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## Production of Cold-Bonding Pelletized Artificial Expired Cement- Fly Ash Lightweight Aggregates with Various Curing Regime

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**Keywords:**

Artificial Lightweight Aggregate; Cold Bonding Pelletization Process; Expired Cement; Fly Ash.

**Highlights:**

- Cold bonding expired cement fly ash aggregates.
- Structural lightweight aggregates.
- Air and water curing.

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**Abstract:** Expired cement (EC) with fly ash can be recycled in artificial lightweight aggregates (ECFLA) manufacturing to manage industrial waste by mixing with fly ash through a cold bonding process, supplying a sustainable material and contributing to circular economy strategies to produce structural lightweight concrete. Four series of (ECFLA) were divided based on curing conditions and foaming agents, including 20 types of (ECFLA) with control mixes; air and water curing were compared, and their effects were investigated on the (ECFLA) properties. The present study identifies the optimum mix combinations. (EC) mixed with fly ash (FA) in varying amounts of 10, 20, 30, 40, and 50 % by weight of EC with and without foaming agent, it contained approximately 22% water by weight. The (ECFLA) was hardened through a cold-bonding air or water curing process for 28 days, followed by testing at different physical and mechanical properties. Particle crushing strength, impact value, specific gravity, and water absorption were tested on the hardened aggregates. The results indicated that it is possible to produce ECFLA from EC in a cold bonding process. The optimum kind of ECFLA was (20EC80FA) mixed with a foaming agent of loose dry density of 862.13 kg/m<sup>3</sup>, and particle crushing strength was 2.29 MPa at 28-day for 12 mm diameter. According to test results, cement content significantly affected (ECFLA) strength, consequently influencing lightweight structural concretes' (LWC) compression strength. The highest 28-day compressive strength of LWCs was 42.3MPa for the (EC50FA50) mix.

## إنتاج ركام من الاسمنت منتهي الصلاحية خفيف الوزن ذات حبيبات مترابطة على البارد مع معالجات مختلفة

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### الخلاصة

يمكن إعادة تدوير الأسمنت منتهي الصلاحية في تصنيع الركام الصناعي خفيف الوزن كطريقة لإدارة المخلفات الصناعية عن طريق الخلط مع الرماد المتطاير من خلال عملية الربط الباردة، وتوفير مادة مستدامة والمساهمة في استراتيجيات الاقتصاد الدائري التي يمكن استخدامها في إنتاج الخرسانة الهيكلية خفيفة الوزن. يتم تقسيم أربع سلاسل من ECFLA على أساس ظروف المعالجة وعوامل الرغوة، بما في ذلك ٢٠ نوعاً من مع خلطات تحكم؛ تمت مقارنة المعالجة بالهواء بدرجة حرارة الغرفة والماء، وتم دراسة تأثيرهما على خواص. تحدد الدراسة الحالية توليفات المزيج الأمثل. تم خلط مع الرماد المتطاير بكميات متفاوتة لـ ٤٠، ٣٠، ٢٠، ١٠، إلى ٥٠٪ بالوزن مع وبدون عامل رغوة، واحتوت على ما يقرب من ٢٢٪ ماء بالوزن. تم تقوية (ECFLA) من خلال عملية المعالجة بالهواء البارد أو بالماء لمدة ٢٨ يوماً تليها الاختبارات على خصائص فيزيائية وميكانيكية مختلفة. تم اختبار مقاومة تكسير الجسيمات وقيمة التأثير والجاذبية النوعية وامتصاص الماء على الركام المتصلب. أشارت النتائج إلى أنه من الممكن إنتاج ECFLA من EC في عملية ربط بارد، وكان النوع الأمثل لـ (ECFLA) هو (20EC80FA) مع عامل رغوة بكثافة جافة سانية تبلغ ٨٦٢،١٣ كجم / م<sup>٣</sup>، وكانت مقاومة تكسير الجسيمات ٢،٢٩ ميجا باسكال. في ٢٨ يوماً بقطر ١٢ ملم. وفقاً لنتائج الاختبار، أثر محتوى الأسمنت بشكل كبير على مقاومة (ECFLA)، مما أثر بالتالي على مقاومة ضغط الخرسانة الهيكلية خفيفة الوزن (LWCs). كانت مقاومة الضغط الأعلى لمدة ٢٨ يوماً من ٤٢،٣ LWCS ميجا باسكال لمزيج (EC50FA50).

**الكلمات الدالة:** الركام الاصطناعي خفيف الوزن، عملية التكوير بالترابطة الباردة، الأسمنت منتهي الصلاحية، الرماد المتطاير.

### 1. INTRODUCTION

Concrete's mechanical and durability properties are greatly affected by the aggregate, which constitutes 60–70% of the volume of concrete. With the development of technology and more complex projects, the need for aggregate demand is rising quickly, while natural aggregate resources are steadily declining. As a result, it is important to discover new sources of aggregate. One approach is to turn waste materials into artificial materials [1]. Environmental and economic factors substantially impacted utilizing waste material and improving performance characteristics. Due to the increasing demand for and interest in recycling refuse products, numerous researchers are interested in producing concrete aggregates from waste materials and fly ash (FA). Typically, FA and a binder, such as expired cement or clay, are combined to form the aggregate particle [2,3]. One ton of cement will emit 900 kg of CO<sub>2</sub> into the air. Cement plants contribute nearly 5-7% of global CO<sub>2</sub> emissions [4]. Without adequate storage, this high energy consumption can turn into expired cement due to bad storage. The expired cement (EC) is unconsumed cement or cement that has not been properly stored and will lose validity if not consumed within the recommended storage period. Cement bags that have been ruptured, the cement that has been stored for a long period, and moisture are the main causes of cement prehydration, which is the interaction of anhydrous cement with water vapor in the air, resulting in partial hydration and carbonation of the cement's surface and change the cement properties [4,5]. The direct effects of EC on the respiratory system can cause serious health problems. It comprises various chemical products with high fineness, which produces various health concerns related to breathing difficulties [6]. Thus, reusing EC provides considerable potential, as it influences the availability of raw materials, energy consumption, and CO<sub>2</sub> emissions from cement manufacture. Using (EC) in the aggregate

manufacturing process is an innovative way to reduce landfill waste. As a result, this process contributes to protecting the environment and decreasing raw material consumption while creating sustainable materials and an innovative approach to circular economies [6]. Two main methods of producing artificial lightweight aggregate pellets exist, i.e., sintering and pelletizing. A well-known disadvantage of the sintering process is that it consumes a lot of energy during pelletization. This process involves bloating pellets at a temperature up to 1200 °C [7,8]. Meanwhile, cold-bonding pelletization has been proposed as a more economical method. Using a wetting agent, usually water, fine materials are agglomerated at ambient temperature to form larger solid particles [1]. Cold-bonded products are rounded and spherical, increasing concrete workability and reducing water-cement ratios. This approach uses the least amount of energy and, therefore, is more cost-effective than the sintering method. LWA is far more ecologically friendly and sustainable, with fewer environmental pollutants. The density and strength of fly ash lightweight aggregates are significantly influenced by mechanical parameters, such as disc or drum speed and angle [2]. The raw material's moisture content and grain size distribution significantly impact aggregate size growth. This approach may be useful for consuming a large volume of by-products and waste materials, such as fly ash, throughout production. Fly ash is formed as a by-product of coal combustion in energy production. At room temperature, fly ash containing expired cement and/or lime is obtained using water as a coagulant [9]. It has been found that cold-bonded LWA can be made using FA with cement, activators, such as sodium silicate, and ground and blast furnace slag. Despite the decreased specific gravity of cold-bonded LWA compared with sintered LWA, it remains significantly higher [10,11]. Hwang et al. [12] analyzed eight types of

aggregates made lightweight using the lightweight aggregate's properties with the foamed agent, fly ash, Portland cement, and chemical foaming of hydrogen peroxide. The results showed that by adding some porous geometries inside the lightweight fly ash aggregate, cold bonding could reduce the specific gravity of LWA. Also, Ibrahim et al. [8] used a foaming agent of protein-based. Thirty-three parts of water were dissolved with one part of the foaming agent, and cement and fly ash with various curing regimes were tested. The result showed that the water curing was stronger than other lightweight aggregates with different curing regimes. The optimum LWA was 20% fly ash with 80% cement. This paper shows novelty and originality for the first time in world research for recycled expired cement in artificial lightweight aggregates manufacturing non-fired aggregate by cold bonding pelletization process as well as examines the physical and mechanical characteristics of the aggregate: strength, specific gravity, density, and water absorption were measured. Hence, the aggregates produced were 20 types of EC and FA with and without foamed agents and treated in air and water for 28 days. The second stage of the LWA is casting to determine the compressive strength.

## 2. MATERIALS AND EXPERIMENTAL PROGRAM

### 2.1. Materials

The new ordinary Portland cement (PC) was used in this study. The chemical and physical specifications of PC and EC agree with the limitation of 5/2019 [13], as shown in Tables 1 and 2. The expired cement (EC) used in manufacturing aggregates, shown in Fig. 1, was in the concrete laboratory, and its expiration date was over three months. A portion of EC was agglomerated and needed to be crushed, while the remaining was a powder that could be directly used. According to Table 2 results and Bogue's equations (Neville) [14] below, the C<sub>3</sub>S, C<sub>2</sub>S, C<sub>3</sub>A, and C<sub>4</sub>AF fractions in EC were calculated. In this study, to present the exact results of the expired cement composition, thermal gravimetric analysis (TG) was performed. From the results in Table 1 and Bogue's equation [14], the fractions of the main

compounds at EC were calculated. The approximate mass of each object of C<sub>3</sub>S, C<sub>2</sub>S, C<sub>3</sub>A, and C<sub>4</sub>AF is shown in Table 3. Calculations based on four minerals reacting with a similar hydrating percentage were conducted. Mass loss results in the average distribution of four mineral ingredients; compared with new cement, EC lost 9.5% of its mineral composition, as indicated in Fig. 2 (a). While the new cement lost 7.5%, as shown in Fig. 2 (b). In contrast to the new cement, the amount of CaCO<sub>3</sub> in EC was four times more than new cement. Since EC does not contain Ca(OH), all the Ca(OH)<sub>2</sub> has carbonated during aging. Additionally, the new cement's peak CaCO<sub>3</sub> was 700°C, while ECs was 724.54°C, perfectly matching poorly crystallized and well-crystallized cement. New cement had hydration products at the first peak in the TG mass curve, which must have occurred during transport. Based on this change, it is possible to explain hydration when cement is in contact with air. Expired cement is considered a type of hydrating cement material. Fly ash was provided by a building chemical company. Since fly ash contains less than 10% CaO, it has a similar composition to ASTM C618 Class F fly ash [15], as shown in Table 1. Cemairin F300 was used as a foaming agent in a liquid state by a chemical company to produce lightweight foam mortar. The technical properties are shown in Table 4. Natural sand aggregate of a specific gravity of 2.65 g/cm<sup>3</sup> was used as fine aggregate for lightweight concrete production. The sieve analysis, fine modulus, and water absorption results are tabulated in Table 5, according to Iraqi Standard No. (45)/1984 [16] zone (2) limit.



Fig. 1 Caked EC, EC Powder, and Fly Ash.

Table 1 Chemical and Physical Characteristics of Portland Expired Cement and Fly Ash.

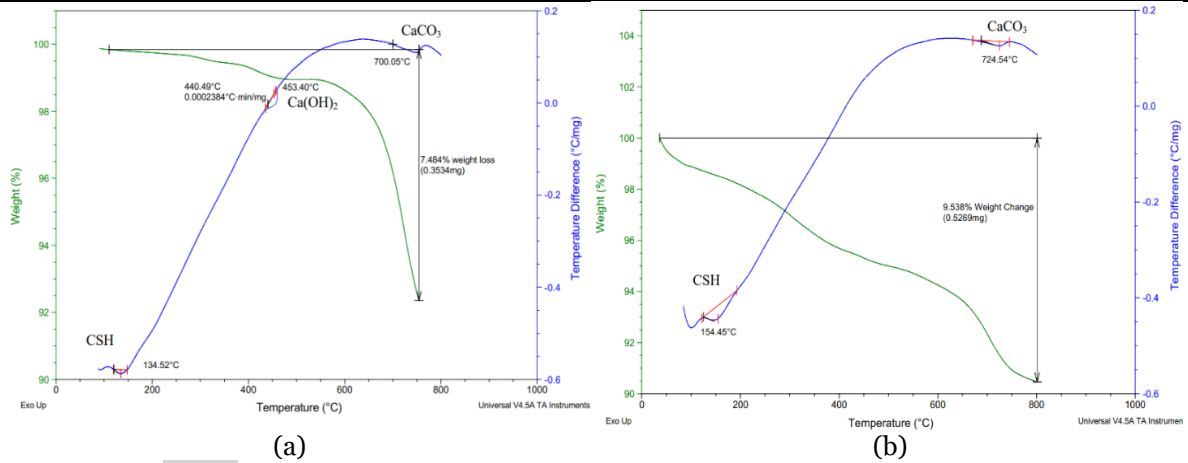
Chemical Composition (%)	EC	New Cement	Limit of Iraqi Specification, No.5/2019 for Cement	FA	Limits of ASTM C 618-08 [15]
CaO	60.51	65.04	----	5.11	--
SiO <sub>2</sub>	20.00	17.11	----	47.67	--
Al <sub>2</sub> O <sub>3</sub>	4.44	3.82	----	27.73	--
Fe <sub>2</sub> O <sub>3</sub>	5.26	2.80	----	18.42	--
SO <sub>3</sub>	2.45	2.75	----	0.34	5
MgO	2.65	1.250	≤5	0.265	--
Na <sub>2</sub> O	0.344	0.357		--	--
K <sub>2</sub> O	0.976	0.841		--	--
Loss on ignition	1.18	2.98	≤ 4	3.71	6
Specific gravity	3.15	3.15		SiO <sub>2</sub> +AL <sub>2</sub> O <sub>3</sub> + FeO <sub>3</sub> =93.82	≥ 70
				2.2	--

**Table 2** Physical Characteristics of New Cement and Portland Expired Cement.

Physical property	New cement Type I	Expired cement	Limit of Iraqi specification No. 5/2019
Specific surface area (Blaine method), m <sup>2</sup> /kg	282	223	230 (Min.)
Setting time (Vicat apparatus), Initial hr.: min	2:05	2:50	00:45 (Min.)
Final hr.: min	4:20	10:30	10:00 (Max.)
Soundness (Autoclave expansion), %	0.13	0.17	0.8 (Max.)
Compressive strength (70.7mm cube), MPa			
2-day	17	8.3	10(Min.)
28-day	34.2	23.2	32.5 (Min.)

**Table 3** The Calculated Minerals Share Effects from Bogue Equations and TG Evaluation of Types of Cement.

Percentage of Mass	C <sub>3</sub> S	C <sub>2</sub> S	C <sub>3</sub> A	C <sub>4</sub> AF	CSH	Ca(OH) <sub>2</sub>	CaCO <sub>3</sub>
New cement	36.87	26.54	8.01	12.45	2.77	1.34	2.43
EC	32.56	24.87	7.3	11.04	3.54	0	8.68

**Fig. 2** Thermal and Gravity Analysis of (a) New and (b) Expired Cement.**Table 4** Technical Properties of Foaming Agent (Cemairin 300).

Form	Specification
color	Pale
Specific gravity	1.02 at 20 °C
Freezing point	0 °C

**Table 5** The Sieve Analysis and Physical Properties of Fine Aggregate.

Sieve size (mm)	Cumulative passing %	Iraqi Standard No. (45)/1984 zone (2) limit
10.0	100	100
4.75	99.6	90-100
2.36	84.4	75-100
1.18	65.6	55-90
0.6	42.8	35-59
0.3	10.5	8-30
0.15	2.4	10 -0
Fineness modulus	2.92	Not available
Absorption %	2.96	Not available
Specific gravity	2.6	Not available

## 2.2. Experimental Program

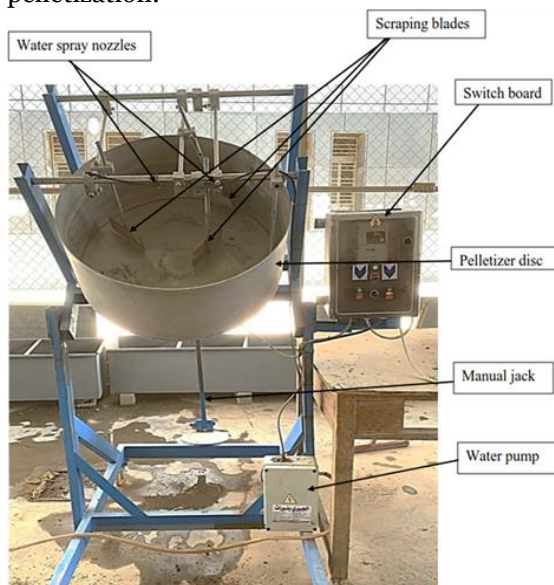
### 2.2.1. Production of Lightweight Aggregates

The first portion of this work involves two series of ECFLA without foaming agents. Each series included five types of ECFLA with EC varied from 10, 20, 30, 40, and 50% and FA, as indicated in Table 6. Series I curing was through the plastic bags in the air, and series II curing in the water for 28-days, as seen in Table 7. Figure 3 shows the instrument of the manufacture of artificial aggregates using the cold bonding method by agglomeration or

pelletization of fine materials, such as expired cement and fly ash, in tilted rotating pans at ambient temperature with a depth of 350 mm and 800 mm of width. According to Baykal and Doven [17], the pace and angle of the dish influenced the ECFLA's strength and specific gravity. Furthermore, Atmaca et al. [2] found that the disc angle and speed affected the water absorption capacity of LWAs. The moisture content of the raw materials and the grain size distribution impacted aggregate size growth [2]. To optimize the Rotational Speed and inclination angle, trials have been performed. A



change combining the inclination angle and rotation speed were tested. Moisture adherence was a factor when observing low revolution speeds at angles more than 45 degrees when particles stuck to the dish side walls during trial operation when the tilt angle was less than 45 degrees with high speed. Reducing the aggregate strength and increasing the spaces inside aggregates that could not pelletize fly ash sufficiently due to large pores within the pellets. To become larger solid particles, wet agents, normally water, are used. Cold-bonded aggregate's rounded and spherical shape reduced its water-cement ratio and made concrete more workable [2,8,18]. The amount of water sprayed is determined based on the speed of rotation and coagulation to form a ball aggregate at the movement of the rolling dish. Sprinkle water on the mixture was 17-22% by water weight. In trial production, pelletization occurred between 10 and 12 minutes, as shown in Fig. 2 (a). To maximize pelletization efficiency, the pan and revolution speed must have been determined to be 22 rpm and 45 rpm, respectively. To produce artificial lightweight aggregates (ECFLA), the powder material was mixed with expired Portland cement during a cold bonding process in 10, 20, 30, 40, and 50% by powder weight, as shown in Table 7. An approximately 5–10 kg mixture of dry powder and Portland cement was fed into the pan and turned continuously to ensure homogeneity for 10 minutes. A rolling pan (disc) was used to determine how much water was sprayed during pelletization.



**Fig. 3** Pelletization Device.

### 2.2.2. Production of Foamed Artificial Lightweight Aggregates

In the second portion of this work, to reduce the ECFLA's density weight and conform with ASTM C 330[19], the foaming agent was used and mixed with potable water through the industrial mixer to produce consistent per-

foaming, as indicated in Fig. 2 (b), creating pores and subsequently producing less dense material. For this purpose, as presented in Table 7, series III of 10, 20, 30, 40, and 50% EC with air curing and series IV with water curing for 28 days were done. Based on prior literary works [16, 17] and from many trials to optimize the dilution ratio, 1:25 was used as the foaming agent dosage, a part of the foaming agent, and 25 parts of water were mixed. Then, the per-foamed was mixed with dry powdered EC and FA to produce lighter aggregates than ECFLA without a foaming agent, as shown in Fig. 2 (c). The curing method was done by air placing the pelletized aggregates inside plastic containers for 28 days and in water for the same duration, as presented in Fig. 2. Then, it was sieved as a fraction from 12.5- 4.75 mm sizes to be utilized as coarse aggregates.



(a)



(b)



(c)

**Fig. 4** (a) New Pellets Toward the Finish of the Pelletization Cycle (b) Foaming Agent Through Industrial Mixer (c) Addition of Foaming Agent to Dry Mixture.



(a)

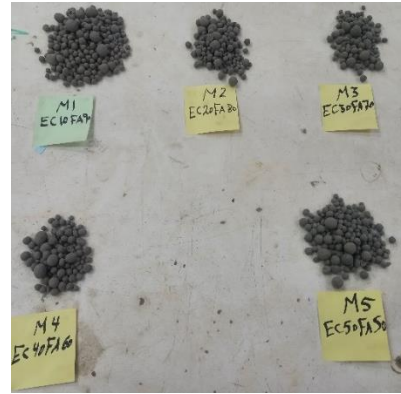


(b)



(c)

**Fig. 5** (a) Air Curing (b) Water Curing (c) Oven Drying.



**Fig.6** Types of (ECFLA).

**Table 6** A Proportional Mix of Materials Produced (ECFLA) at Each Series.

Mix Designation	Expired Cement (EC)%	Fly Ash (FA)%
EC10FA90	10	90
EC20FA80	20	80
EC30FA70	30	70
EC40FA60	40	60
EC50FA50	50	50

**Table 7** Designation for All Mixes with and without Foaming.

Series	Mixes	Percentages	Foaming Agent Water Percent	Curing Condition
Series I	ControlM0	EC0FA100	0%	Air-curing
	M1	EC10FA90	0%	Air-curing
	M2	EC20FA80	0%	Air-curing
	M3	EC30FA70	0%	Air-curing
	M4	EC40FA60	0%	Air-curing
	M5	EC50FA50	0%	Air-curing
Series II	Control		N/A	Water-curing
	M6	EC10FA90	0%	Water-curing
	M7	EC20FA80	0%	Water-curing
	M8	EC30FA70	0%	Water-curing
	M9	EC40FA60	0%	Water-curing
	M10	EC50FA50	0%	Water-curing
Series III	ControlM01	EC0FA100	4%	Air curing
	M11	EC10FA90	4%	Air curing
	M12	EC20FA80	4%	Air curing
	M13	EC30FA70	4%	Air curing
	M14	EC40FA60	4%	Air curing
	M15	EC50FA50	4%	Air curing
Series VI	Control		N/A	Water-curing
	M16	EC10FA90	4%	Water-curing
	M17	EC20FA80	4%	Water-curing
	M18	EC30FA70	4%	Water-curing
	M19	EC40FA60	4%	Water-curing
	M20	EC50FA50	4%	Water-curing

### 2.3. Testing Lightweight Aggregate Grains

ECFLA was sieved, after curing in air and water, into size fractions based on its size varying from 12 mm to 4.76 mm according to ASTM C 330 [19], as shown in Table 8. To determine the properties of lightweight aggregates, tests were performed on their water absorption and specific gravity. The tests were conducted according to ASTM C127 [22]. A standard method for determining aggregate loose bulk density was specified in ASTM 330 [19], with oven-dried density (ODD) of lightweight aggregate must not exceed 1040 kg/m<sup>3</sup>. To remove impurities in the lightweight aggregate, it is necessary to wash it with tap water. Bulk-specific gravity under saturated surface dry conditions (SSD) and apparent specific gravity (ASG) were performed after the oven and saturated surface dry weights were obtained in air and water. During 10, 30, 60, and 120 minutes, 24 hours, and 15 days at room temperature, LWA was submerged in water to determine water absorption capacity. After that, the increase in aggregate weight was measured as the water absorption capacity of the LWAs using the aggregate weight in its saturated-dry surfaced state and the fully oven-dried weight of the aggregates. To determine the ALAs' strength, the ECFLA's impact strength, and particle crushing strength, an impact testing apparatus was used, as shown in Fig. 2. The impact value test was conducted, according to BS812-part 3 [23], as shown in Fig.7 (a). Based on constant impact loading, the aggregate resistance can be approximated using the impact value. Fig. 7 (b) shows the particle crushing strength of individual artificial aggregates tested using the California bearing ratio (CBR) apparatus. This test was conducted to determine the aggregate strength, according to BS-812-112 -1990 [24]. By placing pellets between two corresponding plates and loading them diametrically until failure, the crushing strength of the pellet was determined. Particle failure values were read and recorded on dial gauges as the crushing force P was determined. The average crushing strength of each type of lightweight aggregate was calculated using an average of 10 randomly selected pellets. Utilized a 30 kN capability loading ring, pellets of various sizes, such as 12, 10, 8, and 6 mm, were crushed. According to Eq. 1, the crushing strength of each pellet was calculated using the strength index formula. The sieve analysis of coarse lightweight aggregate is shown in Table 8.

$$\sigma = 2.8 \times P / (\pi \times D^2) \quad (1)$$

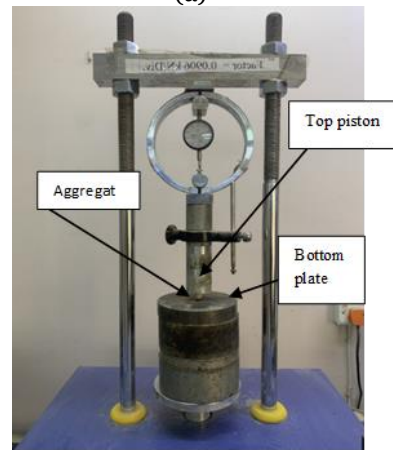
where P is the failure load (N), and D is the diameter of individual pellets (mm) for the sizing.

**Table 8** Sieve Analysis of Coarse Lightweight Aggregate.

Sieve (mm)	Cumulative Passing %	Cumulative Passing % ASTM 330-04
19	100	100
12.5	96	90-100
9.5	56	40-80
4.75	12	0-20
2.36	0	0-10



(a)



(b)

**Fig.7** (a) Impact Value Device (b) Configuration of Particle Crushing Strength Test.

### 2.4. Production of Lightweight Concrete

An experiment was conducted on 20 concrete mixtures to test their compressive strength. According to ACI 211.2-98, the mix design has 450 kg/m<sup>3</sup> of ordinary Portland cement, 0.4 water-cement ratio, and a slump of 110mm. Table 9 shows concrete mixture compositions. Approximately 55% of the lightweight concrete comprised lightweight coarse aggregates, and 45% of the concrete comprised natural fine aggregates. All concrete mixes had the same water-to-cement ratio, cement content, coarse and fine aggregate volume, and superplasticizer content. The only difference was the type of lightweight coarse aggregate. It was possible to achieve a slump of 150 mm for 20 concrete mixes using appropriate superplasticizers during mixing. Due to LWAs' high water



absorption capacity, lightweight concrete production requires special procedures. For this reason, LWAs were soaked for 30 minutes before mixing to maintain saturation, while the aggregates were kept for 30 seconds after mixing to allow excess surface water to drain away [1]. Mixing took about 4 minutes in total. A lightweight aggregate mixture was mixed with Portland cement, then fine aggregates

were added. The mixture was then gradually diluted with water containing a superplasticizer of polycarboxylates-type-based polymer of a specific weight of 1.095 conformed with ASTM C 494 [25]. After the concrete mixture was mixed and poured into the molds, it was layered in two layers and vibrated according to ASTM C129-15.

**Table 9** Proportion of Mixtures for 1 m<sup>3</sup> of Concrete (kg/m<sup>3</sup>).

Mixes	Designation of Mixes	Cement Content (kg/m <sup>3</sup> )	w/c	Water	LWA	Sand	SP	Fresh Density (kg/m <sup>3</sup> )	Dry Density at 28 Days (kg/m <sup>3</sup> )
Control(M0)	EC0FA100	450	0.4	180	655.2	763	2	2052	1987
M1	EC10FA90	450	0.4	180	691.2	763	2	2088	2024
M2	EC20FA80	450	0.4	180	695.1	763	2	2092	2033
M3	EC30FA70	450	0.4	180	734.4	763	2	2131	2058
M4	EC40FA60	450	0.4	180	730.8	763	2	2128	2075
M5	EC50FA50	450	0.4	180	752.4	763	2	2149	2085
M6	EC10FA90	450	0.4	180	637.2	763	2	2036	1995
M7	EC20FA80	450	0.4	180	698.3	763	2	2098	2035
M8	EC30FA70	450	0.4	180	730.2	763	2	2129	2066
M9	EC40FA60	450	0.4	180	712.4	763	2	2108	2073
M10	EC50FA50	450	0.4	180	752.4	763	2	2146	2103
Control(M01)	EC0FA100	450	0.4	180	556.2	763	2	1951	1887
M11	EC10FA90	450	0.4	180	558.0	763	2	1953	1894
M12	EC20FA80	450	0.4	180	558.0	763	2	1956	1897
M13	EC30FA70	450	0.4	180	640.8	763	2	2038	1947
M14	EC40FA60	450	0.4	180	648.0	763	2	2047	1966
M15	EC50FA50	450	0.4	180	655.2	763	2	2053	1971
M16	EC10FA90	450	0.4	180	604.8	763	2	2001	1901
M17	EC20FA80	450	0.4	180	601.2	763	2	1994	1934
M18	EC30FA70	450	0.4	180	633.6	763	2	2025	1944
M19	EC40FA60	450	0.4	180	651.6	763	2	2043	1972
M20	EC50FA50	450	0.4	180	676.8	763	2	2075	2003

### 2.5.Compression Strength Test for Lightweight Concrete

To measure the concrete's compressive strength, three steel cubes of 100×100×100mm were cast. Demolding the specimens after 24 hours was followed by curing in water until day 28 before testing per B.S 1881- part 116 [26]: 89. An average of the three test samples was used to determine the compressive strength.

## 3.RESULTS AND DISCUSSION

### 3.1.Specific Gravity and Bulk Density of Lightweight Aggregates

As defined by the specific gravity of aggregates, it is the ratio between solid and water mass in a given volume. When an aggregate's unit weight is less than 880 kg/m<sup>3</sup>, it is considered a lightweight aggregate according to ASTM C330 [19]. As well as all types of aggregate having an OD-specific gravity of less than 2, the aggregate manufactured for this study was classified as LWA and complied with the N-13055-1 specification [27]. Table 8 shows the influences of EC, foaming agent, and curing condition on ALA's apparent specific gravities and bulk and saturated surface dry. An increment in the percentage of expired cement from 10 to 50% increased the specific weight of all types of (ECFLA), due to the greater specific gravity of cement, and increased in hydration products that act to close the voids in the microstructure

of aggregates and increased the density [28]. As indicated in Table 10, the specific gravity of Series I (ECFLA) (OD specific gravity was 1.53–1.83). Geetha and Ramamurthy [28] and Gesoglu et al. [29] combined the fly ash with cement, resulting in aggregates with a bulk specific gravity of 1.55 to 2.12. The variation in specific gravity may be attributed to the variation in additional binder material, dosage, and testing conditions. The curing effect is very clear in Series I and Series II, in which curing in water was higher in the specific gravity of (ECFLA) than curing in the air because the water increased the EC's hydration and the FA's pozzolanic reaction with EC, reducing open and total porosity of aggregate [8,30]. For instance, the specific gravity of M5 and M10 of the same materials ratio was 1.53 increased to 1.92. Using the foaming agent as a liquid solution reduced the specific gravity of (ECFLA) resulting from forming porous structures by the foaming agent, as compared between Series II and Series III. The curing's effects on the foaming agent are shown in Series III and Series IV [2,8]. Table 8 shows the results for dry loose bulk density of artificial aggregates for all types of ALAs varied between (842.3 to 1060.7) Kg/m<sup>3</sup> as compared to 1600 Kg/m<sup>3</sup> of normal weight aggregate (crushed granite) due to the low specific gravity of raw materials and pores



structure of aggregate particles [31]. EC binder with 50% bulk density was observed to have a high density of 1060 kg/m<sup>3</sup> without foaming and a low bulk density of 10% at 932.3 kg/m<sup>3</sup>. On the other hand, increasing the FA reduced the bulk density. Similar results have been reported by Job Thomas et al. All the cold-bonded lightweight aggregates manufactured satisfied the loose bulk density values, according to code ASTM C 330 [19], which was not more than 880 kg/m<sup>3</sup>. According to the test results, it was determined that the bulk density raised as the EC content increased. The findings demonstrated that greater pore structure caused maximum bulk density; however, pelletization caused less water absorption. Chi et al. [32] and Perumal et al. [33] reported that the bulk density of artificial aggregates was between 972 kg/m<sup>3</sup> to 1247 kg/m<sup>3</sup>, utilizing fly ash with ordinary cement as binder, not expired cement. The corresponding highest bulk density, obtained in the present study for EC aggregate, was 1061 kg/m<sup>3</sup> for the M10 mix of (50EC:50FA) at water curing, as indicated in Table 10.

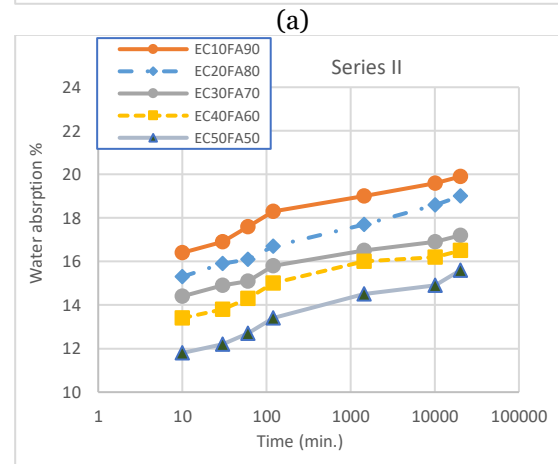
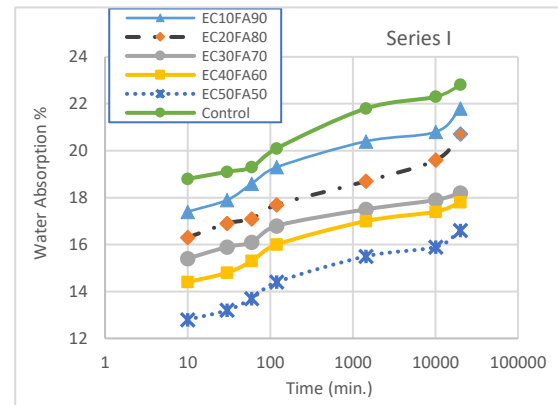
**Table 10** Results of Specific Gravity and Bulk Density.

Series	Mixes	Specific Gravity (g/cm <sup>3</sup> )			Bulk density (kg/m <sup>3</sup> )
		OSD	SSD	ASD	
Series I	M0	1.53	1.86	2.20	912.3
	M1	1.68	1.92	2.23	932.3
	M2	1.70	1.93	2.20	951.5
	M3	1.73	2.04	2.50	1025.2
	M4	1.75	2.03	2.44	1033.6
Series II	M5	1.83	2.09	2.47	1040.7
	M6	1.49	1.77	2.08	949.0
	M7	1.68	1.94	2.26	956.2
	M8	1.76	2.03	2.40	1019.3
	M9	1.74	1.98	2.28	1031.8
Series III	M10	1.92	2.09	2.31	1061.0
	M00	1.35	1.45	1.63	813.3
	M11	1.43	1.55	1.67	842.3
	M12	1.36	1.62	1.69	865.1
	M13	1.54	1.78	2.03	904.5
Series VI	M14	1.67	1.80	1.92	917.2
	M15	1.66	1.82	1.97	1012.7
	M16	1.46	1.68	1.87	847.1
	M17	1.42	1.67	1.88	853.5
	M18	1.53	1.76	1.98	917.2
	M19	1.61	1.88	2.03	936.3
	M20	1.70	1.88	2.08	1030.8

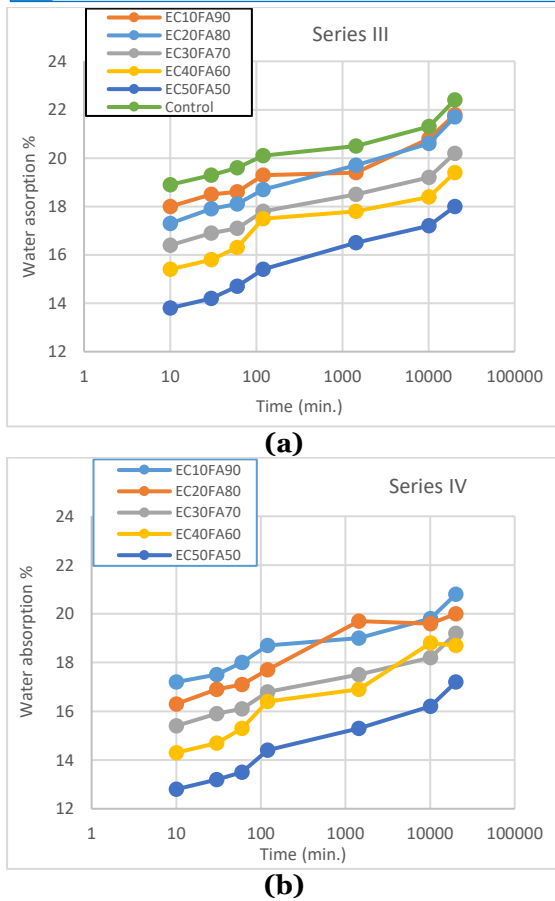
### 3.2. Water Absorption of (ECFLA)

A negative trend was observed in water absorption compared with specific gravity and unit weight. The water absorption of all types of ECFLA increased with time, regardless of the type and proportion of materials and curing method, due to the high porosity, as shown in Figs. 8 and 9. Water absorption was determined at 10, 20, 30, 60, 120 min, 24 hours, and 14 days. Furthermore, about 80% of the water absorption happened at 30 minutes, agreeing with Gesoglu et al. [34]. Therefore, it was decided to saturate the lightweight aggregate with water for 30 minutes before producing lightweight concrete. The water absorption

decreased by increasing the amount of expired cement in the mixture because a higher expired cement content involved more hydration reaction due to the high calcium silicate hydrate content, causing the structure to be denser microscopically. Additionally, the pozzolanic reaction between calcium hydroxide and fly ash with water resulted in more C-S-H, which increased the ECFLA structure's density [8,12,35], as shown in Fig.8 (a),(b). Similar results were reported by Atmaca et al. [9]. It is especially pronounced with higher expired cement contents of 50% as in M5, M10, M15, and M20, with less water absorption of all lightweight aggregates. The effects of the foaming agent were clear in Series III and Series IV compared to the rest of the results. These effects presented that the absorption of foamed lightweight aggregates ECFLA increased with adding the foaming agent, as shown in Fig.9 (a) and (b), increasing air spaces in the ECFLA's microstructure [20,21]. Also, the results showed that the curing in the water reduced the absorption capacity compared to the air curing, increasing the hydration process, closing the capillary pores, and reducing the permeability of aggregates, and thus the aggregate porosity. This result agrees with Hwang and Tran [20] and, Manikandan and Ramamurthy [36].



**Fig. 8** (a) Water Absorption of Series I with Air Curing (b) Water Absorption of Series II with Water Curing.

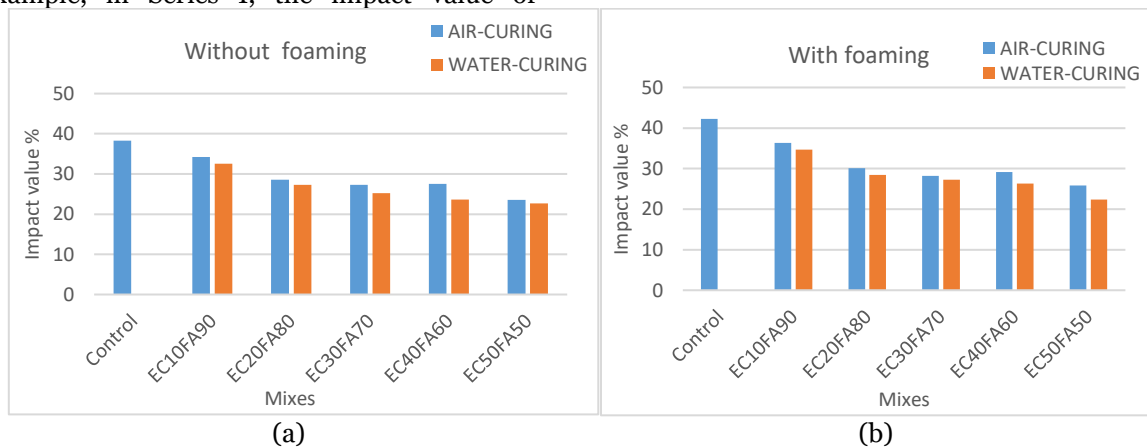


**Fig.9** (a) Water Absorption of Series II with Air Curing (b) Water Absorption of Series VI with Water Curing.

### 3.3. Particle Crushing Strength and Impact Value

The impact test value and particle crushing strength determined the potency of ECFLA in this material. According to Table 11, no manufactured aggregate with an impact strength greater than 30%, as measured by weight and meeting the structural specifications of BS 812-112:1990 [24], was found in this study. Furthermore, it was noted that impact strength depended on the amount of fly ash added during pelletization, the type of curing that improves the microstructure and properties, and the foaming agent. For example, in Series I, the impact value of

aggregate decreased directly with the percentage of fly ash; 90% diminished until 50% of fly ash decreased from 28.61% for (EC90FA10) to 17.5% for (EC50FA50). According to Ibrahim [8] research, aggregate with an AIV greater than 30% cannot produce concrete and was regarded as a very inferior aggregate. The AIV of ECFLA with water curing was stronger than with air, as shown in Series II due to forming discontinuous pores at the matrix-aggregate interface and improving the matrix microstructure. The AIV reduction was 11.5% for (EC90FA10) and 12.5% for (EC50FA50). The impact value of all types of ECFLA increased with using the foaming agent as compared to types of aggregate without foaming agent [37] due to the increase in the pores in the ECFLA's microstructure with foaming agent clearer in series III and VI as compared with series I and II, indicated in Fig.10. Numerous factors affect particle crushing strength of aggregates, including specific gravity, shape, absorption of water, and pore size distribution [20]. Table 11 indicates the particle crushing strength of the (ECFLA) from 6mm to 12mm, compared to small aggregates (6mm) provide the best strength. Also, the results showed that the particle crushing strength of ECFLA increased with adding EC from 10, 20, 30, 40, and 50%, as shown in Series I. The particle crushing strength of water curing is higher than the ECFLA's at air curing, such as Series II, because the water inside the ECFLA was sufficient for improving the hydration products of EC [8]. According to the test results, the crushing strength value for ECFLA agreed with the specific gravity and the water absorption values [35]. ECFLA with larger specific gravity with the least water absorption were stronger. Also, Table 9 demonstrates the foaming agent's effects on the ECFLA's strength. Series III was weaker than Series I due to the air spaces formed inside the ECFLA's microstructure by the foaming agent. The ECFLA became stronger with water curing than air curing, which was clear in Series III and Series IV [16, 17, 24].



**Fig. 10** (a) Impact Value without Foaming (b) Impact Value with Foaming.

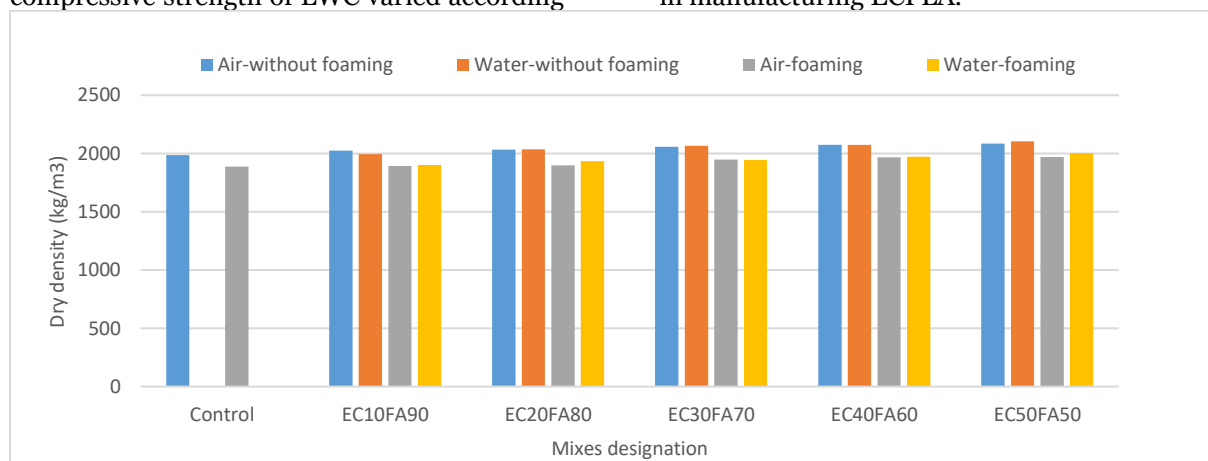
**Table 11** Particle Crushing Strength of All Types of ECFLA.

Series	Mixes	Particle crushing strength (MPa)			
		Diameter mm			
		6mm	8mm	10mm	12mm
Series I	M0	0.74	0.63	0.37	0.40
	M1	2.72	2.65	2.50	1.92
	M2	3.47	3.34	3.30	2.66
	M3	4.71	4.60	3.83	3.16
	M4	5.70	5.29	4.55	4.21
	M5	7.43	6.83	6.06	4.83
Series II	M6	3.22	3.41	2.59	2.23
	M7	4.46	5.16	3.75	3.03
	M8	5.45	5.02	4.19	3.34
	M9	7.68	5.99	4.01	3.41
	M10	8.17	7.25	6.24	5.02
Series III	M00	0.25	0.42	0.36	0.31
	M11	1.98	2.51	1.78	1.67
	M12	2.97	3.48	2.59	2.29
	M13	3.47	3.07	2.59	2.48
	M14	4.46	4.18	3.66	3.34
	M15	6.19	4.88	4.46	3.59
Series VI	M16	2.48	2.93	2.23	1.80
	M17	3.47	3.62	3.21	2.60
	M18	3.72	4.18	3.66	2.97
	M19	4.95	4.74	3.48	3.10
	M20	6.94	5.16	4.64	3.78

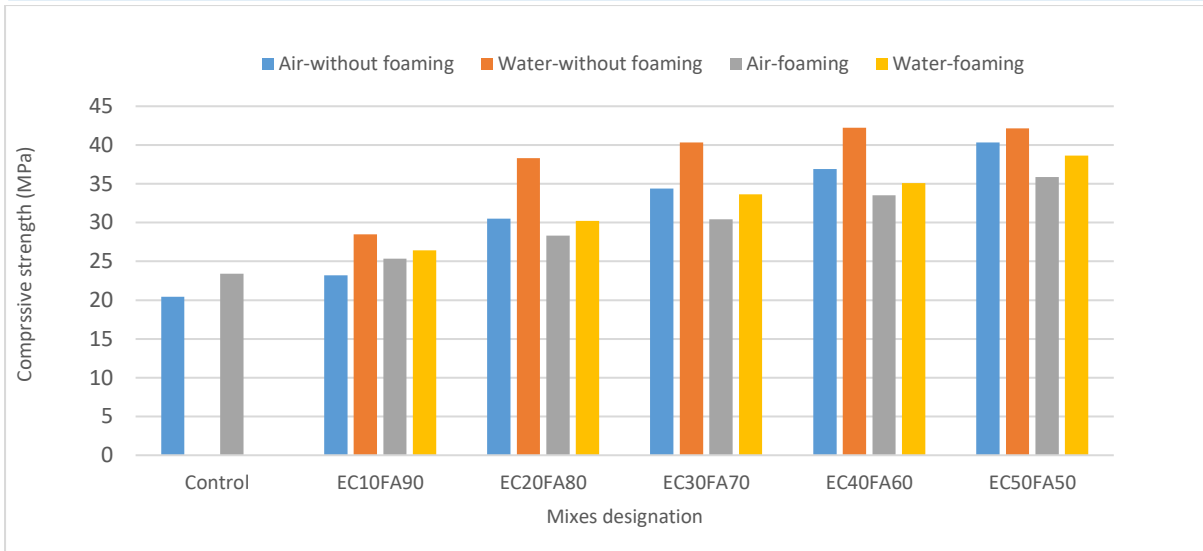
### 3.4. Compressive Strength of Lightweight Concrete

Figure 11 shows the results of dry density for all mixes of LWC. The dry density was reduced with the ECFLA's density. The compressive strength of Lightweight concrete LWC appeared to be influenced more by expired cement content in (ECFLA). The compressive strength of Lightweight concrete LWC increased with the expired cement content within (ECFLA). Since (ECFLA) had higher crushing strength and lower water absorption capacity as expired cement content increased, their compressive strength also increased. A compressive strength test was performed after 28 days, as shown in Fig. 12. LWC's strength cannot be characterized by the density of ECFLA alone because the same density does not produce the same strength for LWC [27]. LWC's strength was significantly affected by the ECFLA's porosity. As a result, it can be concluded that water/cement ratios and cement dosages were constant. The compressive strength of LWC varied according

to its specific gravity, porosity, and crushing strength of ECFLA. These results are corroborated by data given by Güneysi [38] and, Hwang and Tran [20]. Although the SSD-specific gravity of M11 and M12 or M19 and M20 are similar, these aggregates produced concrete with significantly different compressive strengths. In concrete containing M11 ECFLA, the compressive strength was 30.34 MPa at 28 days, while in concrete containing M12 ECFLA, it was 32.33 MPa. Similarly, the compressive strength of concrete with M19 and M20 LWAs was 33.67 MPa and 34.65 MPa, respectively. According to this finding, the compressive strength of lightweight concrete was controlled by the lightweight aggregates crushing strength. By increasing the cement content in the ECFLA production, LWC had a higher compressive strength since ECFLA had higher crushing strengths. These results agree with other research [21,39,40]; however, the compressive strength of this study was low due to using expired cement and foaming agent in manufacturing ECFLA.

**Fig.11** The Dry Density of LWCs with Different Mixes.

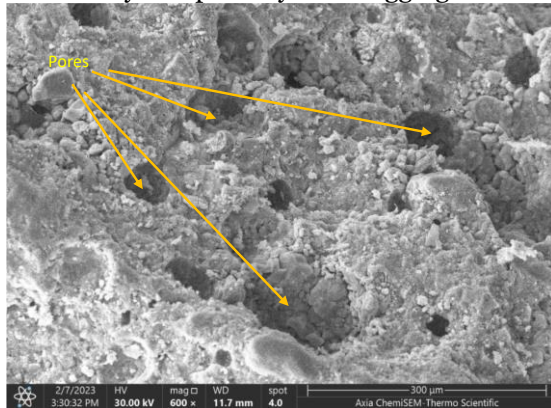




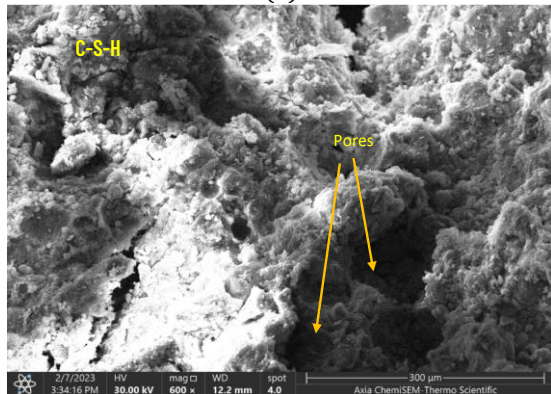
**Fig.12** The Compressive Strength of LWCs with Different Mixes.

### 3.5. Scanning Electron Microscopy (SEM) Analysis

Figure 13 (a) and (b) shows that the EC10F90 mix of air curing was less dense and had more hole structure than the EC10F90 mix at water curing due to the more hydration of EC with water, increasing calcium silicate hydrate gel C-S-H that filled the pores in the microstructure of ECFLA. This result supports increasing the specific gravity density and reducing the absorbency and porosity of the aggregates.



(a)



(b)

**Fig. 13** SEM Image of Microstructure for EC10F90A Mix (a) Air Curing and (b) with Water Curing without Foaming Agent at Magnification (600X).

### 4. CONCLUSION

The main conclusions of the present study could be summarized as follows:

- Producing artificial aggregates can be successfully completed by mixing expired cement and fly ash with a cold bonding method. This technology may contribute to reducing waste and saving energy. Besides possessing good engineering properties, the products were also reusable in concrete. It may be an alternative to ordinary concrete, more environmentally friendly, and suitable for most conditions.
- To achieve maximum pelletization efficiency, the optimum rotation speed and inclination angle were 15 rpm and 45 degrees for a device with dimensions of 800mm and a depth of 350mm.
- Using a binder of expired cement in ECFLA production reduced the water absorption and increased the ECFLA specific gravity. Also, increasing the content of expired cement increased the ECFLA crushing strength.
- EC20F80 can be selected as the optimum ECFLA among the specimens because it had the least AIV, i.e., 13.9%. As the AIV gained, it can be classified as a strong aggregate. The water absorption value of this product was also within the range of the water absorption test criterion from 5 to 25%, and fewer specimens at 20% water absorption value. Furthermore, its density, i.e., 740 kg/m<sup>3</sup>, classified it as LWA.
- Because of the foaming agent employed in this study, the specific gravity of ECFLA decreased due to the increase in pores and microstructures in the internal aggregate.

- In lightweight concrete, compressive strength was determined by the crushing strength and specific gravity and water absorption of ECFLA. With increasing expired cement content in ECFLA production, LWC had higher compressive strengths since ECFLA had higher crushing strengths. Furthermore, the internal curing of ECFLA with high porosity was demonstrated to impact the LWC's compressive strength considerably.

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