td>
<td>157</td>
</tr>
<tr>
<td>CSLWAs 4 – 8 mm (SSD)</td>
<td>0</td>
<td>116</td>
<td>232</td>
</tr>
</tbody>
</table>

* Normal-weight concrete. ** LWAC produced with 50% NA and 50% CSLWAs. *** LWAC produced with 100% CSLWAs.

2.3. Test procedures

Water absorption and oven-dried particle density of the manufactured aggregate were determined according to EN 1097-6, Annex C [12]. Loose bulk density was measured in accordance with EN 1097-3 [13]. While, 28-day bulk crushing strength of the produced aggregate was investigated according to EN 13055 Annex C – procedure 1 [14] as shown in Fig. 3. The bulk crushing strength was calculated using the following equation:

\[ C_a = \frac{L + F}{A} \ N/mm^2 \]

Where \( C_a \): the bulk crushing resistance (MPa), \( L \): the weight of the piston (N), \( F \): the force recorded at 20 mm of compression for 100 seconds (N), and \( A \): the area of the piston (mm²).
Fig. 3. Determination of bulk crushing resistance: (a) vibrated sample; (b) loading the sample via a piston; (c) crushed specimen and thermal conductivity test: (d) Hot Disk sensor; (e) sensor with a specimen

Consistency of fresh concrete was assessed using a flow table test according to EN 12350-5 [15]. Dry density and 28-day compressive strength of hardened concrete were conducted as specified in EN 12390-7 [16] and EN 12390-3 [17], respectively. Furthermore, water absorption of concrete samples was calculated using the following equation:

$$\text{Water absorption (\%)} = \frac{W_s - W_d}{W_d}$$

Where: $W_s$ and $W_d$ = the mass of saturated surface-dry and oven-dry specimen, respectively.

A transient plane source method, meeting the requirements of ISO 22007-2 [18] was used for measuring the thermal conductivity via the Hot Disk device as shown in Fig. 4. Before performing the thermal conductivity test, the concrete samples were first dried in an oven at 105 °C until a constant mass was achieved and then cooled down to room temperature at moisture free conditions.

3. Results and discussion

3.1. Properties of CSLWAs

The evaluated properties of the aggregate produced are presented in Table 3. A particle density of 1.14 g/cm$^3$, loose bulk density of 636 kg/m$^3$ with corresponding crushing strength of 2.04 MPa were achieved. Since the oven-dry particle density and bulk density of the aggregate produced are less than 2 g/cm$^3$ and 1200 kg/m$^3$, respectively, the CSLWAs are classified as lightweight aggregates and confirm EN 13055-1 requirements [14]. As can be seen in Table 3, an insignificant difference in the water absorption was observed at 5 and 60 minutes, moreover, the water absorption at 5min was equal to about 80% of that at 24h. This indicates the high water-avidity of the aggregate produced. Therefore, to avoid the adverse effect of the water absorption on the workability of fresh concrete, the CSLWAs were used in concrete at saturated surface-dry (SSD) condition.

Table 3. Physical and mechanical properties of CSLWAs

<table>
<thead>
<tr>
<th>Size of aggregate</th>
<th>Particle density (g/cm$^3$)</th>
<th>Loose bulk density (Kg/cm$^3$)</th>
<th>Bulk crushing strength 28 days (MPa)</th>
<th>Water absorption over time (wt.-%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4-8 mm</td>
<td>1.14</td>
<td>636</td>
<td>2.04</td>
<td>5 min 15 min 30 min 60 min 24 h</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>26.9 27.8 28.1 28.7 33</td>
</tr>
</tbody>
</table>

The effect of EPP as a partial replacement of fly ash on the properties of manufactured aggregate is presented in Table 4. The results show that replacing fly ash by EPP, at replacement level of 40 vol.-%, resulted in a marked decrease in the particle and bulk density by about 17% and 14%, respectively. This is attributed to the porous structure of the cover matrix formed by the EPP. On the contrary, due to the high porosity of EPP, water absorption exhibited an opposite trend to that of particle and bulk density. It increased from 33 to 44 wt.-% when the EPP was incorporated as a fly ash replacement. Interestingly, the use of EPP contributed to improving the crushing strength of the aggregate produced. The aggregate produced with cement, fly ash, and EPP showed a crushing strength of 2.66 MPa, which is about 30% higher than that of aggregate prepared without EPP. The reason for this lies in the high pozzolanic activity of EPP, mainly stemming from its very high specific surface area. The high surface area of EPP contributed to
accelerating the pozzolanic reaction in the aggregate shell, thus improving the 28-day crushing strength. This pozzolanic activity of expanded perlite has been studied and proven in previous studies [19-20].

The properties of CSLWAs with a size of 2-4 mm produced with cement, fly ash, and EPP are also presented in Table 4, where OD and SSD refer to the oven-dry and saturated surface dry particle density, respectively.

<table>
<thead>
<tr>
<th>Size of LWAs</th>
<th>Particle density (g/cm³)</th>
<th>Bulk density (kg/cm³)</th>
<th>Bulk crushing strength 28 days (MPa)</th>
<th>Water absorption (wt-%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SSD OD</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4-8 mm</td>
<td>1.41</td>
<td>0.98</td>
<td>549</td>
<td>2.66</td>
</tr>
<tr>
<td>2-4 mm</td>
<td>1.44</td>
<td>1.00</td>
<td>566</td>
<td>3.54</td>
</tr>
</tbody>
</table>

3.2. Properties of normal and lightweight concrete

Table 5 shows the physical and mechanical properties of normal and lightweight concrete. The measured flow diameters of all mixtures ranged from 42 to 45 cm, which are classified as type (F3) according to EN 206-1 [21]. The cut surface of the concrete samples are shown in Fig. 5. As can be seen, both normal and lightweight aggregates are homogeneously distributed in the concrete matrix. This indicates the sufficiently viscosity of the cement paste, which helped to avoid the segregation in the concrete mixes.

The dry density of the concrete mixes ranged from 2130 to 1700 kg/m³. These results demonstrate that concrete produced with CSLWAs has a lower density than concrete with NA. The dry densities of LWAC50 and LWAC100 mixtures were about 10% and 20% less than NWC, respectively. The concretes produced using 50% and 100% CSLWAs have a dry density of 1915 and 1700 kg/m³, and can be classified under EN 206-1 as light-weight concrete D2.0 and D1.8, respectively.

The compressive strength values were in the range of 26-50 MPa. The compressive strength was reduced by increasing the replacement of NA with CSLWAs. The concrete produced only with the normal aggregate has a strength of 50 MPa, while, the LWAC50 and LWAC100 showed a compressive strength of 33.7 and 26 MPa, respectively. This reduction in compressive strength is mainly attributed to the crushing strength of CSLWAs, which is much lower than that of normal-weight aggregate. Based on the results of compressive strength, the lightweight concrete produced (LWAC50 and LWAC100) are classified as a structural lightweight-aggregate concrete [22].

On the other hand, NWC showed the lowest water absorption of 5.3 wt.-%, while, increasing the CSLWAs content resulted in a significant increase in the water absorption, which was 10.2 % for LWAC50 and 16 % for LWAC100. The reason for this lies in the high water absorption of the CSLWAs compared to normal aggregate.
Due to the low density and high porosity of the manufactured lightweight aggregates, the thermal insulation properties of the concrete were enhanced when the normal-weight aggregates were replaced with CSLWAs. The lightweight concrete made of 50 and 100% CSLWAs (LWAC50 and LWAC100) showed a 28 and 43% reduction in thermal conductivity compared to NWC respectively.

4. Conclusions

Based on the obtained results, the following conclusions can be drawn:

- This study presents a new method for producing cold-bonded lightweight aggregate, with a core-shell structure that satisfy both a low particle density and good mechanical properties.
- The using of expanded perlite powder as a fly ash replacement in the cover matrix of the manufactured aggregate contributed effectively to lowering the particle density and enhancing the 28-days crushing strength of the CSLWAs.
- The properties of concretes produced indicate that the core-shell lightweight aggregates are feasible for the production of structural lightweight-aggregate concrete.

Acknowledgments

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4. Publications

4.4. Properties of lightweight concrete made with core-shell structured lightweight aggregate

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Authors: Feras Tajra, Mohamed Abd Elrahman, Christian Lehmann, Dietmar Stephan
Properties of lightweight concrete made with core-shell structured lightweight aggregate

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b Department of Structural Engineering, Mansoura University, Egypt

HIGHLIGHTS
- Core-shell structured lightweight aggregate (CSA) was produced by cold bonding method.
- Effects of curing method and surface treatment on properties of CSA were investigated.
- CSA and expanded clay aggregate (ECA) were used for production of lightweight concrete.
- Mechanical and durable Properties of concrete made of either CSA or ECA were compared.

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ABSTRACT
In this study, core-shell structured lightweight aggregates (CSA) were produced through the cold bonding method, by encapsulating an expanded perlite particle (as a core structure) within a shell matrix composed of cement, fly ash and expanded perlite powder. The effect of different curing regimes on the mechanical and microstructural properties of the CSA were studied. To enhance the characteristics of the aggregates produced, they were surface treated by a mixture of cement and silica fume, using two different treatment methods. Afterwards, the properties of lightweight concrete made of either CSA or expanded clay aggregate (ECA) were compared closely in terms of their potential economic and environmental benefits, in response to the high energy consumption associated with the production of ECA. The results revealed that curing at a relative humidity of 99% is the most appropriate curing method for CSA. In addition, treating the CSA surface contributes significantly to enhancing its bulk crushing strength, by about 14–18%. The findings also demonstrate the feasibility of using CSA to produce lightweight aggregate concrete, with a dry density and compressive strength ranging from 1115 to 1540 kg/m³ and from 17.9 to 25.8 MPa, respectively, with a corresponding thermal conductivity range of 0.3169–0.6660 W/m K.

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1. Introduction

Lightweight aggregate concrete (LWAC) has recently attracted great interest and large industrial demand in a wide range of construction projects [1,2]. LWAC has lower density and thermal conductivity than conventional concrete and offers many technical, economic and environmental advantages [3,4]. Compared to normal-weight concrete (NC), LWAC is made either by replacing all normal-weight aggregates, or only the coarse fraction, with lightweight aggregates (LWAs), which can be natural or artificial [5]. Due to the depletion of natural resources, particular attention has been paid to the utilization of artificial LWAs produced either by sintering or by cold bonding method.

Sintered LWAs such as expanded clay (Liapor and Leca) and expanded glass (Poraver and Liaver), produced at a temperature of about 1200 °C, are widely utilized because of their very low density and good mechanical properties [6]. Yu et al. [7] have used LWAs manufactured from recycled glass to produce ultra LWAC with a dry density range of 650–700 kg/m³ and a corresponding compressive strength of 8–12 MPa. Lo Yu et al. [8] have produced LWAC with a 28-day compressive strength of 29–43 MPa and a density of 1617–1850 kg/m ³, using normal-weight sand and expanded clay coarse aggregate.

Compared with sintering method, cold bonding method is characterized by minimal energy consumption and lower pollutants emission [9]. Moreover, being the aggregates occupy about
65–75% of the total volume of concrete [10], cold bonding method can be considered a great opportunity for converting different and an enormous amount of waste and by-product materials into added value product. Therefore, many studies in recent years have concentrated on the behavior of LWAC made of cold-bonded produced with different waste materials [11–15].

However, although the performance of cold-bonded LWAC has been demonstrated, little attention has been paid to emulating the performance of LWAC produced with sintered LWAs available in the market; the main reason for this could be the high density of cold-boned LWAs, as compared to that of sintered ones. Therefore, a new cold-bonded method was developed in previous study to produce core–shell structured LWA with low density as a potential construction material for the production of lightweight aggregate concrete. The production process, as well as the properties of core–shell LWA (CSA), have been exhaustively explained and studied in [16].

In this study, the practical applications of CSA in the production of LWAC are studied deeply by evaluating the mechanical and durability behavior of CSA concrete, in comparison with ECA concrete, aiming to increase the use of cold-bonded LWAs, which may have a positive impact from an environmental and economic perspective.

2. Experimental program

This research was divided into two phases. The first phase consisted of producing CSA by the encapsulation of expanded perlite particles, within a cover matrix composed of cement, fly ash and expanded perlite powder. Being that this kind of aggregate is new, two necessary investigations were performed as an important step toward studying its practical applications in concrete. The scope of these investigations covered two different aspects: (1) determining the effects of different curing methods on the properties of the aggregate produced, to choose and employ the most appropriate curing process; (2) treating the aggregate surfaces with a cement-silica fume mixture for the purposes of enhancing their properties. The second phase of the experimental work was extensively carried out in regard to the feasibility of using CSA for the production of LWAC. The behavior of CSA concrete was studied and compared with concrete produced with expanded clay aggregates, in terms of mechanical properties and durability.

2.1. First stage

2.1.1. Raw materials and the production of CSA

As demonstrated in detail in a previous study [16], a pelletizer disc of 40 cm diameter and 10 cm depth was used and operated at an angle of 35° and a speed of 30 rpm. To produce CSA, expanded perlite particles (EP) were encapsulated in a shell structure composed of 18 vol% ordinary Portland cement (OPC, CEM I 42.5R), 49 vol% class F fly ash (FA) and 33 vol% expanded perlite powder (EPP), with a particle size <125 μm. Two different sized fractions of CSA, 2–4 and 4–8 mm, were manufactured by using 1–2 and 2–4 mm perlite particles as cores, respectively. The particle size distributions of the raw materials are shown in Fig. 1. The physical and chemical properties of OPC, FA and EPP are presented in Table 1.

2.1.2. The effects of curing methods on the strength development of CSA

Cold-bonded LWAs require an appropriate curing regime and a sufficient curing period to achieve enough strength to be put into practice [17,18]. In this investigation, to identify the suitable curing method for the CSA, three different practical methods were applied: under water (UW), under a controlled relative humidity of 65% (RH 65%) and under a controlled relative humidity of 99% (RH 99%). These curing methods were applied at a temperature of 20 ± 1 °C until the day of testing. Since the strength of LWAs has a considerable impact on the properties of LWAC [19,20], the bulk crushing strength of CSA, subjected to different curing methods, was closely studied. It was tested according to EN 13055, Annex C [21], using a steel test cylinder, 113 mm in diameter, and 100 mm in height. The bulk crushing strength was measured by dividing the force, recorded at 20 mm of compression for 100 s, by the specimen area. This investigation was performed on oven-dried CSA with a size range of 4–8 mm. Three aggregate samples of each curing method were tested, and the mean value was taken into consideration.

The results in Fig. 2 show that, up to an age of 28 days, an increase in curing duration resulted in a significant increase in the crushing strength. All the aggregates gained about 75% of their 28-day strength within 7 days, exhibiting a further marginal improvement in their crushing resistance when the curing period was extended to 56 days. A maximum crushing strength of 3.13 MPa was achieved by the aggregate cured at RH 99%, whereas the aggregate cured at RH 65% showed the lowest strength, of 2.12 MPa. Compared with the UW curing method, the RH 99% method enhanced the crushing strength by about 17%, while it decreased by about 29%, 26% and 21% at an age of 7, 28 and 56 days, respectively, when RH of 65% was applied. This evaluation shows that curing at RH 99% is more beneficial than the other methods for the crushing strength enhancement of CSA. This tendency will be explained and discussed deeply in the following investigations.

2.1.3. The effects of curing methods on the microstructural characteristics of CSA

In this aspect, thermogravimetric analysis (TGA), X-ray diffraction (XRD) and SEM tests were performed on the cover matrix of the aggregate produced to justify why the aggregate cured at RH 99% possessed higher crushing strength than that cured under water and at RH 65%. To perform the XRD and TGA tests, the aggregate sample was firstly dried in an oven at 105 °C until reaching a constant weight, after removing the perlite core, the cover matrix was ground and used for the tests.

2.1.3.1. Thermogravimetric analysis (TGA). Fig. 3a and 3b show the thermogravimetric analysis of the aggregate under different curing methods, at an age of 7 and 56 days, respectively. Calcium hydroxide content was calculated based on the mass loss due to decomposition of Ca(OH)2, between 400 and 500 °C [22], using the following equation:

\[
CH = W_{CH} \times \frac{74}{18}
\]
Where: $W_{CH}$ is the mass loss due to the evaporation of water between 400 and 500 °C, 74 and 18 are the molecular masses of calcium hydroxide and water (g/mol), respectively.

It was noticed that, from the calculated values of CH content (Fig. 4), at an age of 7 days the aggregates cured at RH 99% exhibited the highest CH content, followed by those cured under water and finally those cured at RH 65%. As the hydration of OPC produces about 15–25 wt% of calcium hydroxide (CH) [23], the CH content at an early age of a cement-pozzolan system indicates the extent of cement hydration; therefore, at an age of 7 days, a high degree of cement reaction, associated with the highest quantity of CH and highest crushing strength, might have been caused by curing at RH 99%. Alternatively, after 28 days, the pozzolanic reaction starts by consuming the available CH, converting it into more hydration products [14]. Therefore, at an age of 56 days, less CH content can be considered as an indicator of a high pozzolanic reaction rate and thus more formation of C-S-H phases associated with higher strength. This correlation is clearly demonstrated at an age of 56 days, where the findings in Figs. 3 and 4 show an obvious inverse relation between the CH content and the crushing strength. Consequently, it can be concluded that among the curing methods studied, curing at RH 99% effectively enhances the reaction in the cover matrix of CSA, thus improving its bulk crushing strength.

2.1.3.2. X-ray diffraction (XRD). The correlation between TGA analysis and crushing strength results was further proved by observing the intensity of the CH peak from XRD analysis. Consideration of the CH peak, located at around the two theta angle of 18°, is the key performance indicator for a cement-pozzolan system [24]. As evident in Fig. 5a, the 7-day-old CSA cured at RH 99% showed the highest CH peaks, followed by that cured under water and then by that cured at RH 65%. The highest CH peak can be a consequence of a high rate of cement hydration accompanied by more C-S-H. Curing at RH 99% therefore resulted in a significant increase in the crushing strength of CSA, compared to the other curing methods. The XRD patterns of 56-day-old CSA in Fig. 5b, indicate that the intensity of CH peaks decreased significantly with an increase

<table>
<thead>
<tr>
<th>Table 1</th>
<th>The chemical and physical properties of OPC, FA, and EPP.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>CaO</td>
</tr>
<tr>
<td>OPC</td>
<td>64.2</td>
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<tr>
<td>FA</td>
<td>4.4</td>
</tr>
<tr>
<td>EPP</td>
<td>0.9</td>
</tr>
</tbody>
</table>

![Fig. 2. Bulk crushing strength development.](image)

![Fig. 3. TGA results of CSA under different curing methods at a) 7 days, b) 56 days.](image)

![Fig. 4. Calcium hydroxide content under different curing methods and ages.](image)
in the curing period. This is attributable to the pozzolanic reaction, as mentioned before. It can be noticed from Fig. 5b, that the CSA cured at RH 99% exhibited the lowest CH peak, which can be explained by the high pozzolanic reaction rate achieved with this method.

2.1.3. Scancling electron microscopy (SEM). The microstructures of 7 and 56-day-old CSA are shown in Figs. 6 and 7, respectively, with a noticeable microstructural difference between the differently cured aggregates being visible. At an age of 7 days, the microstructures of CSA cured at RH 65% and under water (Fig. 6a and b) were porous. Only a low amount of hydration products, on the surface of fly ash particles, can be seen in the CSA cured at RH 65% (Fig. 6a), with the microstructure of CSA cured under water (Fig. 6b) being somewhat denser, due to the formation of more hydration products. These observations denote a relatively low stage of cement hydration achieved by these two methods. Conversely, in the case of CSA cured at RH 99% (Fig. 6c), the availability of a high concentration of CH, which later takes part in the pozzolanic reaction, is a sign of a high degree of cement hydration.

After 56 days of curing, the presence of CH and ettringite in Figs. 7d and 7e can be explained by a relatively low pozzolanic reaction rate, caused by curing at RH 65% and under water. At the same time, the absence of CH crystals, which were consumed and converted into more hydration products (Fig. 7f), confirm that the pozzolanic reaction sped up when the RH 99% method was applied. Accordingly, it can be concluded that curing at RH 99% resulted in an increase in the rate of cement and pozzolanic reaction in the cover matrix of the CSA, which is consistent with its high crushing strength, as compared with the CSA cured under water and at RH 65%.

2.1.4. The effects of surface treatment on CSA properties

The high water absorption of LWAs negatively affects the mechanical and durability performance of LWAC [25]. Many researchers have reported that surface treatment of lightweight aggregate substantially decreases its water absorption while enhancing its mechanical properties [14,26]. In the present study, the manufactured aggregates with a size range of 4–8 mm were surface-treated by a cement-silica fume mixture, using two different methods. A schematic diagram of the CSA cross section is pre-
sented in Fig. 8. After producing the CSA, the fresh pellets were kept in sealed bags for 24 h, after which they were treated either by a cement-silica fume slurry, or by coating with a thin film of cement-silica fume. In the first method, the slurry was made of 90 wt% OPC, 10 wt% silica fume (SF) and a water/powder ratio of 1.2. The aggregate was immersed in the slurry for 30 min and then sieved to filter out the excess slurry. Thereafter, the treated aggregate was spread over a mesh to avoid particle-particle cohesion and cured at RH 99%, until the date of testing. However, in the second method, the aggregates were first soaked in water for 5 min, after which the water was totally filtered out. The saturated particles were introduced into the pelletizer disc, which was rotated at a speed of 15 rpm. Next, cement-silica fume powder mixture composed of 90 wt% OPC and 10 wt% SF was sprayed on the particle surfaces. Afterwards, the treated samples were removed from the pelletizer and cured at RH 99%, until the date of testing.

The optical microscope images in Fig. 9 clearly show the shape and cross section of ECA. As it is known, ECA is prepared by heating clay at a temperature of 1200 °C, the high temperature inciting the clay to expand to several times its original size and to form spherical pellets with a very highly porous structure [27]. Fig. 9 also shows the shape and cross section of the CSA. Compared with non-treated CSA, the slurry treatment led to the formation of an irregular layer on the particle surface. Moreover, the cross section of the slurry-treated particle clearly indicates the inefficiency of this method to completely cover the whole surface of the particle. In contrast, the nearly spherical shape of the CSA particle was maintained by the application of a coating treatment, performed using the pelletizer disc. This method enabled the powder mixture to be uniformly distributed on the whole surface of the particle, covering it with a thin uniform film of approximately 300 μm thickness, as shown clearly in Fig. 9.

After 28 days of curing at RH 99%, the effect of the surface treatment was evaluated through the physical and mechanical properties of the treated and non-treated CSA. The physical properties were measured in terms of water absorption, oven-dry (OD) particle density and loose bulk density, in accordance with EN 1097-6 [21] and EN 1097-3 [28]. These tests were performed on three samples of each aggregate type, and the average value was taken into evaluation. The manufactured aggregates were coded in Table 2 as: CSA for non-treated aggregates as a control sample; CSA.SL and CSA.CO for slurry-treated and coating-treated aggregates, respectively.

The results in Table 2 indicate that the cold bonding technique can be successfully employed to produce CSA with a low particle and loose bulk density of 0.98 g/cm³ and 549 kg/m³, respectively, with a corresponding crushing strength of 3.11 MPa. Compared to CSA, as a control specimen, the particle density increased slightly from 0.98 to 1.00 g/cm³, while water absorption decreased marginally from 44 to 42 wt%, when the slurry treatment was applied. This insignificant effect is attributable to the inability of this method to secure full cover on the particle surface. Moreover, this method resulted in the formation of protrusions on the surfaces of the aggregates, as shown in Fig. 9. These protrusions contributed to weakening the filling ability of the CSA.SL, thus decreasing both the loose bulk density and bulk crushing strength, as illustrated in Table 2. In contrast, the coating treatment method

![Fig. 6. SEM images of CSA at age of 7 days cured at; (a) RH 65%, (b) UW, (c) RH 99%.

![Fig. 7. SEM images of CSA at age of 56 days cured at; (d) RH 65%, (e) UW, (f) RH 99%.

![Fig. 8. Schematic diagram of the pellet cross section.](image)
resulted in a marked improvement in the physical and mechanical properties of the aggregate produced. The particle density, loose bulk density and bulk crushing strength of CSA.CO increased by about 13%, 11% and 14%, respectively, compared with CSA. In addition, the formation of a dense thin film on the aggregate surface effectively decreased water absorption by about 27%. Consequently, it can be concluded that the coating treatment achieved a good efficiency, resulting in a stronger and much more impermeable shell structure, as shown by the higher bulk crushing resistance and lower water absorption, as compared with the non-treated aggregates.

Based on the results of the first stage, it was decided to use the RH 99% curing condition and the coating surface treatment, to produce the aggregates for the second stage of the research.

2.2. Second stage

In this part, a large quantity of CSA with a size range of 2–4 and 4–8 mm was produced and treated by the coating method as described above, the aggregates then cured at RH 99% for 28 days. Afterwards, the applicability of these kinds of LWAs for the production of lightweight aggregate concrete was studied in detail.

2.2.1. Raw materials and production of lightweight aggregate concrete

A total of six lightweight concrete mixtures were prepared and tested. In all the mixes, a binder content of 450 kg/m³, composed of 90 wt% OPC and 10 wt% SF and a water/binder ratio of 0.36 were used. The mixes were divided into two groups, differing in terms of the type of fine aggregate used. Natural quartzite sand (NS) and lightweight expanded clay sand (ECS) were utilized to produce the mixes of groups 1 and 2, respectively. Furthermore, expanded clay aggregate (ECA), non-treated CSA and surface-treated CSA (CSA.ST) were used as a coarse LWA. To study only the effects of aggregate type on concrete properties, the particle size of all aggregate types was controlled by sieving; 0–2 mm for the fine fraction and 2–4 and 4–8 mm for the coarse fraction. The physical and mechanical properties of the aggregates used are presented in Table 3. To eliminate the effects of aggregate water absorption on the properties of the fresh concrete, the LWAs were immersed in water before mixing and used in a saturated surface-dry condi-

<table>
<thead>
<tr>
<th>Type of CSA</th>
<th>Particle density g/cm³</th>
<th>Loose bulk density kg/cm³</th>
<th>Water absorption wt.-%</th>
<th>Bulk crushing strength MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSA</td>
<td>0.98</td>
<td>549</td>
<td>44</td>
<td>3.11</td>
</tr>
<tr>
<td>CSA.SL</td>
<td>1.00</td>
<td>540</td>
<td>42</td>
<td>2.77</td>
</tr>
<tr>
<td>CSA.CO</td>
<td>1.11</td>
<td>608</td>
<td>32</td>
<td>3.55</td>
</tr>
</tbody>
</table>

![Fig. 9. Microscopy images of ECA, treated and non-treated CSA.](image-url)
tion (SSD), as recommended in [29,30]. Polycarboxylic ether superplasticizer (PCE), with a density of 1.08 g/cm³ was used to achieve reasonable consistency. A viscosity modifying admixture was utilized as a stabilizer to prevent concrete segregation. Table 4 presents the mix proportions of the concrete mixtures, labeled by aggregate type as ECA-NS, CSA-NS and CSA.ST-NS for group 1 and as ECA-ECS, CSA-ECS and CSA.ST-ECS for group 2. The fresh concrete was cast into 100 mm cubical steel molds and 100 mm/200 mm cylinder molds, in accordance with EN 12390-1 [31]. The specimens were covered with a polyethylene sheet to avoid evaporation and then demolded after 24 h and cured at a relative humidity of 99%, until the testing day.

2.2.2. Test methods

The consistency of the fresh concrete was measured using a flow table test, according to EN 12350-5 [32]. The dry density and compressive strength tests of 100-100-100 mm³ specimens were conducted as specified in EN 12390-7 [33] and EN 12390-3 [34], respectively. A modulus of elasticity test was performed on 100-200 mm cylinder specimens, in accordance with EN 1048-5 [35]. The thermal conductivity of 100 mm oven-dried cube samples was evaluated via the Hot Disk device, model TPS 2200, as shown in Fig. 10.

The durability of LWAC was investigated by means of capillary water absorption and water penetration tests. The capillary suction of water was evaluated according to EN ISO 15148 [36], as shown in Fig. 11a. The specimens were first dried at 105 °C, until a constant weight was achieved. The side surfaces of the specimen were then coated by a paraffin layer, to ensure unidirectional absorption. Afterwards, the concrete specimens were placed in a container, which was partially filled with water at a level of 5 mm above the bottom of the specimen. The weight gain due to the capillary water absorption was measured at several times intervals: 20 min, 1, 2, 3, 4, 5, 6, 7, 8 and 24 h. The capillarity coefficient was calculated using the following equation:

$$K = \frac{\Delta m_f - \Delta m_0}{\sqrt{t_f}}$$

Table 3

<table>
<thead>
<tr>
<th>Type of LWAs</th>
<th>Size of LWAs</th>
<th>Particle density</th>
<th>Bulk density</th>
<th>Water absorption</th>
<th>Bulk crushing strength</th>
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<tr>
<td></td>
<td></td>
<td>SSD g/cm³</td>
<td>OD g/cm³</td>
<td>kg/cm³</td>
<td>wt%</td>
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<td>ECA 4–8 mm</td>
<td>0.70</td>
<td>0.51</td>
<td>310</td>
<td>35</td>
<td>1.44</td>
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<tr>
<td>ECA 2–4 mm</td>
<td>0.74</td>
<td>0.61</td>
<td>345</td>
<td>23</td>
<td>2.08</td>
</tr>
<tr>
<td>ECA 0–2 mm</td>
<td>1.00</td>
<td>1.17</td>
<td>620</td>
<td>37</td>
<td>–</td>
</tr>
<tr>
<td>NS 4–8 mm</td>
<td>2.65</td>
<td>2.6</td>
<td>1650</td>
<td>0.5</td>
<td>–</td>
</tr>
<tr>
<td>CSA 4–8 mm</td>
<td>1.41</td>
<td>0.98</td>
<td>549</td>
<td>44</td>
<td>3.11</td>
</tr>
<tr>
<td>CSA 2–4 mm</td>
<td>1.44</td>
<td>1.00</td>
<td>566</td>
<td>43</td>
<td>3.54</td>
</tr>
<tr>
<td>CSA.ST 4–8 mm</td>
<td>1.48</td>
<td>1.11</td>
<td>608</td>
<td>32</td>
<td>3.55</td>
</tr>
<tr>
<td>CSA.ST 2–4 mm</td>
<td>1.50</td>
<td>1.10</td>
<td>622</td>
<td>36</td>
<td>4.20</td>
</tr>
</tbody>
</table>

* Saturated surface-dry.
** Oven-dry.

Table 4

<table>
<thead>
<tr>
<th>Mix No.</th>
<th>1</th>
<th>2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>M1</td>
<td>M2</td>
</tr>
<tr>
<td>Mix ID</td>
<td>ECA-NS</td>
<td>CSA-NS</td>
</tr>
<tr>
<td>w/b</td>
<td>0.36</td>
<td>0.36</td>
</tr>
<tr>
<td>Silica fume</td>
<td>45</td>
<td>45</td>
</tr>
<tr>
<td>Cement</td>
<td>405</td>
<td>405</td>
</tr>
<tr>
<td>Water</td>
<td>162</td>
<td>162</td>
</tr>
<tr>
<td>Superplasticizer</td>
<td>4.4</td>
<td>4.4</td>
</tr>
<tr>
<td>Stabilizer</td>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>Coarse aggregate 4–8 mm</td>
<td>170</td>
<td>348</td>
</tr>
<tr>
<td>Coarse aggregate 2–4 mm</td>
<td>119</td>
<td>228</td>
</tr>
<tr>
<td>Sand 0–2 mm</td>
<td>593</td>
<td>593</td>
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</tbody>
</table>

* Saturated surface-dry condition.
Where: $K$ is the capillarity coefficient $\text{kg/(m}^2 \cdot \text{h}^{0.5})$, $\Delta m_0$ and $\Delta m_t$ are the amounts of water absorbed per unit area (kg/m$^2$) at 0 and 24 h respectively and $t_f$ is the testing period (h).

The water penetration test was performed in accordance with DIN EN 12390-8 [35], as shown in Fig. 11b, by applying 1 bar water pressure for 72 h, and then measuring the water penetration depth. All the aforementioned investigations were carried out on three concrete samples of each mix and the mean value was considered.

Furthermore, SEM was also performed on the concrete specimens to evaluate the interfacial transition zone between the aggregate and the cement matrix. The sample prepared for the SEM test is shown in Fig. 12.

### 3. Results and discussion

The measured flow diameter of the fresh concrete mixtures ranged from 42 to 45 cm, which classifies them as type F3, according to EN 206-1 [37]. It is clear from the slump flow of fresh concrete (Fig. 13) that no segregation or bleeding occurred in the concrete mixtures. In addition, as can be seen in Fig. 14a, b and c, the CSA, ECA and CSA.ST maintained their original shape, without apparent damage, which denotes that the aggregates had enough strength to avoid the probable crushing caused by the mixing of concrete.

#### 3.1. Physical and mechanical properties evaluation

The aim of this study was to evaluate the physical and mechanical properties of LWAC made with treated and non-treated CSA, compared with LWAC produced with expanded clay aggregate. Considering the mixes of group 1, produced with NS, the replacement of ECA in M1 with CSA (M2) resulted in an approximately 15% and 13% increase in dry density and compressive strength, respectively. M2 therefore presented a slightly lower strength/density ratio than M1, as shown in Fig. 15c. In contrast, as shown in Fig. 15a and b, M3 produced with CSA.ST showed a compressive strength of 29.1 MPa, associated with a dry density of 1540 kg/m$^3$; values which are higher than that of M1 by about 19 and 27%, respectively. The use of CSA.ST in M3 therefore leads to concrete with a strength/density ratio of 18.90, which is higher than that of M1 and M2. Interestingly, in comparing M2 (CSA-NC) and M3 (CSA-ST-NC), aggregate surface treatment contributed strongly to enhancing concrete strength by about 13%, from 25.8 to 29.1 MPa, despite its marginal effect on concrete density. This is mainly attributable to the crushing strength of CSA.ST, which is higher than that of CSA, as was shown above in Table 3. This evaluation confirms that the strength of LWAC is mainly affected by aggregate strength [38,39].

Fig. 13. Flow table test according to EN 12350-5.

The results of tests performed on concretes produced with expanded clay sand (group 2) show the same trend as those produced with NS. Compared to M4, replacing ECA by CSA.ST in M6 increased both the density and strength of concrete by about 26% and 42%, respectively. A marked increase in strength/density ratio was therefore also observed in Fig. 15c. Consequently, it can be emphasized that the surface treatment of CSA, described herein, is a convenient technique for enhancing concrete strength without a strong impact on its density.

Moreover, Fig. 16 indicates a strong correlation between the dry density and the compressive strength of the concrete produced, with a correlation coefficient $R^2$ of 0.9463. However, the results show that the dry density and compressive strength of concretes produced with treated and non-treated CSA ranged from 1115 to 1540 kg/m$^3$ and from 17.9 to 29.1 MPa, respectively. Therefore, this concrete covers the EN 206-1 LWAC density classes from D1.2 to D1.6. It can also be classified as a structural LWAC, according to its compressive strength [40].

Fig. 17 shows the crushed specimen after performing the compressive strength test. It is clear that both CSA and ECA were broken along their diameter, with the clear emergence of the perlite cores, shown in Fig. 17a, being proof of this. Concrete failure therefore took place in the LWAs, being the weakest ingredient of LWAC [41,42] and not in the interfacial transition zone (ITZ) between the LWAs and the cement matrix. This confirms that the failure of LWAC with a high cement content of more than 350 kg/m$^3$, usually happens in the LWA and as such, that the strength of LWAC containing a high cement content, is mainly affected by the strength of LWA [43].

The modulus of elasticity of LWAC is affected by the type and volume of the LWAs, as well as by the modulus of elasticity of the cement matrix [44,45]. Generally, the results in Fig. 15d show that the modulus of elasticity increased with the compressive strength of concrete, stemming from the replacement of ECA with CSA. In group 1, the modulus of elasticity increased from 10.85 to 11.75 GPa and from 10.85 to 12.5 GPa, when the ECA was replaced with CSA and CSA.ST, respectively. This is mainly attributable to the crushing strength of the CSA and CSA.ST, which is higher than that of ECA, as shown in Table 3. The possible reason for this might also be the difference in the pore structure of these aggregates, in consideration of the work of Kockal and Ozturan [46], who have reported that the porosity and pore size of LWAs have an impact on their modulus of elasticity, thus influencing the modulus of elasticity of LWAC. The results of modulus of elasticity tests in the mixes of group 2 show the same trend, with significantly lower values than that of those of group 1. This confirms that the cement matrix composition has a strong influence on the modulus of elasticity [47].

Table 5 also shows the estimated values of the modulus of elasticity, in accordance with BS and ACI standards. They were calcu-
lated in terms of dry density and cubic compressive strength, using the following equations, as specified in ACI 318 and BS 8110, respectively.

\[ E_c = W_c^{1.5} \times 0.043 \times \sqrt[0.8]{f_c} \]  
(5)

\[ E_c = W_c^2 \times 0.0017 \times 0.8^{0.33} \]  
(6)

Fig. 14. Cross section of concrete specimens made of ECA (a), CSA (b), and CSA.ST (c).

Fig. 15. Properties of LWAC (a) dry density; (b) compressive strength; (c) strength/density ratio; (d) modulus of elasticity.
The ratio between the measured and estimated values ranged from 98 to 118% in the mixes made with treated and non-treated CSA. This ratio is located in the typical range of 80–120%, as specified in ACI 318. At the same time, the values measured in the ECA concretes were 123–157 percent of the calculated values. Accordingly, the correlation coefficient R², for the values calculated as specified in ACI 318 and BS 8110, decreased from 0.9932 and 0.9962 to 0.9124 and 0.9117, respectively, when the mixes M1 and M4 were included in the analysis, as shown in Fig. 18a and b.

3.2. Thermal conductivity evaluation

Since the aggregate occupies the largest share of the concrete volume, it has a direct and significant influence on the thermal properties of concrete [48]. In this study, the thermal conductivity of the dried concrete was measured via a Hot Disk, as illustrated in Table 6. Compared with M1, thermal conductivity increased by about 39 and 45% when the CSA and CSA.ST were used in M2 and M3, respectively. This is attributable to the highly porous structure and low density of ECA, compared with CSA. Moreover, due to the density of ECS, which is much lower than that of NS, the thermal conductivity of the mixes in group 2, produced using ECS, was significantly lower than those in group 1. A rise in thermal conductivity, of about 6 and 14%, can be observed when ECA was replaced by CSA (M5) and CSA.ST (M6), respectively.

Asadi et al. (2018) [49] have demonstrated a relationship between thermal conductivity (\(\lambda\)) and dry density (\(\rho\)), by analyzing 185 results reported in the literature. They have proposed Eq. (7) for calculating the thermal conductivity of concrete, with a density range of 150–2350 kg/m³.

\[
\lambda = 0.0625e^{0.0015\rho}
\]  

(7)

In this study, Fig. 19 represents the relationship between the values measured for dry density and thermal conductivity. It confirms a good correlation between them, with a correlation coefficient R² of 0.9476. From this figure, Eq. (8) can be deduced to predict the thermal conductivity of the LWAC produced, being almost the same as Eq. (7):

\[
\lambda = 0.064e^{0.0015\rho}
\]  

(8)

3.3. Durability property evaluation

Fig. 20 shows the cumulative increase in specimen mass per unit area (\(\Delta m\)) resulting from water absorption by capillarity. It was determined at 20 min, 1, 2, 3, 4, 5, 6, 7, 8 and 24 h, as a function of the square root of time. Next, the concrete samples’ water capillarity coefficient was calculated with Eq. (2), as an average of three different samples for each concrete mix. The results, in Fig. 20, indicate that the type of lightweight aggregate and the composition of the cement matrix had a significant effect on the capillary water absorption of LWAC [50]. Fig. 21 shows that the substitution of ECA (M1) with CSA in M2 increased the capillary suction coefficient of concrete, from 0.33 to 0.48 kg/(m² h⁰.⁵). This is mainly attributable to the water absorption of CSA, which is higher than that of ECA. Interestingly, the water absorption coefficient dropped from 0.48 to 0.34 kg/(m² h⁰.⁵), which is roughly similar to that of M1, when the surface treated CSA was used. A similar trend can be discerned for the mixes of group 2, with a water capillary coefficient higher than the mixes of group 1, by about 65–70%. The reason for this lies in the very high water absorption of ECS, compared to NS. The results of M2, 3, 5, and 6 confirm the effectiveness of aggregate surface treatment in reducing the water permeability of concrete, thus enhancing its durability.

Furthermore, the effectiveness of aggregate surface treatment on concrete durability was also proved through the water penetration test performed on M1 and M3. Fig. 22a and b show the water penetration depth in the split samples of M1 and M3, respectively. The penetration depth of M1 was 19 mm, whereas M3 exhibited a penetration depth of 16 mm meaning that, although the difference between the water absorption of ECA and CSA.ST was as stated in

Table 5

<table>
<thead>
<tr>
<th>Group</th>
<th>Mix ID</th>
<th>Modulus of elasticity (GPa)</th>
<th>Measured value</th>
<th>Estimated values</th>
<th>Measured/Estimated ratio (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>BS 8110</td>
<td>ACI 318</td>
<td>BS 8110</td>
</tr>
<tr>
<td>1</td>
<td>M1 (ECA-NS)</td>
<td>10.85</td>
<td>7.44</td>
<td>8.58</td>
<td>146</td>
</tr>
<tr>
<td>1</td>
<td>M2 (CSA-NS)</td>
<td>11.75</td>
<td>10.23</td>
<td>11.22</td>
<td>115</td>
</tr>
<tr>
<td>1</td>
<td>M3 (CSA-ST-NS)</td>
<td>12.50</td>
<td>11.39</td>
<td>12.54</td>
<td>110</td>
</tr>
<tr>
<td>2</td>
<td>M4 (ECA-ECS)</td>
<td>5.35</td>
<td>3.41</td>
<td>4.34</td>
<td>157</td>
</tr>
<tr>
<td>2</td>
<td>M5 (CSA-ECS)</td>
<td>6.00</td>
<td>5.09</td>
<td>6.06</td>
<td>118</td>
</tr>
<tr>
<td>2</td>
<td>M6 (CSA-ST-ECS)</td>
<td>7.20</td>
<td>6.11</td>
<td>7.33</td>
<td>118</td>
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</table>
Table 3, the LWAC produced with treated CSA showed better resistance to water penetration than that produced with ECA. This could be due to the microstructure of the interfacial transition zone (ITZ), between the aggregate and the cement matrix, which affects the mechanical properties and durability of concrete [51,52].

The microstructure of the ITZ between the cement matrix and CSA.ST and ECA are shown in Fig. 23a and b, respectively. A good and continuous bond between the CSA.ST and the cement matrix can be seen in Fig. 23a. The boundaries of CSA are therefore relatively indistinguishable, especially since both the CSA and the cement matrix have nearly the same composition. On the contrary, the boundaries of ECA can be clearly recognized in Fig. 23b, because of its microstructure, which is totally different from that of the cement matrix. It can be noticed that the ITZ surrounding CSA.ST is denser than that surrounding ECA. This may be attributable to the internal curing caused by the water stored within the aggregates [53]. Consequently, the amount of water reserved in CSA, which is much higher than that stored in ECA, contributed continuously to supplying the needed curing for the hydration of ITZ, thereby forming a homogeneous and dense transition zone. Here, it should be pointed out that sample...
preparation was the main reason for the appearance of cracks in the cement matrix.

4. Conclusions

From the findings obtained in this experimental study, the following conclusions can be drawn:

- The cold bonding technique can be successfully employed to produce a new kind of cold-bonded LWA with low density, besides the possibility of producing different aggregate sizes.
- Core-shell LWAs in a size range of 2–4 mm and 4–8 mm were produced, having a particle density of 0.98 and 1 g/cm³ and crushing strength of 3.11 and 3.54 MPa, respectively.
- Among the curing methods studied, curing at a relative humidity of 99% is the most suitable method to ensure the best crushing strength of CSA.
- The strength development of CSA showed a strong correlation with the results of SEM, TGA and XRD tests.
- The dry density of CSA concrete ranged from 1115 to 1540 kg/m³. CSA are therefore feasible for the production of lightweight concrete with density classes from D1.2 to D1.6, as specified in EN 206-1.
- The compressive strength of CSA concrete ranged from 17.9 to 29.1 MPa and the thermal conductivity from 0.3169 to 0.666 W/m·K. This concrete can thus be classified as a structural and insulating concrete, with a higher thermal conductivity than ECA concrete.
- The incorporation of surface treated CSA increased the strength/density ratio and modulus of elasticity of CSA.ST concrete, as compared with ECA concrete.
- The water capillarity coefficient of LWAC made of CSA.ST is comparable to that of ECA concrete, while the water penetration resistance is better.

The results of this study encourage development of the production and application of core-shell aggregate produced through cold bonding techniques, which may offer important economic and environmental benefits.

Conflict of interest

The authors declare no conflicts of interest.

Acknowledgments

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References

5. Complementary experimental study

5.1. Drying shrinkage of concrete

Since the lightweight aggregate concrete mixtures designed and studied in section 4.4 have low water-binder ratio and high cementitious material content, they can be prone to high drying and autogenous shrinkage due to the environmental drying and cement hydration. A complementary study has, therefore, been performed to measure and evaluate the shrinkage of concretes under the influence of aggregate type. In this aspect, the fresh concrete was cast into prisms steel molds (40·40·160 mm$^3$) and covered with a polyethylene sheet to avoid evaporation. After 24 h, the concrete samples were demolded and exposed to drying in a humidity cabinet at 20 ± 1 °C and 65 ± 5% relative humidity. The length change measurements were performed at 3, 7, 14 days, and then every 14 days until an age of 56 days, and then every 28 days until age of 112 days. The shrinkage measurements, as well as the weight loss of the lightweight concrete mixes concerning drying time, are shown in Figure 5.1 and Figure 5.2, respectively. As mentioned in section 4.4, the concrete mixtures are labelled by the aggregate used; where ECA expanded clay aggregate, CSA core-shell aggregate, CSA.ST surface treated core-shell aggregate, NS normal sand and ECS expanded clay sand.

It is clear from Figure 5.1 that the highest shrinkage was measured in the concretes with ECA, followed by the concretes with CSA and then by that made of CSA.ST. For the mixes made with normal sand (group 1), namely, M1, M2 and M3, shrinkage at the age of 112 days decreased by about 8.5 % and 40.5 % when ECA was replaced with CSA and CSA.ST, respectively, whereas these differences were only 2.5 % and 7.7 % in the case of expanded clay sand concretes, M4, M5 and M6.

Figure 5.2 indicates that the weight loss of concretes made of ECA increased continuously up to drying age of 56 days. While in the case of CSA and CSA.ST, the weight loss steadily increased during the whole drying period. Since the water-cement ratio is constant for all mixes, the continuous increase in the weight loss means that the saturated CSA acts as a water reservoir, which is able to provide
5. Complementary experimental study

water into the concrete matrix for more extended period than that in the case of saturated ECA, thus delaying the drying shrinkage of CSA concrete.

On the other hand, despite the higher water absorption of CSA compared to CSA.ST, which can provide more internal curing amount of water to the concrete matrix during the drying, CSA concrete exhibited higher shrinkage than CSA.ST concrete. This means that the drying shrinkage of concrete is not only related to the role of aggregate as a water reservoir but also to the stiffness of the aggregate used; the higher the stiffness of aggregate, the lower the concrete shrinkage. Moreover, as can be observed in Figure 5.1, concretes made with ECS showed much higher shrinkage compared to that produced with NS. This confirms that the composition of the cement matrix also has a strong influence on concrete shrinkage. The use of ECS caused the production of concrete with higher water capillarity coefficient; thus lower dense microstructure compared to that made of NS, thereby resulting in the formation of lower strength and higher shrinkage concretes. The results of this investigation are consistent with the results presented in [1,2], where the shrinkage of lightweight concrete has been observed to be affected by the water absorption and crushing strength of the aggregate used as well as by the quality of the cement matrix.

However, at age of 112 days, the shrinkage values of the mixes made of normal sand, M1, M2 and M3, are 0.3719, 0.5719 and 0.625 mm/m, respectively, which are located in the standard shrinkage range of lightweight concrete, as recommended in EN 1992-1-1 [3]. While the mixtures of group 2 showed higher shrinkage than that of group 1, confirming that the replacement of normal-weight sand by lightweight sand has a significant impact on the shrinkage of the concrete. That high shrinkage can be reduced by the incorporation of steel or polypropylene fiber in the concrete mix design [4].
5. Complementary experimental study

**Figure 5.1.** Shrinkage of concrete mixes

**Figure 5.2.** Weight loss of concrete mixes
5. Complementary experimental study

5.2. Effect of CSA age on the concrete properties

Since the cold-bonded LWA needs an extended curing period, sometimes more than 28 days, to achieve an adequate strength for the practical applications [5], an experimental study has, therefore, been carried out to evaluate the effectiveness of the use of different age core-shell aggregates in the production of LWAC. In this study, a total of three concrete mixtures M1, M2 and M3, having same proportions and differing only in the age of the aggregate used (7, 14 and 28 days) were produced using similar materials to that used in section 4.3. Table 5.1 presents the mix proportions of the concrete mixtures, labelled by aggregate age as M7, M14 and M28.

<table>
<thead>
<tr>
<th>Mix type</th>
<th>M7 / M14 / M28</th>
</tr>
</thead>
<tbody>
<tr>
<td>w/b</td>
<td>0.36</td>
</tr>
<tr>
<td>Cement</td>
<td>405</td>
</tr>
<tr>
<td>Silica fume</td>
<td>45</td>
</tr>
<tr>
<td>Water</td>
<td>162</td>
</tr>
<tr>
<td>Superplasticizer</td>
<td>4.4</td>
</tr>
<tr>
<td>Normal sand 0-2 mm</td>
<td>939</td>
</tr>
<tr>
<td>CSA 2 – 4 mm</td>
<td>157</td>
</tr>
<tr>
<td>CSA 4 – 8 mm</td>
<td>232</td>
</tr>
</tbody>
</table>

To avoid possible crash of CSA, especially 7-day-old CSA, during mixing of concrete, the following mixing procedures were carried out: (1) sand, cement and silica fume were first dry mixed until they were thoroughly blended; (2) water and superplasticizer were then added and the mixture was mixed until complete homogenization; (3) CSA was lastly added and mixed with all constituents until a homogeneous concrete mix was achieved. The fresh concrete was cast into cube molds 100x100x100 mm³ and covered with a polyethylene sheet to avoid evaporation. The specimens were demolded after 24 hours and cured in controlled curing conditions with a relative humidity of 99% until the testing day.
5. Complementary experimental study

Figure 5.3 shows the microscopic images of the cross-section in the concrete sample made of 7-day-old CSA (M7). It can be seen that the CSA maintained its original shape without apparent damage. This means that the 7-day-old CSA has enough strength to avoid crushing during the mixing process of concrete.

![Microscopy images](image)

**Figure 5.3.** Microscopy images of cross-section in concrete made of 7-day-old aggregate (M7)

The results of dry density, compressive strength and thermal conductivity of concretes at the age of 28 days are illustrated in Table 5.2.

<table>
<thead>
<tr>
<th>Mix type</th>
<th>Dry density (kg/m³)</th>
<th>Compressive strength (MPa)</th>
<th>Thermal conductivity W/(m*k)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M7</td>
<td>1700</td>
<td>23.6</td>
<td>0.896</td>
</tr>
<tr>
<td>M14</td>
<td>1700</td>
<td>24.9</td>
<td>0.897</td>
</tr>
<tr>
<td>M28</td>
<td>1710</td>
<td>26.0</td>
<td>0.899</td>
</tr>
</tbody>
</table>

The results show that the compressive strength of M28, produced with 28-day-old CSA is higher than that produced with 7 and 14-day-old aggregates by about 10 and 4.5 %, respectively. Moreover, it can be noticed that the three mixes have the same dry density, and the differences between their thermal conductivity values are minimal. These findings show that the utilization of early age CSA is feasible without a significant impact on the mechanical properties of the concrete produced.
6. Main Results and achievements

6.1. Production and properties of CSA

As shown in section 4.2, a new kind of cold-bonded aggregate was produced through an encapsulation process using a pelletizer disc, 40 cm in diameter and 10 cm in depth. The core-shell structured lightweight aggregate (CSA) was firstly produced by encapsulating 2-4 mm expanded perlite particles within a shell matrix composed of cement and fly ash. The angles and speeds of the pelletizer were adjusted at (35° and 40°) and (20, 30, and 40 rpm), respectively, while the cement was used at a content of 5, 10, 15 and 20 wt.-%. The main results of this section can be summarized as follows:

The production efficiency of core-shell aggregate was in a range of 60 to 95 %; it has been observed to be dependent on the cement content of the cover matrix as well as on the angle and speed of pelletizer. The minimum productivity (60 %) was achieved at an angle of 35°, speed of 20 rpm and 5 wt.-% cement content and increased to 95 % when the angle, speed and cement content became 40°, 40 rpm and 20 wt.-%, respectively.

The encapsulation process used in this study showed the ability to produce CSA with a size range of 4 - 8 mm, particle density between 1.090 and 1.185 g/cm³, a loose bulk density from 581 to 649 kg/m³, water absorption from 30 to 38 wt.-% and a bulk crushing resistance varying between 0.15 and 2.08 MPa. The properties of CSA have been observed to be profoundly affected by the cement content and speed of rotation. Increasing the rotation speed leads to densifying the shell structure of the aggregate, thereby, resulting in the formation of CSA with lower water absorption and higher bulk crushing strength. While increasing the cement content generates more hydration product resulted from the cement hydration itself and from the pozzolanic reaction between fly ash and calcium hydroxide released from cement reaction, hence leading to a stronger CSA. It was observed that the use of an angle of 35°, speed of 30 rpm and cement content of 20 wt.-% leads to produce CSA with highest crushing strength / particle density ratio, with corresponding particle density, crushing
6. Results and achievements

resistance, and loose bulk density of 1.14 g/cm$^3$, 2.04 MPa, and 636 kg/m$^3$ respectively. Those parameters have been therefore adopted for the next experiments.

In the next step, expanded perlite powder (EPP) was incorporated in the shell structure to reduce its density and thus produce cold-bonded lightweight aggregate with a particle density lower than that produced in the literature. The results in section 4.2 showed that the incorporation of EPP as fly ash replacement contributes effectively to decrease both the particle and loose bulk density of CSA and meanwhile increases its crushing strength. This is mainly attributable, on the one hand, to the unit weight of EPP which is much lower than that of fly ash, on the other hand, to the amorphous structure of EPP and its high surface area compared with fly ash. The replacement of fly ash with EPP, at levels of 0, 10, 20, 30, 40 and 50 vol.-%, succeeded in producing cold-bonded CSA with a particle density between 0.88 and 1.14 g/cm$^3$, a loose bulk density from 510 to 636 kg/m$^3$ and a bulk crushing resistance varying between 2.04 and 2.66 MPa. The CSA produced at a replacement level of 40 vol.-% exhibited the highest crushing strength of 2.66 MPa, and has been therefore chosen for use in concrete.

6.2. Utilization of CSA as normal-weight aggregate replacement

In section 4.3, the CSA was used in concrete as a replacement of normal weight aggregate (NWA) to investigate its influence on concrete properties. Three concrete mixes have been prepared by replacing NWA with CSA at replacement levels of 0, 50 and 100 vol.-%. The results show that the 28-day compressive strength of the concrete made with CSA decreased with increasing the volume content of CSA. It was decreased from 33.7 MPa to 26 MPa, by about 23 %, when the replacement ratio increases from 50 to 100 vol.-%. This is attributed to the low strength of CSA compared with NWA. These findings are in agreement with the results of Tang et al. [6] who reported that increasing the cold-bonded aggregate content from 50 to 100 vol.-% has been observed to decrease the compressive strength of concrete by about 25 %.
6. Results and achievements

Being that the particle density of CSA is lower than that of NWA, the dry density of NWC decreased by about 10 and 20 % when NWA was replaced with CSA at levels of 50 and 100 vol.-% respectively. However, the CSA concrete produced in this section exhibited a dry density of 1915 and 1700 kg/m³ and can be therefore classified as lightweight aggregate concrete D2.0 and D1.8, in accordance with EN 206-1 [7].

The thermal conductivity of NWC has been found to decrease by about 28 % and 43 % when the NWA was replaced with 50 and 100 vol.-% CSA, respectively. Based on the results of these investigations, it can be said that the CSA has a potential to be used for the production of lightweight aggregate concrete, which gave the incentive to perform more investigations about the behavior of CSA concrete in comparison with the performance of expanded clay aggregate concrete.

6.3. Influences of curing regimes on the crushing strength of CSA

Since the manufactured aggregate in this research is new, it is necessary to determine its appropriate curing method before using it in concrete. Three different curing methods were therefore applied: under water (UW), under a controlled relative humidity of 65% (RH 65%) and under a controlled relative humidity of 99% (RH 99%). The influences of those methods on the crushing strength and microstructural properties of the CSA have been evaluated in section 4.4 to determine the best among them which can enhance the structure of the cover matrix and thus improve the crushing resistance of CSA. The results of bulk crushing strength (Figure. 2, section 4.4) showed that the aggregate cured at RH 99% exhibited a bulk crushing resistance of 3.11 MPa at the age of 28 days, while the CSA cured under water and at RH 65% demonstrated lower crushing resistance of 1.97 and 2.66 MPa, respectively. A similar trend can be recognized for the crushing strength results at the age of 7 and 56 days. This means that RH 99% is more profitable than the other methods to enhance the crushing strength development of CSA. Moreover, to explain and justify that, thermogravimetric analysis (TGA), X-ray diffraction (XRD) and SEM investigations have been performed on the shell structure of CSA to study the
6. Results and achievements

Changes in its microstructure and phase composition due to applying different curing methods. TGA is used to measure the calcium hydroxide (CH) content in the cover matrix of CSA, which depends mainly on the hydration degree of cement as well as the rate of the pozzolanic reaction; cement hydration produces CH, whereas the pozzolanic reaction consumes it. The TGA results at the age of 7 days show that the CSA cured under RH 99% has the highest CH content compared with that cured under water and at RH 65%, which means that a high degree of cement hydration was achieved by RH 99%. As expected, due to the pozzolanic reaction, reduction in CH content was observed at the age of 56 days. The liberated calcium hydroxide in the cover matrix of CSA cured under RH 99% was higher than that cured under water and at RH 65%, which indicates the high degree of pozzolanic reaction. Therefore it can be argued that curing at RH 99% contributed significantly to enhancing both the cement and pozzolanic reaction in the cover matrix of CSA, thereby improving its crushing strength. This effect was also emphasized by the results of XRD test, where the CSA cured at RH 99% showed the highest and the lowest CH peaks at the age of 7 and 56 days, respectively. Furthermore, the SEM images of CSA confirm the changes in their microstructure under the effect of curing methods. The highest concentrations of CH crystals were observed in the cover matrix of 7-day-old CSA cured at RH 99%. These CH crystals were converted, due to the pozzolanic reaction, into more hydration product added to that produced from cement hydration itself, hence leading to a stronger aggregate. Accordingly, it can be said that the results of crushing strength are well correlated with the observations of TGA, XRD and SEM.

6.4. Effects of surface treatment on the properties of CSA

As shown in section 4.1, different techniques, such as the incorporation of additives and alkaline activators in the production process, and surface treatments for the manufactured aggregate, can be performed for enhancing the performance of cold-bonded aggregate. In section 4.2, EPP was incorporated in the cover matrix of CSA as fly ash replacement and succeeded to reduce its particle density and improve its crushing resistance. In section 4.4, surface treatments for the aggregates
6. Results and achievements

produced were also performed to upgrade their physical and mechanical properties. In this aspect, the manufactured aggregates were treated by two methods: (1) immersing it in a slurry composed of 90 wt.-% OPC, 10 wt.-% silica fume (SF) and water/powder ratio of 1.2; (2) by covering their surfaces, during its rolling motion in the pelletizer, with a dense thin film of 90 wt.-% OPC and 10 wt.-% SF. The results of this investigation showed that the slurry treatment was unable to create a wholly surface-treatment and it was, therefore, infeasible to improve the properties of CSA. However, the coating method succeeded in creating a uniform film on the aggregate surfaces, thereby resulting in the formation of stronger and more impermeable CSA. Compared with the non-treated CSA, the particle density, loose bulk density and bulk crushing strength of treated CSA increased by about 13%, 11% and 14%, respectively, while its water absorption decreased by about 27%.

After determining the best effective curing method and the most sufficient surface treatment for CSA, large quantities of treated and non-treated CSA, with size ranges of 2 - 4 mm and 4 - 8 mm, were manufactured and cured at RH 99% in preparation for the next step of this research scheduled to study its practical applications in concrete.

6.5. The behavior of CSA concrete in comparison with ECA concrete

Generally, the effects of aggregate type and content on the mechanical properties of concrete are related to the strength of the cement matrix itself, which is mainly dependent on the cement content used. Therefore, the use of a high cement content helps to evaluate the influence of aggregate quality on the performance of concrete. Joseph and Ramamurthy reported that the strength of concrete made of cold-bonded aggregate and cement content up to 350 kg/m³ is dependent on the strength of the cement matrix. While, at a higher cement content, the concrete failure takes place through the aggregate, and thus, the strength of concrete becomes depended mainly on the content and type of the aggregate used [8]. Hwang and Tran also investigated the effect of aggregate properties on the performance of concrete
6. Results and achievements

contains a binder content of 400 kg/m³ cement and 150 kg/m³ fly ash [9]. Also, concrete mixtures with a cement content ≥ 400 kg/m³ were designed to study the effect of different type of cold-bonded aggregate on the hardened properties of concrete [10,11].

Consequently, in order to evaluate the application of CSA in concrete in comparison with that of expanded clay aggregate, six concrete mixtures divided into two groups have been designed to have a cement content of 405 kg/m³ as shown in section 4.4. Among these two groups, the only difference was the type of fine aggregate; natural quartzite sand (NS) and lightweight expanded clay sand (ECS) were utilized in the mixes of groups 1 and 2, respectively. In each group, except the type of the coarse aggregate used, all the parameters; binder content, w/b ratio, superplasticizer dosage, stabilizer amount, sand content, grading and the volume content of the aggregate were kept constant. The type of aggregate; expanded clay aggregate (ECA), treated and non-treated CSA, is therefore the main influential factor on the concrete properties.

The physical, mechanical and thermal properties of concrete have been evaluated in terms of dry density, compressive strength, modulus of elasticity and thermal conductivity. In general, based on the results of concrete density, all mixes have a dry density < 2000 kg/m³ and can be therefore classified, according to EN 206-1, as lightweight concrete. However, it is clear from the results illustrated in section 4.4 that the use of treated CSA contributed significantly to enhancing the mechanical properties of concrete. In the mixtures composed of normal sand (group 1), the compressive strength of surface-treated CSA concrete was higher than that made of non-treated CSA by about 13 %. Meanwhile, the difference between their dry densities was only 4 %. Whereas, in concretes made of expanded clay sand (group 2), the dry density and compressive strength of concrete produced with treated CSA were higher than that made with non-treated one by about 23 % and 5 %, respectively. This is mainly attributed to the crushing strength of surface-treated CSA, which is higher than that of non-treated. The ratio of compressive strength to dry density of concretes was considered to compare the performance of CSA and ECA concrete. The results show that the concrete made of surface-treated CSA
6. Results and achievements

exhibited higher strength to density ratio than that made of either ECA or non-treated CSA, which showed approximately the same strength to density ratio. It is evident from the results of the modulus of elasticity that concretes produced with treated CSA recorded the highest modulus of elasticity, followed by those made of untreated CSA, and finally those composed of ECA. Based on these observations, it can be concluded that the mechanical characteristics of CSA concrete are superior to that of ECA concrete. In contrast, according to the thermal conductivity results, it has been found that ECA concrete shows superior thermal performance to that made of CSA. This is attributable to the highly porous structure of ECA compared with CSA.

To assess the durability of concrete, capillary water absorption and water penetration tests have been implemented. The water capillarity coefficient of CSA concrete made of untreated aggregate was higher than that of ECA concrete. Interestingly, the use of surface-treated CSA contributed significantly to reducing the water capillarity coefficient to approximately the same value as shown by ECA concrete. Although the ECA concrete and surface-treated CSA concrete exhibited the same water capillarity coefficient, the latter showed better water penetration resistance. This can be explained by the quality of interfacial transition zone between the aggregate and the cement matrix; where the SEM images clearly showed that the ITZ of CSA concrete is denser and more homogeneous than that of ECA concrete.

Furthermore, the complementary experimental study on drying shrinkage showed that the concrete made of ECA having higher drying shrinkage as compared to that of CSA concrete. It was also observed that the drying shrinkage of both kinds of concrete increased significantly when the normal-weight sand was totally replaced by lightweight sand. However, the drying shrinkage in CSA concrete made of normal sand is located in the standard shrinkage range of normal concrete, as specified in EN 1992-1-1.
7. Conclusions

7. Conclusions and recommendations for future research

The main contributions of this thesis can be summarized as follows:

The development of an encapsulation process has been improved to offer an advanced and promising approach to produce cold-bonded lightweight aggregate with a low particle density and good mechanical properties. This approach offers the feasibility of manufacturing different aggregate sizes by controlling the size of the core structure as well as the thickness of the cover matrix. Different particle densities for the manufactured aggregate can also be achieved by changing the composition of the shell structure.

As shown in Figure 7.1, the cold-bonded aggregates produced in this research by the encapsulation technique show a lower particle and bulk density than that produced in the available literature through an agglomeration process, achieving, therefore, the first objective of this study.

Figure 7.1. Comparison of particle and bulk density of CSA and cold-bonded aggregate produced in literature
7. Conclusions

The characteristics of the core-shell structured lightweight aggregate have been improved by determining the appropriate curing and surface treatment methods. Curing at RH 99% and treating the aggregate surface by a cover of cement and silica fume succeeded to enhance the crushing strength of 2-4 mm and 4-8 mm aggregate from 3.54 to 4.20 MPa and from 3.11 to 3.55 MPa, and reduce their water absorption from 43 to 36 wt.-% and from 44 to 32 wt.-%, respectively.

The applicability of CSA in the production of lightweight aggregate concrete, the second goal of this research, has been achieved by producing CSA concrete having a dry density ranged from 1115 to 1540 kg/m³, which can be categorized as D1.2 and D1.6 based on the classification of EN 206-1. Being that the compressive strength of CSA concrete ranges from 17.9 to 29.1 MPa and its thermal conductivity from 0.3169 to 0.666 W/(m·K), it can be emphasized that the CSA manufactured in this study has a sufficient potential to be used for the production of lightweight aggregate concrete for both structural and insulating purposes. Moreover, the results showed, interestingly, that CSA has the capability to be used at an early age in concrete which saves time and storage space and reflects, therefore, positively on production costs.

The experimental results reveal that the concrete produced with core-shell aggregate has a superior mechanical and microstructural performance and inferior thermal properties as well as lower drying shrinkage than that made of expanded clay aggregate, one of the wildly used sintered lightweight aggregate available in the market. The lower thermal conductivity of CSA concrete can be justified by the high energy consumption associated with the production process of sintered aggregate, which has negative economic and environmental consequences.

Finally, the results in this thesis encourage more work on the production of cold-bonded lightweight aggregate by the encapsulation process, which may offer significant economic and environmental benefits. Of course, the type of core, as well as the composition of the cover matrix, are the major influential factors on the cost of encapsulated aggregate. Therefore, future work focusing on the production of core-shell LWA using low-cost raw materials, aiming to reduce the cost of the final product has been planned. This work has been initiated because of the widespread use of lightweight concrete and the urgent need to study its recycling. In this concern, two
7. Conclusions

plans can be performed, in which both the lightweight aggregate concrete and foam concrete are totally recycled into core-shell LWA. A core of recycled foam concrete particle and cover matrix composed of a binary mixture of recycled foam concrete-powder and cement would be used in the production process of the first plan, as shown in Figure 7.2. While in the second plan, a core of recycled lightweight aggregate concrete would be encapsulated into a cover matrix composed of recycled lightweight aggregate concrete powder and cement, as shown in Figure 7.3.

Figure 7.2. Core-shell LWA made of recycled foam concrete; (a) core of foam concrete particle, (b) cross-section of aggregate specimen with a cover matrix composed of recycled foam concrete powder and cement.

Figure 7.3. Core-shell LWA made of recycled lightweight aggregate concrete; (a) core of lightweight aggregate concrete particle, (b) cross-section of aggregate specimen with a cover matrix composed of recycled lightweight aggregate concrete powder and cement.
8. References


8. References


9. Appendix

A list of all papers published during the PhD time as well as the co-authors' contribution statements are given in this appendix

“The production and properties of cold-bonded aggregate and its applications in concrete: A review”

Published in the journal “Construction and Building Materials”

Volume 225, 20 November 2019, Pages 29-43

https://doi.org/10.1016/j.conbuildmat.2019.07.219

Authors: Feras Tajra, Mohamed Abd Elrahman, Dietmar Stephan

<table>
<thead>
<tr>
<th>Co-author</th>
<th>Contribution percentage</th>
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<tbody>
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<td>Feras Tajra</td>
<td>80%</td>
<td>Compiling the literature sources. Preparation of the paper before the first review including figures, tables and graphs. Implementation of changes during the review process, as well as response to the reviewers.</td>
</tr>
<tr>
<td>Mohamed Abd Elrahman</td>
<td>10%</td>
<td>Support in the preparation of final version of the paper</td>
</tr>
<tr>
<td>Dietmar Stephan</td>
<td>10%</td>
<td>Support in the production of final version of the paper. Acted as corresponding author.</td>
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9. Appendix

“Performance assessment of core-shell structured lightweight aggregate produced by cold bonding pelletization process”

Published in the journal “Construction and Building Materials”
Volume 179, 10 August 2018, Pages 220-231
https://doi.org/10.1016/j.conbuildmat.2018.05.237
Authors: Feras Tajra, Mohamed Abd Elrahman, Sang-Yeop Chung, Dietmar Stephan

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<td>Feras Tajra</td>
<td>70%</td>
<td>Conceiving and planning the experiments. Designing and performing experiments as well as analyzing the results. Preparation of the paper before the first review including figures, tables and graphs. Implementation of changes during the review process, as well as response to the reviewers.</td>
</tr>
<tr>
<td>Mohamed Abd Elrahman</td>
<td>10%</td>
<td>Support in planning the experiments and preparation of final version of the paper.</td>
</tr>
<tr>
<td>Sang-Yeop Chung</td>
<td>10%</td>
<td>Performing micro-CT test</td>
</tr>
<tr>
<td>Dietmar Stephan</td>
<td>10%</td>
<td>Supervision in planning the experiments and preparation of final version of the paper. Acted as corresponding author.</td>
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9. Appendix

“An experimental study of using core-shell structured lightweight aggregate in producing lightweight concrete”

Published in International Conference on Sustainable, Environmentally Friendly Construction
Materials, Szczecin, Poland, 24th- 25th May 2018, pages 35-40
Authors: Feras Tajra, Mohamed Abd Elrahman, Dietmar Stephan

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<th>80%</th>
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<th>Corresponding author.</th>
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<td>Mohamed Abd Elrahman</td>
<td>10%</td>
<td>Support in planning the experiments and preparation of final version of the paper.</td>
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<td>Dietmar Stephan</td>
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<td>Supervision in planning the experiments and preparation of final version of the paper.</td>
<td>Co-author 2</td>
</tr>
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"Properties of lightweight concrete made with core-shell structured lightweight aggregate"

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<table>
<thead>
<tr>
<th>First author</th>
<th>Contribution percentage</th>
<th>Type of contribution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feras Tajra</td>
<td>70%</td>
<td>Conceiving and planning the experiments. Designing and performing experiments as well as analyzing the results. Preparation of the paper before the first review including figures, tables and graphs. Implementation of changes during the review process, as well as response to the reviewers.</td>
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<tr>
<td>Mohamed Abd Elrahman</td>
<td>10%</td>
<td>Support in planning the experiments and preparation of final version of the paper.</td>
</tr>
<tr>
<td>Christian Lehmann</td>
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<td>Performing SEM and XRD tests</td>
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<td>Dietmar Stephan</td>
<td>10%</td>
<td>Supervision in planning the experiments and preparation of final version of the paper. Acted as corresponding author.</td>
</tr>
</tbody>
</table>

- I affirm that all information given in this appendix is true.
- The list of contribution was confirmed, signed by the authors, and included in the doctoral file.