



Types of Failure in Cellular Concrete Blocks (Thermostone)

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ABSTRACT:

Cellular or aerated autoclaved concrete blocks (called Thermostone in Iraq) may undergoes several problems during and after manufacturing ,these problems cost both time and money.

The present work aims to determine the main types and causes of failure occurred in cellular concrete blocks (Thermostone) during and after production process. Physical and mechanical tests, chemical analysis, as well as X-ray diffraction technique were conducted to investigate the main causes of failures and to suggest solutions.

The results showed that the complete reaction (Hydrogen bubbles formation) between lime (CaO) , water ,and Aluminum powder always occurs when the minimum reaction temperature is 43 °C or more, No observed reaction took place when the hydration temperature was less than 40 °C . Aluminum powder diameter of (50-100) µm was found to be suitable for best product properties.

X- Ray diffraction results showed a presence of detrimental components causing moisture spots and excessive expansion in both green and autoclaved products. Controlling raw materials, increasing autoclaving period from 10 to 14 hours or adding up to 5% limestone aggregate are essential to get a product fulfills the specifications requirements.

الخلاصة

تعاني كتل الخرسانة الخلوية (والتي تسمى في العراق بالثرمستون) عدة مشاكل خلال وبعد الانتاج مما يكلف الكثير من الوقت والمال. يهدف البحث الحالي الى ايجاد أنواع وأسباب الفشل الرئيسية التي تحدث في هذه الكتل واقتراح الحلول من خلال اجراء الفحوصات الفيزيائية والميكانيكية ، التحليل الكيماوي ، وطريقة انحراف الأشعة السينية. وقد بينت النتائج ان التفاعل بين النورة والماء ومسحوق الالمنيوم (والذي ينتج عنه تولد فقاعات الهيدروجين) يكون تفاعلا كاملا عندما تبلغ درجة حرارته 43 درجة مئوية او اكثر. كما أظهرت النتائج ان قطر حبيبات الالمنيوم المناسب لاعطاء افضل خواص للمنتج يتراوح بين (50 – 100) ميكروميتر. ومن خلال تقنية انحراف الأشعة السينية فقد ظهر وجود مركبات ضارة في المواد الاولية تؤدي الى فشل على شكل بقع رطوبة او تمدد مفرط في حالتي المنتج الطرية والمتصلبة ، لذلك فان السيطرة على المواد الأولية وزيادة فترة الانضاج بالاتوكليف من 10 الى 14 ساعة أو اضافة ركام حجر الكلس كانت ضرورة اساسية للحصول على منتج مطابق للمواصفات المطلوبة .

1. Introduction:

The main advantage of manufacturing cellular or autoclaved aerated concrete (called Thermostone blocks in Iraq) is to get high thermal insulating material with suitable density and compressive strength to be used as a lightweight units in masonry works. Unlike many other concrete products, Thermostone blocks could be drilled, sawed, nailed, or screwed easily by using conventional carpentry tools .On other hand, cellular concrete blocks may undergoes several problems during and after manufacturing ,these problems cost both time and money. The cost of failure for manufacturing companies includes the cost of raw materials, the cost of manufacturing (fuel, machines , workers costetc.) and the time lost in producing failed blocks.

The main purpose of this work is to determine the main types and causes of failure occurred in cellular concrete blocks (Thermostone) during and after production process. Physical and mechanical tests as well as X-ray diffraction technique were conducted to investigate the main causes of failures and to suggest solutions.

The common raw materials used to manufacturing Thermostone blocks are Portland cement, hydrated lime (calcium oxide), aluminum fine powder, water, and sand. The raw materials are mixed into a slurry by a special machine and then poured into greased molds (600 x 120 x 60 cm). Aluminum reacts with the hydrated lime and free water to evolve millions of tiny hydrogen gas bubbles⁽¹⁾. These macroscopic, unconnected cells cause the material to expand to nearly twice its original volume with a cellular structure. It takes from 30 minutes to 4 hours for the mixture to harden enough to be cut by wires into the desired shapes and transported to an autoclave (180°C and 10 atmosphere pressure) for curing period of 10 hours.

The present work investigated the main problems occurred in the factory of Najaf Company for Insulating Materials Industry as a case study. These problems could be classified as follows:

- 1-The cake failure due to no pores formation after mixing the raw materials.
- 2-The cake failure due to hydrogen bubble escaping after formation .
- 3- The product failure due to excessive volume expansion during hydrogen bubble formation when the product is still green and not hardened or dried .
- 4- Brittleness due to residual moisture in the cake after drying .
- 5-The failure of blocks after 10 hours curing in the Autoclave due to excessive expansion or low compressive strength .

Abdulameer and Muhammed ⁽²⁾ used experimental-theoretical method to determine the mechanism of fracture, depending on taking two images. The first one was a stereo microscope with suitable

magnification, so that all different phases can be seen as possible ,the second one was a video image to show pores and cracks. A numerical solution (finite element method) was applied on the images. They found that the existence of pores in Thermostone structure is the source of micro and macrocracks which in turn reduce the resistance to fracture under loads, or causing damage to the product during manufacturing processes .

Abdulameer et. al. ⁽³⁾ investigated the effect of pores characteristics (type of pores - closed and open - and fineness of the added aluminum powder) on each of thermal conductivity and compressive strength of cellular concrete blocks (Thermostone).They applied a numerical technique (Finite Element) to estimate the internal stresses and to get a theoretical cracks pattern. The results showed that open pores blocks exhibits higher thermal insulation but lower compressive strength than closed pores blocks. Finer aluminum powder resulted in higher thermal insulation and lower compressive strength for the two types of pores. 70% of the theoretical crack pattern seemed to be coincide with the actual crack pattern.

Dariusz, J. ⁽⁴⁾ used numerical and experimental methods to measure thermal properties, especially thermal conductivity of cellular concrete, he found that the most important factor is the initial moisture content of the test specimens.

Meille,S. et. al. ⁽⁵⁾ used a general purpose elastic finite element technique designed for use on images of random porous composite materials to study the linear elastic and thermal properties of cellular concrete models. They showed that young's modules, Poisson's ratio and thermal conductivity depend on each of porosity and properties of individual solid phases.

Justness, H. et. al. ⁽⁶⁾ studied pores in concrete and their effect on compressive strength, they also added accelerator materials through the process of production to increase compressive strength and to reduce cracking.

2. Materials and Methods:

2.1. Materials:

The proportions of raw materials usually used to manufacture the Thermostone cake are shown in Table-1.

Table - 1: proportions of raw materials

Component	Proportion (% by wt.)
Sand	25.3%
Lime (Calcium Oxide)	7%
Ordinary Portland Cement	25.2%
Aluminum (Powder)	0.04%
Water	40.38
Clay (as impurities)	Less than 2%

The chemical analysis of the above raw materials is shown in table - 2.

Table- 2 : Chemical analysis of raw materials.

Chemical analysis (% by weight)	Portland Cement	Lime	Sand
Loss on Ignition %	0.69	0.78	2.54
CaO	61.52	90.12	3.42
Al ₂ O ₃	6.02	2.04	3.74
SiO ₂	20.51	4.09	84.47
MgO	4.66	1.92	1.14
Fe ₂ O ₃	3.34	0.45	0.92
SO ₃	2.74	0.65	2.95

2.2.Methods:

In an attempt to solve the problems mentioned previously, Thermostone blocks (20 X 20 X 60 cm) with different ratios of additives were made and tested. Each result represents the average of three tests and the following tests were conducted during the present work:

2.2.1. Compressive strength test: (According to ASTM C 1386)

2.2.2.Dry bulk density test: (According to ASTM C 1386)

2.2.3. Apparent porosity test: (According to ASTM C-830)

2.2.4. Thermal conductivity test: (According to ASTM C518)

2.2.5. Capillary absorption test: According to Iraqi specification for cellular concrete blocks (Thermostone) No.1441/2000), samples were put vertically in a water bath for 24 hours and the level of water ascendance was measured.

2.2.6. Measuring the reaction temperature:

In order to investigate the problem of no pores formation or weak reaction between lime and Aluminum powder a special device (Fig.1) was used to measure the reaction temperature after adding Aluminum. It mainly consists of a mixing motor and a thermometer. Aluminum powder is added after mixing with small amounts of sand to prevent the powder from floating on the surface of the mixer, the mixing continues up to 2 minutes, and the whole test time ends within 3 hours .



Fig. 1: Measuring the temperature of reaction of lime and aluminum.

2.2.7. X-Ray diffraction technique:

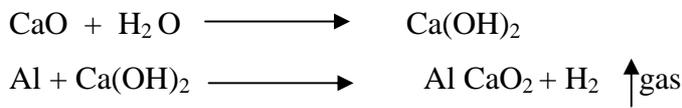
X-ray (powder) diffraction technique and chemical analysis were carried out on raw materials and Thermostone blocks after autoclaving to determine the phases formed during curing. Analysis of the samples (powder method) was carried out by XRD with diffractometer DRON-7 (the wave length of radiation $\lambda=17902$ nm).

3.Results and Discussion:

The aim of the present work is to solve some of the problems associated with the production of Thermostone blocks so the results will be reported according to the problems stated in the introduction:

Problem No. 1: No pores formation:

The pores formation in cellular concrete is the result of complete chemical reaction between lime (CaO) , water ,and aluminum powder due to the liberation of very large number of hydrogen bubbles according to the following equations.



The associated temperature is an indication of completion of this reaction. Fig.2 is plotted to show the required ratio of aluminum powder to get complete reaction (to get 40-60% porosity) according to the reaction temperature.

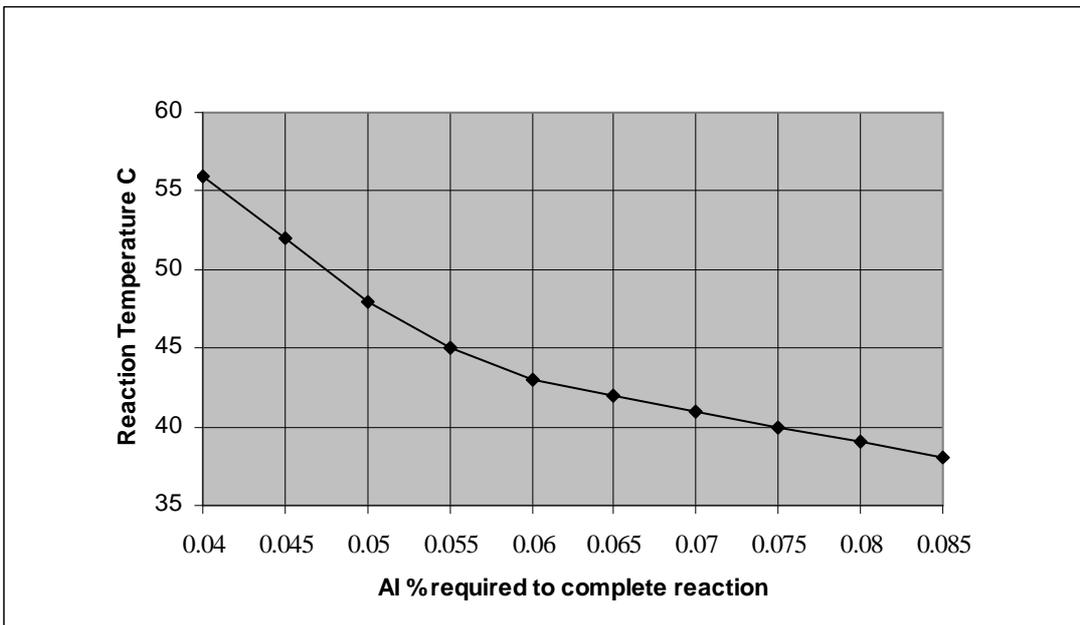


Fig.2 : Relationship between temperature and the added ratio of Al-Powder required to get complete reaction.

During these tests ,it was found that the complete reaction (Hydrogen bubbles formation) between the lime (CaO) , water ,and Al-powder always occurs when the minimum reaction

temperature is 43 °C or more, No observed reaction took place when the hydration temperature was less than 40 °C.

It can be seen from this figure that more Aluminum powder is needed to get the required temperature. For example, 0.075% Al powder was needed to get complete reaction when the temperature was 40°C while 0.04% was needed when the temperature was 55°C. However, Narayanan ⁽⁷⁾ suggested addition of an alkali component like NaOH to get complete reaction when the temperature is within the critical limit.

Problem No. 2: The cake failure due to hydrogen bubble escaping.

Hydrogen bubble escaping indeed is a result of large bubble size which affect thermal and mechanical properties. Table- 3 shows the results of thermal and mechanical tests for samples made with different diameters of Aluminum powder for the same ratio of powder (0.04%).

Table- 3: The effect of Aluminum powder diameter on thermal and mechanical properties

Sample No.	Aluminum powder diameter (µm)	Density(ρ) kg/m ³	Porosity ratio%	Thermal Conductivity (W/m. ° C)	Compressive Strength (MPa)
1	Without Aluminum powder	1250	<6	2.81	5.5
2	200	709	43.28	0.74	2
3	150	652	47.84	0.21	2.1
4	100	601	51.92	0.14	2.5
5	75	540	56.80	0.09	2.7
6	50	538	56.96	0.09	2.9
7	25	512	60	0.09	2.5

Table-3 shows that the density of the product decreases as the radius of the added Al-powder decreases for the same percentage of aluminum. Large diameters of Al-powder generates larger bubbles of hydrogen gas, and some of these bubbles can escape out of the mix and this make the mix more dense . Also it is shown that thermal conductivity (k) decreases with increasing porosity (i.e. the lower the density the lower the thermal conductivity and the higher is the thermal insulation).

It can be seen also that compressive strength increases with lower diameter of aluminum powder and this may be attributed to better distribution of bubbles within the Thermostone block.

Table-3 shows that the suitable diameter of Aluminum powder is in the range [50-100 μm] to get both perfect thermal and mechanical properties which are consistent with ASTM C-1386 , ASTM C518 and Iraqi standard specifications No. 1441/2000.

The capillary absorption test results for the seven samples are shown in Fig.3

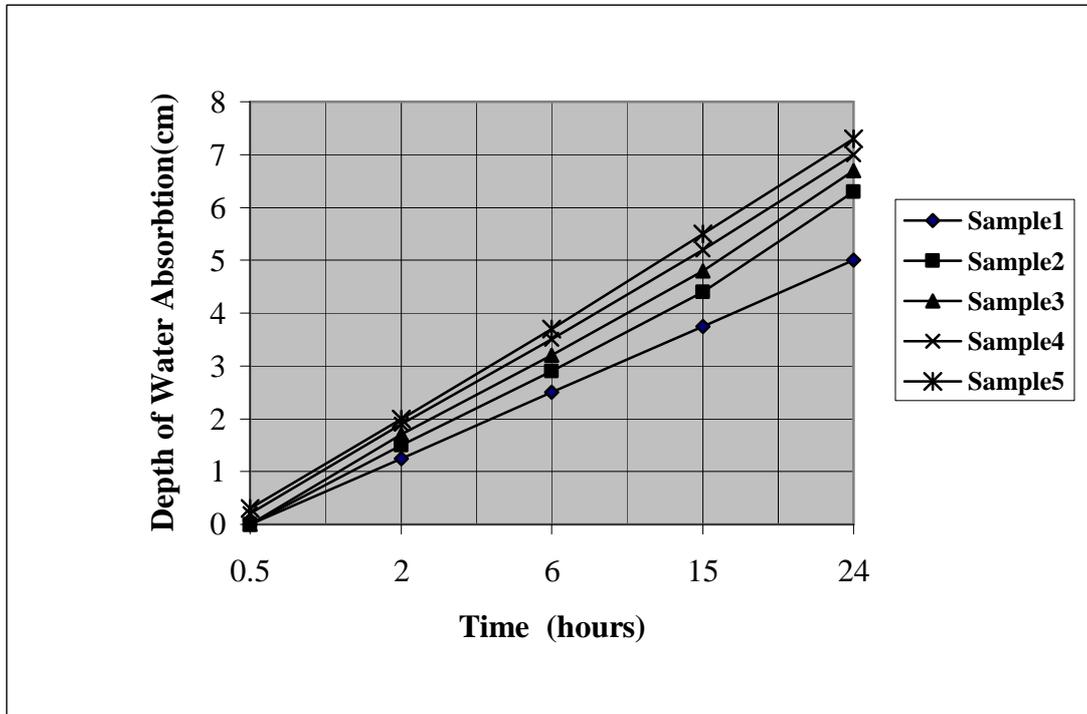


Fig. 3 :The capillary absorption test results for Thermostone samples.

It can be seen from this figure that the difference in water absorption between sample No.1 and the other samples were in the range (1.5-2.5 cm) which is not significant. Macropores, which are spherical and large in size, are almost closed and not connected so they possess negligible capillary suction. Accordingly, capillary suction takes place only through the micropores. Hence the rate of water penetration through Thermostone is very low. Iraqi standard specification No. 1441/2000 state that the capillary rise for all types of Thermostone should not exceed 12.5 cm. and according to ASTM C1386 is not exceed 7 cm

X – Ray diffraction results:

X-ray diffraction technique was used to show the final chemical phases of raw materials as well as green and autoclaved samples. Table- 4 and Table- 5 summarizes the results of XRD for raw materials and green and autoclaved samples respectively.

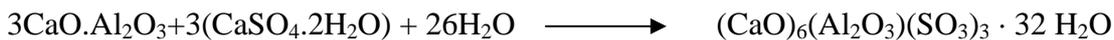
Table -4 :The X-Ray diffraction results of raw materials.

Cement	lime	Sand
60.13% CaO	90% CaO	85% SiO ₂
21.38 % SiO ₂		
6.85% Al ₂ O ₃		
3.71% Fe ₂ O ₃		

Table- 5 : X-Ray diffraction results of autoclaved and green samples.

Problem 3 Green samples (volume expansion)	Problem4 Green Samples (moisture spot)	Problem5 Autoclaved Samples (volume expansion)	10hr Autoclaving	14hr Autoclaving
34.12 %Ca(OH) ₂	31.3%Ca(OH) ₂	59.4%Tobermorite	75 % Tobermorite	84%Tobermorite
37.43% free SiO ₂	34.43% free SiO ₂	13.7% free SiO ₂	11% free SiO ₂	11% Xonotlite
15.7%Ettringite	21.4 % Ettringite	9.47% Ca(OH) ₂	8% Ca(OH) ₂	2% free SiO ₂
3% Monosulphate	9% Monosulphate	4.15% Hydroxyle ellestadite	4% Hydroxyle ellestadite	2% Hydroxyle ellestadite
		%3.45 Thaumasite		
		%2.1Magnesium silicate hydrates		
		%2.63 Magnesium sulfate (MgSO ₄)		
		2.48% Brucite		

The mechanism of formation of the above components can be summarized as follows: **Ettringite** : (CaO)₆(Al₂O₃)(SO₃)₃ · 32 H₂O : A medium temporary component that formed in the drying stage in the presence of calcium sulfate.



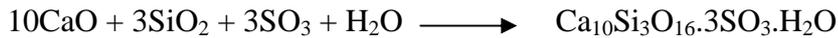
Monosulphate : Ca₄Al₂O₆(SO₄).14H₂O: A brittle detrimental component formed in the presence of high amount of C₃A and high Sulfate ,high ratio of Monosulfate causes moisture to remain in the cake as combined water after drying process.



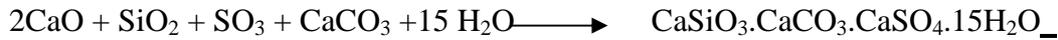
Tobermorite Ca₅Si₆O₁₆ (OH)₂. 4H₂O: A component with high mechanical and physical properties.



Hydroxyl- ellestadite: $\text{Ca}_{10}\text{Si}_3\text{O}_{16} \cdot 3\text{SO}_3 \cdot \text{H}_2\text{O}$

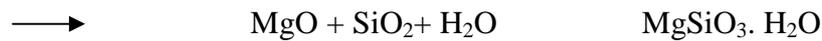


Thaumasite $\text{CaSiO}_3 \cdot \text{CaCO}_3 \cdot \text{CaSO}_4 \cdot 15\text{H}_2\text{O}$ which is a brittle component.



Thermostone is severely affected by Thaumasite formation and can easily be broken by hand fingers.

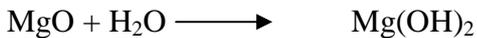
Magnesium silicate hydrates $\text{MgSiO}_3 \cdot 2\text{H}_2\text{O}$: A component causes volume expansion after autoclaving.



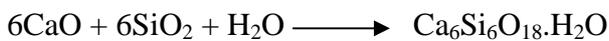
Magnesium Sulfate MgSO_4 : A brittle component causes volume expansion



Brucite : $\text{Mg}(\text{OH})_2$: A white component causes volume expansion



Xonotlite $\text{Ca}_6\text{Si}_6\text{O}_{18} \cdot \text{H}_2\text{O}$: A high quality component formed when the time of autoclaving increased from 10 to 14 hours ,this component increases the compressive strength of Thermostone to more than 3 MPa .



Problem No. 3: excessive expansion of green product

Table-5 shows that the formation of Ettringite and Monosulfate which resulted in volume expansion of the green (non autoclaved)product.

Problem No. 4: Moisture spots on the green product

Table - 5 shows that the formation of higher ratios of Ettringite and Monosulfate which resulted in moisture spots on the faces of the blocks because these two phases containing high combined water (32 H_2O in Ettringite and 14 H_2O in Monosulfate).

So it can be concluded that the main reason of both problems 3 and 4 is the formation of these two detrimental compounds (Ettringite and Monosulfate) which may be attributed to the presence of high ratio of C_3A and SO_3 in raw materials. C_3A as in table - 4 is a main ordinary Portland cement compound and it is difficult to eliminate this compound during cement manufacturing. So in order to solve these two problems (3 and 4) it is strongly recommended to eliminate SO_3 in raw materials other than cement.

Problem No. 5: Excessive expansion and low compressive strength of autoclaved product

Table - 5 shows a formation of relatively high ratio of Tobermorite phase (59.4%) after autoclaving which leads to high mechanical and physical properties of the product. On the other

hand, and because there was no control on raw materials ,the results shows a formation of detrimental phases like Thaumassite (brittle material), Magnesium silicate hydrate, Magnesium sulfate, and Brucite (responsible of volume expansion). So , it is again strongly recommended to control raw materials to avoid presence of detrimental materials.

The results of XRD of 10 hours autoclaved samples (manufactured with high control on raw materials) shows higher ratio of Tobermorite which leads to high compressive strength of the product. No detrimental phases were noted . However, the resulting compressive strength, sometimes , was lower than the specification requirements and the resulting blocks were considered as losses materials.

Increasing curing or autoclaving time from 10 to 14 hours improved properties of the resulting product. XRD results showed higher ratio of Tobermorite and a precence of new phase called Xonolite which increases the compressive strength to more than 3 MPa. So, when compressive strength more than 3 MPa is needed it is recommended to increase autoclaving time from 10 to 14 hours.

In an attempt to increase compressive strength of the product , different ratios of limestone aggregate (4 mm maximum size) was added to the raw materials. Table -6 shows densities and compressive strength of Thermostone block samples containing limestone.

Table-6: Effect of added limestone on compressive strength of blocks.

Density ρ kg/m ³	Added limestone ratio (% by wt.)	Compressive Strength (MPa)
540	1%	2.8
543	2%	2.85
557	3%	3.04
563	4%	3.17
568	5%	3.19

According to the above results, adding limestone to the mix of Thermostone improved compressive strength and reduces cracking. Iraqi specification for Cellular Concrete Block (Thermostone) requires a compressive strength not less than 2 MPa (Iraqi standard specifications No.1441/2000),whereas ASTM C 1386 requires a compressive strength not less than 3 MPa. So it may not be necessary to add more than 5% limestone.

Limestone particles work as a crack arrestor to stop macrocracks and preventing them from growing together into larger cracks which may cause failure of the product. Carlos⁽⁸⁾ reported that

addition of limestone has been found to reduce expansion as well as increasing compressive strength. Fig.4 shows free limestone particles in the structure of Thermostone block.

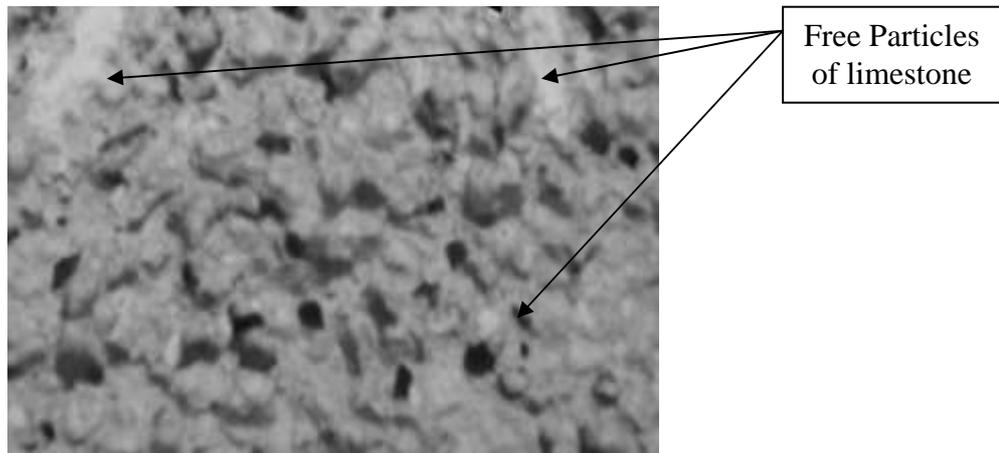


Fig.4 :Presence of limestone particles in the structure of Thermostone.

3.Conclusions:

Within the limitations of the present work and according to the results shown , the following conclusions could be reported to eliminate types and causes of failures in cellular concrete blocks (Thermostone) :

1- the main types of failure noted in the present study were:

- a-The cake failure due to no pores formation.
- b-The cake failure due to hydrogen bubble escaping.
- c- The product failure due to excessive volume expansion during hydrogen bubble formation.
- d- Brittleness due to residual moisture in the cake after drying.
- e-The failure of blocks after 10 hours curing in the Autoclave .

2- The complete reaction (Hydrogen bubbles formation) between lime (CaO) , water ,and Al-powder always occurs when the minimum reaction temperature is 43 °C, No observed reaction took place when the hydration temperature was less than 40 °C . In low reaction temperatures additional amounts of Al powder should be added. Uncompleted reaction resulted in failure of no pores formation.

3- Formation of large bubble size resulted in failure of Hydrogen bubble escaping which affect thermal and mechanical properties. Compressive strength increases with lower diameter of

aluminum powder for the same Al powder content. The suitable diameter of Aluminum powder was in the range of [50-100 μm] to get both perfect thermal and mechanical properties as well as capillary absorption which are consistent with ASTM and Iraqi specifications.

4- XRD (X-Ray diffraction) results showed that the formation of Ettringite and Monosulfate resulted in volume expansion failure of green (non autoclaved)product. Higher content of Ettringite and Monosulfate resulted in moisture spots failure of green product.

5- Excessive expansion failure after autoclaving was noted as a result of no control on raw materials and presence of detrimental Magnesium compounds and the brittle phase of Thaumasite after 10 hours of curing.

6- In spite of controlling raw materials ,compressive strength of 10 hours curing period may be less than the requirements of specifications in some cases of failure .Increasing curing time from 10 to 14 hours resulted in more than 3 MPa compressive strength which fulfill the specifications requirements.

7- Increasing compressive strength and reducing cracking could be achieved by adding up to 5% by weight limestone aggregate.

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