Synthesis of Light expanded clay aggregates from Iraqi raw materials

Samer Kareem Turki Khafaji1 and Elham Abed Al-Majed2

Abstract - In this study and by using available raw materials (clay) for producing lightweight aggregate in order to used them in many construction applications such as producing light weight aggregate concrete for thermal insulating purposes and lightweight in same time and also used them in multistory building as structural materials . This type of lightweight aggregate is called lightweight expanded clay aggregate (Leca) has wide ranges of uses for many reasons , easy to produce and available raw materials and it have good mechanical, physical and chemical properties.This study assesses the possible use of local raw materials (clays) for producing expanded clay pellets and test the product with American standard specifications (ASTM C330), and used it as lightweight aggregate for different construction applications like thermal insulation, lightweight structural concrete , decoration pieces ,etc. The rich calcite clay from Al-Anbar province used in this research to study their heat-treatment behavior. The rich calcite claywas bloated at 1150 Ĉ without adding any impurities ,just mechanical treatment and perfect firing program, in order to obtain good bloating coefficient that is related with firing parameters (temperature , access time, socking time). We was obtained low density aggregate about (448 Kg/m3) from clay I and (280 Kg/m3) from clay II.

Index Terms— Expanded clay, Lightweight aggregate, Clay, Leca, Light expanded clay aggregate

1. INTRODUCTION

THE porous medium as a lightweight aggregate(LWA) is an important and versatile material, used in concrete mixtures to make lightweight aggregate concrete (LWAC). LWAC has many wide rangeof applications like multistory building frames, curtain walls, shell roof, floors, pre-stressed, pre-cast elements and others [1].

Lightweight aggregate concrete consists of two types, the first one is structural lightweight aggregate concrete, which has unite density in the range of(1400-1840 kg/m³) compare with normal concrete with density about(2240-2400 kg/m³), for structural applications the concrete strength should be greater than (17 MPa) .The second type is insulation lightweight aggregate concrete, which has unite weight less than 1400 to about 800 kg/m³ and the compression strength less than (7 MPa) .LWAC used in a wide range in recent years and the requirements to it are increased, a reducing in the weight of the structure means that the foundations can be minimized and other structure element parts, and cost will be saved in erection and handling of components so that smaller lifting equipment can be used and larger per-cast unites can be made and handled [2].

Today, lightweight aggregates are available in wide range of densities, size and strength. This makes designers capable to design concrete with more options of densities and strengths for different applications [2].

There are three types of lightweight aggregates, the first one is natural lightweight aggregate materials, prepared by mechanical methods, crushing and sizing such as scoria, pumice, tuff etc. The second type is manufactured lightweight aggregates prepared by thermal processing (pyroprocessing) in rotary kiln or in traveling grate sintering machines, the raw materials to produce this type of aggregate are expanded clay, shale, perlite, vermiculite and slate. The third type is industrial by-product such as expanded slag, fly ash, and cinder[1].

Expanded clay is a powdery building material with good thermal insulation properties and resistance to the external impacts. It is obtained by feeding clay in a rotary kiln at a temperature of (1150Ĉ) . In this temperature raw materials start bloat turning into light pellets with a porous and firm structure. Properties of the obtained product depend not only on the properties of the start materials , but also on its additives and production technology [8].

The manufacturing steps that used in this research for producing expanded clay pellets are the raw materials supplied as rock forms, then we will crush and mill rock into powder . After milling process, test the powder using the following tests Particle Size , XRD, DTA and Plastic & Liquid limit. The clay prepared with mixing it with water into pasts, then drying and firing process will be done. The final product expanded clay pellets will test several tests.

2. MATERIALS AND METHODS

2.1. RAW MATERIALS

Clay samples supplied by "S. C for mining industries – Department of mineral extraction " . The first type of clay

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was collected from Al-Najaf province, it is called in this study clay I, and which is characterized by not self-bloating clay. The second type of clay was collected from Al-Anbar Province, it is called in this study clay Π, and which is characterized by self-bloating clays. It is worth mentioning that the clays are classified according to the coefficient of bloating, clays are divided into the following groups:Kp**>4** clays of good bloatability, Kp = 2.5 - 4 middle bloat ability, Kp = 2–2.5 poorly self-bloating, and Kp< 2.5 – not self-bloating clay [23].

Compositions of the clay were determined by the classical methods of chemical analysis for silicate material and are presented in table 1.Dehydrated and ground clay have (specific surface area for clay I (SSA= 0.692 m2/kg) and for clay (SSA= 0.447 m2/kg)by Battersize 2000 Device) shown in (figure 1-A, 1-B).

TABLE 1 CHEMICAL ANALYSIS FOR CLAY I AND CLAY II

Clay type	SiO_2 %	Al_2O_3 %	CaO %	K_2O %	L.O.I %
Clay I	55.57	14.91	3.88	0.37	8.79
Clay Π	44.28	8.62	13.86	1.46	17.51

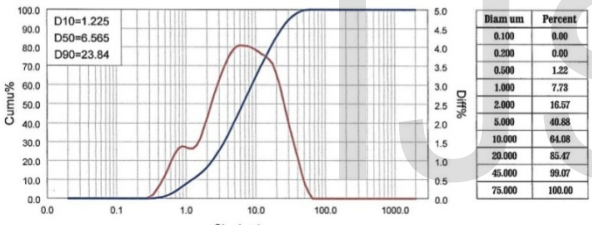

Fig. 1.A Particles distribution of clay I

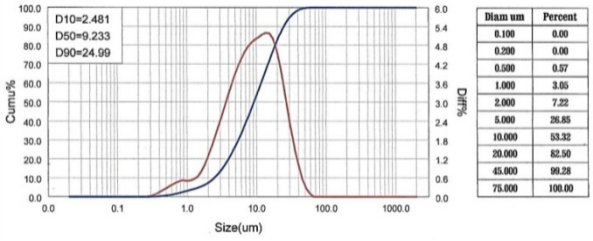

Fig. 1.BParticles distribution of clay II

2.2. XRD X-RAY DIFFRACTION

It is a rapid analytical technique, primarily used for phase identification of crystalline materials and also can provide information on unite cell dimensions. It is generated in cathode ray tube by heating a filament to produce accelerated electrons toward the target by applying high voltage. The interaction of the incident rays with sample produces constructive interference and diffracted ray when conditions satisfy the Bragg law[15].

$n\lambda=2d \sin 2\theta$

Where θ, is the angle of incidence of the x=ray. λ ,is the wave length of the X-rays used and d, is the spacing between atom layer. X-ray diffraction (XRD) type (Shimadzu 6000, Japan) used to characterize the structure of clay samples .

The measurement conditions are show below:

Measure Conditions
X-raySlit
Target : Cu Divergence : 1.0(deg)
Wave : 1.54060 (A) Scatter : 1.0(deg)
Voltage : 40.0 (KV) Receiving : 0.3 (mm)
Current : 30.0 (mA)
Measure
Axis : Theta- 2 theta
Scan Mode : Continuous Scan
Range : 10.0 – 70.0 (deg)
Step : 0.02 (deg)
Speed : 6.0 (deg/min)
Preset Time : 0.2 (sec)

Subsequently, the start powder of clay samples was prepared for the XRD test after milling and sieving (mesh 200). All data were compared with XRD standard cards.

2.3. DTA THERMAL ANALYSIS

Differential thermal analysis is widely used in ceramic industry. By this technique, the temperature differences ΔT relative to a thermally inert material are measured during heating or cooling of sample. The DTA curves records these differences during reactions in the sample, showing thermal effects as deviations from the zero line. The diagram of DTA is coordinated by ΔT ordinate and sample temperature in abscissa, both given in C°. The curve slope of DTA is directed toward the upper side of the zero line to represent endothermic .

The lower side differential thermal analysis is used for many purposes. It is useful in identifying the peculiarity of minerals, determining the phase changes which occur when the material is subjected to various temperature ranges, and investigating the temperature degree at which the reaction commences, and investigating the bulk crystallization especially for the glass ceramic materials.

2.4 PLASTIC LIMIT AND LIQUID LIMIT

This test measured the plastic and liquid limits of the clay samples used in this study. The liquid limit (LL) is arbitrarily defined as the water content, in percent. By using Liquid Limit Device(mechanical device). Amount of clay mixed with water and put in a standard cup, then cut by a groove of standard dimensions after this the clay is subjected to 25 shocks from the cup being dropped 10 mm with a rate of two shocks per second, to find the liquid limit of the clay samples, the clay will flow together at the base of the groove for a distance of (13 mm) by using equation below, according to ASTM(D 4318).

The plastic limit (PL) is the water content, in percent, at which a soil can no longer be deformed by rolling into 3.2 mm, diameter threads without crumbling, according to ASTM(D 4318).

The plasticity index (PI) of a soil is the numerical difference between its liquid limit and its plastic limit, and is a dimensionless number. Both of the liquid and plastic limits are moisture contents.

The next equations used to calculate LL,PL and PI.

LL=W (N/25)0.12

PL = (Weight of water/ Weight of oven - dry soil)*100%

Plasticity Index = Liquid Limit - Plastic Limit

PI=LL- PL Where, N : number of shocks , W : weight percentage of water.

The table 3 shows the PL, LL, PI of two types of clay

TABLE 3.THE PL, LL, PI OF TWO TYPES OF CLAY

Clay type	*PL*	*LL*	*PI*
Clay I	*0.672*	*0.3*	*0.372*
Clay II	*0.942*	*0.5*	*0.442*

3. PRODUCTION OF EXPANDED CLAY PELLETS

Dehydrated clay was crushed with a laboratory jaw crusher in a laboratory and milling it. In this study, mix clay I with different organic impurities to enhance their ability to bloat, start with (3 – 5 – 7 – 10 %)using Sawdust, Dolomite ore and clay II with (10 – 20 – 30 – 40 %),then mix with water and finally mold by extruder for producing pellets with diameter 8mm and different length with range (10 – 15) mm as shown in the figure (2). The clay Π does not need any additions only use mechanical treatment milling and mixing with water and finally molding by using extruder in same way with clay I. Then supplied for bloating to a laboratory electric furnace fired to a temperature of 1050 °C – 1170 °C and kept therein for10 min. Bloatability tests were performed at temperatures differing by 20 °C until the semi-melt temperature of then specimens was attained.

Upon heating completion, the hot specimens were took out of the laboratory furnace , cooled at the room temperature, weighed and submersed into a water bath for 24 hours by burdening them. The soaked specimens were removed from water, wiped with a moist cloth and weighed, and their volume was measured with a volume meter. The next steps of producing expanded clay pellets are drying and firing, the firing program is shown in (table 4-A,4-B).

In expanded clay production, setting of the optimum firing temperature is very important because temperature is very low, clay pellets do not bloat up to the full extend, and if it is very high clay pellets stick together forming monolith, which is undesirable. In order to select the optimum clay firing temperature, the specimens were fired in the range (1050 – 1170 C°).

Fig. 2. The shape of samples.

TABLE 4.A DRYING AND FIRING PROGRAM FOR CLAY I

drying & firing	*access time(min)*	*Socking time(min)*
Room Temp to 105C°	*5*	*10*
105 C to 200 C	*10*	*10*
200 C to 1170 C	*25*	*40*

TABLE 4.B DRYING AND FIRING PROGRAM FOR CLAY II

drying & firing	*access time(min)*	*Socking time(min)*
Room Temp to 105C°	*5*	*10*
105 C to 200 C	*10*	*10*
200 C to 1170 C	*20*	*25*

4. RESULTS AND DISCUSSIONS

Table 5 shows the XRD results for the sample clay I, that contain quartz, feldspar. Table 6 shows the XRD results for the sample Clay II, that contain quartz, calcite, dolomite and small amount of feldspar. Behind that figure 3-A, and figure 3-B show the phases of XRD test for clay I and clay Π respectively.

TABLE 5.THE XRD RESULTS FOR THE SAMPLE CLAY I

2θ	*Phase*
20.88	*Quartz*
26.50	*Quartz*
27.95	*Feldspar*
36.50	*Quartz*
38.75	*Quartz*
40.30	*Quartz*
50.07	*Quartz*

TABLE 6.THE XRD RESULTS FOR THE SAMPLE CLAY II

2θ	*Phase*	*2θ*	*Phase.*
20.80	*Quartz*	*43.15*	*Calcite*
23.05	*Calcite*	*45.79*	*Quartz*
26.63	*Quartz*	*47.50*	*Calcite*

27.95	*Feldspar*	*48.50*	*Calcite*
29.39	*Calcite*	*50.07*	*Quartz*
30.98	*Dolomite*	*55.29*	*Quartz*
35.95	*Calcite*	*59.95*	*Quartz*
36.50	*Quartz*	*67.74*	*Quartz*
39.31	*Quartz .Calcite*	*68.14*	*Quartz*
42.61	*Quartz*	*68.31*	*Quartz*

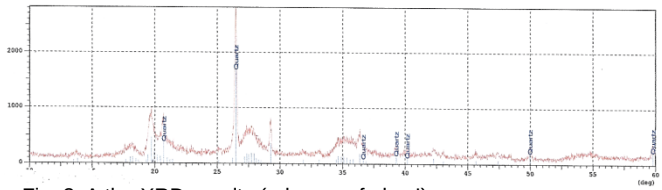

Fig. 3. A the XRD results (phases of clay I).

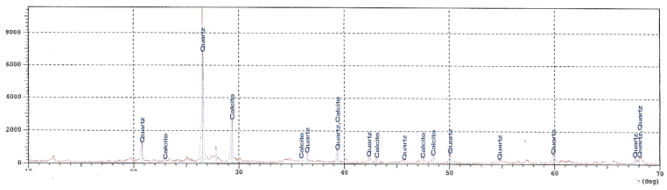

Fig. 3.B the XRD results (phases of clay II).

The DTA curve of clay I sample is shown in figure 4, this curve shows the clearly exothermic reaction at 332.6 C°, that is caused by crystallization of phases that have low temperature crystallization. In the present study differential thermal analysis was recorded for the samples at an average heating 5°/min. Figure 5 of clay II curve shows that there are different reactions, the first reaction is endothermic at 110.7 C° because of the evaporation of water .The second one is more less reaction, it's for removing absorbed water . The third peck is exothermic reaction at 590.8 C° for crystallizations. And the last peck is also exothermic reactions at 823.2 C°, it is for TG transformation.

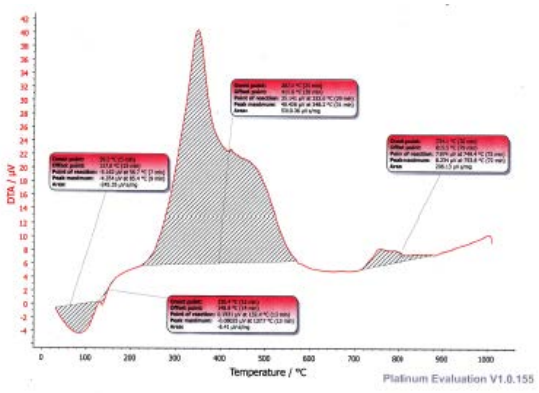

Fig. 5 the DTA curve of clay I.

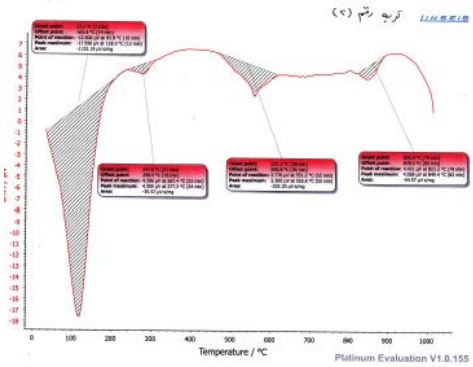

Fig. 6 the DTA curve of clay II.

4.1 BLOATING COEFFICIENT OF CLAY SAMPLES

In this research clay formed, 8 mm in diameter using a laboratory piston press and cut into specimens, 10 mm long. The specimens, dried at a temperature of 105 °C, then weight and volume are calculated.

Afterward, the dry specimens were heated at a temperature of 200 °C for 20 min and fired at high firing rate by using a laboratory electrical furnace to a temperature of 1050 °C –1150 °C and stay in the furnace for 25 min

Bloating tests were performed at temperatures differing between(20-50 °C)until the semi-melt temperature of the specimens was attained. After heating the specimens are taken out of the furnace , cooled at the room temperature, then weighted and submersed into a water for 24 hours by flooded them. The specimens are removed from water, wiped with a cloth then weighted, the volume (V2) is measured . According to the obtained data, bloating coefficients Kp are measured by using equation below

$$Kp = \frac{V2}{V1}(4\text{-}1)$$

Where,

Kp: Coefficient of bloating, V1: Volume of sample before firing.V2: Volume of sample after firing.

4.1.1 EFFECT OF FIRING TEMPERATURE ON BLOATING COEFFICIENT

Figure 6 shows the relation between coefficient of bloating and firing temperature for clay I.From the results we find the bloating coefficient proportional directly to a certain limit with increasing temperature then decrease, this is because the clay start to melt after this degree, so that the firing should be happened under melting temperature about (50-30) degree, this degree allows to make porous medium because the skin begins to melt and the core continue to gases free, this gases are the reason of bloating.

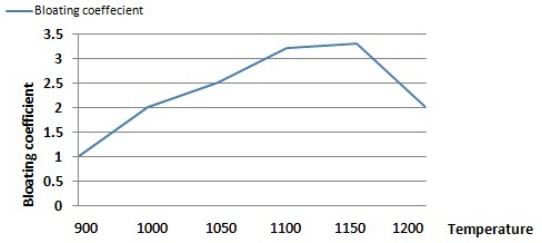

Fig. 6 the relation between coefficient of bloating and firing temperature for clay I with(30% clay II)

The figure 7 shows the relation between coefficient of bloating and firing temperature for clay Π. The results show that the pellets produced from clay Π have the ability of bloating more than clay I, because the clay Π has more gases source as shown in different tests (XRD, Chemical analysis). According to the standard specifications, clay Π has good boatability, but clay I has no ability to bloat because there is no enough amount of gases source, so for this reason, some impurities must be added . Figure 8 shows the effect of temperature on ability for bloating of expanded clay pellets.

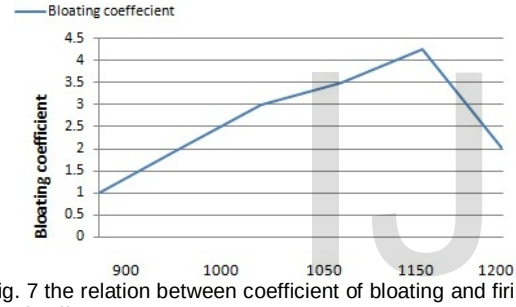

Fig. 7 the relation between coefficient of bloating and firing temperature for clay II .

Fig. 8 the shape of pellets change with temp.

4.1.2 Effect of staying time on bloating coefficient

Figure 9 shows the relation between coefficient of bloating and staying time for clay I(with 30% clay II) From the results, the bloating coefficient depended on staying time. It is increased with the increasing time to a certain limit 40 min for clay I , then starting to drop, this

because of the input energy increased with time for clay I, so it begins softening, and then melting. Figure 10 shows relation between bloating coefficient with staying time for clay II, also we found the bloating coefficient increases with the increasing time, but to a certain limit (25 min) less than clay I, because the clay II has more percentage from fluxes in its composition.

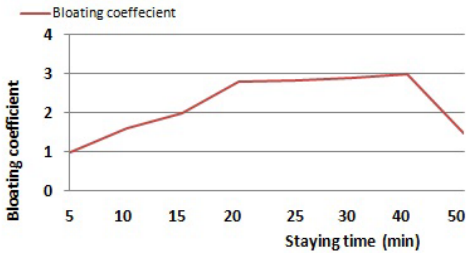

Fig. 9 the relation between coefficient of bloating and staying for clay I (30% clay II).

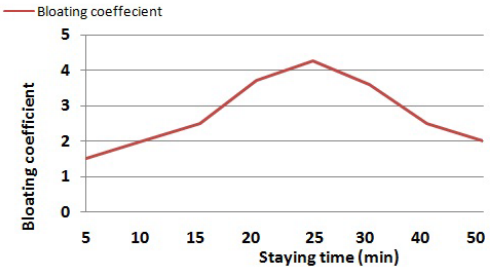

Fig. 10 the relation between coefficient of bloating and staying for clay II.

4.1.3 Effect of impurities on bloating coefficient

The impurities used only with clay I, because clay II expanded without need to any impurities. So the Figures 11, 12, 13 show the relation between bloating coefficient for clay I and different impurities (sawdust, dolomite ore, clay II).From the result we recognized the amount and type of impurities that have the direct effect on the coefficient of bloating. It was noticed that the coefficient of bloating increased with the increasing amount of sawdust to a certain limit (5%) of total weight, then stop raising. In case of using dolomite we noticed that there is no change because the dolomite has a high melting temperature and difficult to free gases. We also used clay II as impurities to increase bloatability, because it has calcite and dolomite in its structure, this phases frees gasses in high temperature, and these gases produce porous medium, that we found the perfect amount of clay II which is 30% from total weight.

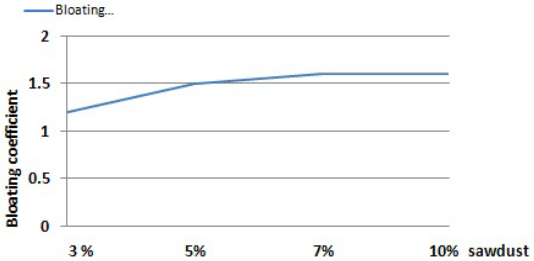

Fig. 11 the relation between coefficient of bloating with sawdust ratios.

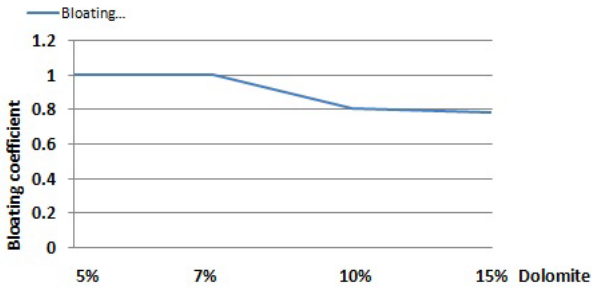

Fig. 12 the relation between coefficient of bloating with dolomite ratios

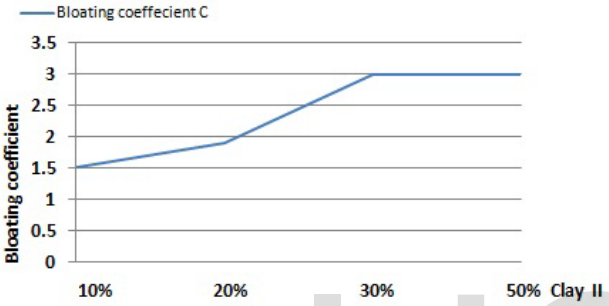

Fig. 13 the relation between coefficient of bloating with clay II ratios

4.2 PHYSICAL PROPERTIES

4.2.1 BULK DENSITY

The bulk density of expanded clay pellets that was produced from clay I and clay II were measured according to American standard ASTM (C 29/C 29M – 97) , shown in the table (4-7).The density of lightweight pellets that produced from clay II (260-290 kg/m³), was less than pellets that produced from clay I (448-1200 kg/m³), because clay II has big amounts of calcite that produce more gases during firing compare with clay I. Finally the two type of pellets when using them as lightweight aggregate have density smaller than normal aggregate and when compare them with ASTM C330 that identify the lightweight aggregate should be no more than 880 kg/m³ , except the pellets produced from adding dolomite ore because the dolomite has a high melting tempera ture

Sieve size mm	*Passing %*	*Limits of America specifications ASTM (C 136)*
25	100	100
19	100	90-100
9.5	30	10-50
4.75	3	0-15

4.2.2 POROSITY MEASUREMENT

Table 7 shows the results of porosity for expanded clay as lightweight aggregate concrete that produced in this study by using clay I and clay II. The results show that the structure of expanded clay have high porosity, that means the weight will be decreased and thermal conductivity will be also decreased. The porosity of clay II larger than clay I because the structure of clay II contain amounts of calcite, that release gases during firing that make the structure more porous.

4.2.3 SPECIFIC GRAVITY

Table 7 shows the results for specific gravity for clay I and clay II pellets The results of specific gravity are very important because of identifying the expanded clay whether it is lightweight or not, after compare the results with ASTM C330, it is found that the two types of expanded clay are lightweight aggregates.

4.2.4 WATER ABSORPTION MEASUREMENTS

Absorption calculations shown in the following ,as well as comparing the results with the specification and find out the validity of the product in the table 7. Note that the test was conducted according to standard (ASTM C 127). Clay I has different values of water absorption because of difference composition and different impurities .These values show that clay II pellets has low water absorption percentage because of the shield that covers the surface of pellets such a glass that prevents water from enters inside pellets.

TABLE 7. THE PHYSICAL PROPERTIES OF CLAY PELLETS

Property	*Clay I*	*Clay II*
Bulk Density	448 kg/m³	280 kg/m³
Porosity	32.5 %	46.6 %
Specific Gravity	886 kg/m³	448 kg/m³
Water absorption	7.8 %	6 %

4.4.4 SIEVING.

Table 8 shows the grading of expanded clay pellets as lightweight aggregate according to ASTM (C 136), the results refer to the aggregates that have the grading (4.75-19 mm) [12].

TABLE 8. GRADING OF EXPANDED CLAY PELLETS

CONCLUSIONS

In this research for the first time , the expanded clay pellets has been produced equal in quality with the expanded clay pellets that manufactured outside the country, by using the available local raw materials and by simple technology.

During the study of physical properties , the expanded clay as lightweight aggregate has been according to ASTM C 330 .Through laboratory tests, it is considered successful by all standards and can be used to produce lightweight concrete.

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