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Evaluate the Compressive Strength of Cement Paste Modified with High Reactivity Attapulgite and Affected by Curing Temperature

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Abstract

This study explores the influence of partial replacement of high reactivity Attapulgite (HRA) with cement by weight and evaluate the effect of curing temperatures on the compressive strength of modified cement paste (MCP). Recently, the Iraqi clay(Attapulgite) has been processed to pozzolanic material HRA, after extracting it from the quarry the clays crushed and grind to filler then specific the suitable calcinations temperature to make this clays as a pozzolnic material. The possibility of replacing the Iraqi clays with cement can be reduce the cost and the impact of cement manufacturing on environment. In this study, three percentages of high reactivity Attapulgite used as a replacement 0, 10 and 20 % by weight of cement. The samples cured in four temperatures 20, 40, 60 and 80°C. The samples with dimension 50x50x50 mm³ were casted and tested at ages 7, and 28 days. The test result shows that the compressive strength at early ages without HRA and cured at 20°C were higher than samples with HRA. The maximum percentage of HRA as a replacement with cement has a reduced the compressive strength of the concrete. The result at 28 days for the samples with 10% of HRA at curing temperature 40 °C shows increased in compressive strength up to 60 °C, while when the samples were cured at curing temperature 80 °C shows decreased the compressive strength.

Keywords: Cement paste; high reactivity Attapulgite; curing temperature; compressive strength; pozzolanic material

1. Introduction

Many variables affect cement hydration, like water to cement ratio, the chemical composition of cement and the presence of mineral filler additives or replaced. However, another variable is playing a significant effect on the early hydration process, and the hardening cement properties paste (CP) that variable is the environment temperature (ET) [1]. The environment temperature (ET) during the mixing process of cement and curing for samples varied; as in practice, it is essential to understand the affect of the curing temperature on the behaviour of the cement.

Previous studies have indicated that the strength can be increase at an early age of the curing due to increased hydration in cement, resulting from elevated temperatures treatment [2-4]. A new study [5] displays a high increase in the compressive strength for a sulfate-resistant cement at an early age when treated with high temperatures; however, the high-temperature effect on the mechanical properties un favourable in15 days age. Conversely, observed when cement paste treated at curing temperature at 22°C or less, the development in compressive strength was slowly with time. The high initial rate of hydration produces irregular distribution and dense clusters consistency shown in the

microstructure of paste, the hydrating grains retarding the following hydration, resulting in an increase in paste and adverse porosity effects on long-term of compressive strength.

Maltaisand and Marchand [6] studied the curing temperature effect on the compressive strength of mortar samples containing fly ash FA as additional material. Three percentage of FA were used 10, 20 and 30% by weight of cement with the temperature of curing 20°C and 40°C. The study showed a decreased compressive strength of the samples without FA when increased the curing temperature, while the samples with FA showed no effect on long-dated compressive strength.

Teixeira et al. [7] used TiO₂ nanoparticles to modify cement paste and studied the properties of the cement paste mixes at high temperatures of curing. The research results showed that cement paste acquired early compressive strength when cured with high temperature against the normal temperature curing; however, the paste's increase of compressive strength decreases in later ages. It was also noticed that high temperature curing lowers the compressive strength of cement pastes at later ages as higher levels of TiO₂ nanoparticles are added.

Elkhadiri et al. [1] studied the hydration of cement with a high temperature of curing. Two cement paste CP types were used CEMI //42.5R and CEMII /A-V// 42.5R. The CP samples were cured at a temperature between 4 to 80. The results showed an increase in the curing temperature leads to increased compressive strength at early age and inversion at delay ages.

Turuлло and Mallisa [8] used the FA as a replacement material with different percentage of 0, 10, and 15 % by weight of cement. They studied the effect of three temperatures of curing 25, 30 and 50°C on compressive strength. The results showed that the compressive strength of concrete samples without FA at 25°C in early ages was higher than concrete samples with FA. The tests on samples showed decreased in compressive strength with increase in replacement percentage of FA. Moreover, when a higher temperature of curing was applied at an early age resulted in an increased value of concrete samples' compressive strength.

Narmluk and Nawa [9] also studied the curing effect on cement's pozzolanic activity containing FA. The study proved that past the temperature of curing has a significant effect on pozzolanic activity. All the researchers proved that an increase in the treatment temperature with a decrease in fly ash percentage leads to an increase in the pozzolanic activity.

2. Objectives of the study

There are two objectives of the study: (i) Evaluating the effect of using the high reactivity Attapulgit (HRA) as a replacement material with cement in the paste. (ii) Measuring the compressive strength of samples under the effect of curing temperature.

3. Experimental Work

Wooden moulds of 50x50x50 mm³ size were used to cast samples of specimens. The curing period was about 7 and 28 days. To study the curing temperature influence on the samples' performance, they were cured to different temperatures of 20, 60, 40 and 80°C in a water bath oven. Then all the samples were tested to find the compressive strength.

Materials

3.1. Cement: Type 1 was used, both the physical properties and the chemical composition of the cement used in this study are shown in Tables 1 and 2, respectively. The results are corresponded to the Iraqi Specification No. 5/1984 [7].

3.2. High Reactive Attapulgit: Table 3 shows the physical and chemical properties of the Attapulgit. The Attapulgit is a fibrous clay mineral as kind of crystalloid hydrous Magnesium - Aluminum silicate mineral and have chemically absorbed water in its structure The Attapulgit is the chemical name, and the other name is Palygorskite. It is complex

magnesium, aluminium and silicate with a structure can be classified as an open-channel structure that forms extended 'needled-shaped' crystals. The chemical formula that can describe the HRA is $\text{Si}_8 \text{Mg}_5 \text{O}_{20} (\text{OH})_2 (\text{OH}_2)_4 \cdot 4\text{H}_2\text{O}$ [11]. The high reactivity Attapulguite is a chemical stage formed by the heat treatment of Attapulguite powder, and as a result of this heat treatment, the water is pushed to form an amorphous substance that reacts with the lime. Attapulguite rocks are milled with a blowing technique to achieve a high-quality surface. The used Attapulguite was conformed to the ASTM C618 [12] with SiO_2 (45.87) and a specific gravity of 2.6 and the pozzolana activity index (130).

3.3. *Preparing the (mineral filler) High Reactive Attapulguite (HRA)*: This type of clay found in Al-Najaf and Karbala regions, Injana formation are exposed in Al-Najaf region (Tar Al-Najaf) as bluish-green, and grey claystone, 0.5 m thick with plants remains. The used Attapulguite was prepared by crashed the big rocks into small parts by hammer then burned in an electric oven at 750°C with a steeping time of 1/2 hour and then allowed to cool as shown in Fig.1. This burning process is based on the work done by Al –Amide [13,14]. The material was ground into a fine material and then graded, the passing filler throw sieve No. 200 was used in this study.

4. Thermal analysis of Attapulguite

A DTA test was performed to identify the thermal behaviour of the altabolate at different temperatures, as shown in Fig.2. The bottom curve refers to thermal energy absorption due to the evaporation of free and combined water. Then the release of gases as carbon dioxide results from the combustion of organic materials and the disintegration and decomposition of the mineral components of the material. At the same time, the tops of the curve upward indicate the emission of energy due to the formation of new phases in the body.

5. Result and Discussions

The pozzolanic materials can be used in concrete and cement manufacturing to lower the carbon footprint associated with Portland cement. The Attapulguite HRA replacement with cement can decrease the cost and environmental hazard. The possibility of replacing the Iraqi clays with cement in concrete mixture, which is available on large areas in Iraq and for a small fee per ton comparison with cement prices in Iraq which cost 9 dollars per 50 Kg. This leads to reduce the cost and reduce the effect of cement manufacturing. Where previous studies showed That the cement production is one of the most energy intensive of all industrial manufacturing process. Including direct fuel use for mining and transporting raw materials. The industry's heavy reliance on coal leads to especially high emission levels of CO_2 , nitrous oxide and sulphur, among other pollutants. A sizeable portion of the electricity used is also generated from coal [15].

The specimens in this study were prepared according to ASTM C-305-20[16]. The mix proportion chosen was 1:0.36 (cement: w/c) (see Table 4). The compressive strength test followed the ASTM C109/C109M-05 [17] procedure for $50 \times 50 \times 50 \text{ mm}^3$ cubes. The samples were tested at two different curing ages, the first one is 7 days then 28 days (with three specimens per age per temperature).

The test results indicated a rise in compressive strength with time this behavior attributed to the significant reduction in capillary porosity of the cement matrix, as well as to a good dispersion of the cement grains throughout the mix. And also the results show a reduction in the value of compressive strength, when increasing the percentage of HRA replacement (see Fig.3). The reduction in the compressive strength for M10 and M20 was reported to be (27.82% and 42.02%) at 7-days age, and (8.60% and 49.78%) 28-days age at 20°C . Due to the hydration rate of HRA, which is a lower than cement hydration rate, especially at the early hydration ages [18]. The value of all sample's compressive strength within requirement limitation.

The Fig.4 to Fig.6 showed the relationship between compressive strength at different ages and curing temperatures for all the replacement percentage (M0, M10 and M20). The results show an increase in compressive strength with age for all the mixes and for all temperature curing. Moreover, the results in Table 5 indicated that the maximum value of compressive strength for M0 was at curing temperature 40°C and the value decreased to (14.49% and 30.12%) at (7-28- days age) respectively for 80°C curing temperature. The results show a little drop in value up to 80°C compared with M10 with the same curing temperature. This might be because all cementitious materials reactions were close to completion or had stopped; mainly because the reactions between HRA and cement mixtures were slowed down with time.

The compressive strength of samples M10 at early ages, and 60°C curing temperature was higher than that samples with different curing temperatures, the advance of compressive strength value were 184.33% and 8.433% for (60 °C and 80 C°) respectively and at later ages, the samples of 40 C° curing temperatures was the higher. This increase is mainly due to the pozzolanic reaction of the HRA with calcium hydroxide liberated during the hydration of cement. This reaction contributes to the densification of the concrete matrix, thereby strengthening the transition zone and reducing the micro cracking leading to a significant increase in compressive tensile strength. It is explained back to the dense hydrated phases formed around the unreacted cement particles, which stops the hydration. However, when the paste was cured at 80 C°, a decrease in the compressive strength was observed. This is due to the high hydration temperature, which caused a decrease in the porosity gel and increased the capillary porosity [19].

The mixture with a high replacement percentage of Attapulgit HRA M20 continuous increases in compressive strength with temperature curing (64%, 46.42% and 19.28%) at 28-days age for (40 C°, 60 C° and 80 C°) respectively. The principal reaction between HRA and calcium hydroxide was derived from cement hydration in water. HRA/ Calcium Hydroxide ratio and temperature of the reaction has controlled the production of crystalline and cementitious gel, and this reaction improves the binding properties of cement paste [17].

6. Conclusions

This study has shown an experimental program set to look at the effect of curing temperature on cement paste modified with high reactivity Attapulgit HRA. The main finding was that the value of compressive strength decreased with the increase in the percentage of replacement with HRA up to 10%. For the limitations presented in this paper, the below conclusions can be drawn:

- The compressive strength increased when increasing the curing temperature for all mixes.
- The maximum compressive of M0 mixes was at 40 C° (65.5 MPa) at 28-days age; meanwhile, the compressive strength for M10 mixes at an early age was at 60 C° (70.8MPa), and the late age was at 40 C° (75.7Mpa).
- The increase of the HRA content up to 10% negatively affected on compressive strength value. Resulting in a reduction at 7-days age by in the compressive strength for (M10 and M20) (27.82% and 42.02%), respectively. Furthermore, at 28-days age the reduction was by (8.60% and 49.78%), respectively at 20 C°.
- The best results shown in at 40C° curing Temperature and 10% of HRA as a replacement with cement.

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References

- [1] Elkhadiri I, Palacios M, and Puertas F.,” Effect Of Curing Temperature On Cement Hydration” *Ceramics – Silikáty* 53 (2) 65-75 (2009).
- [2] Lanh Si Ho, Kenichiro Nakarai, Kenta Eguchi, and Yuko Ogawa“Difference in Strength Development between Cement-Treated Sand and Mortar with Various Cement Types and Curing Temperatures”, 6 Nov2020.doi: 10.3390/ma13214999.
- [3] Ki-Bong Park and Takafumi Noguchi"Effects of Mixing and Curing Temperature on the Strength Development and Pore Structure of Fly Ash Blended Mass Concrete", *Advances in Materials Science and Engineering*, Vol. 2017 ,<https://doi.org/10.1155/2017/3452493>.
- [4] Ch.Pichler, M.Schmid, R.Trax , R.Lackner,"Influence of curing temperature dependent microstructure on early-age concrete strength development", *Cement and Concrete Research*, Volume 102, December 2017, Pages 48-59.
- [5] Elkhadiri I., and Puertas ., F. “The effect of curing temperature on sulphate-resistant cement hydration and strength” *Construction and Building Materials* Volume 22, Issue 7, July 2008, Pages 1331-1341.

- [6] Maltaisand Y. and Marchand J.” influence of curing temperature on cement hydration and mechanical strength development of fly ash mortars” *Cement and Concrete Resesmh*, Vol. 27, No. 7, pp.1009-1020,1997.
- [7] Teixeira, K., P., Rocha I., P., , Carneiro, L., De Sá, Flores, J., Dauer E., A. and Ghahremaninezhad A.” The Effect of Curing Temperature on the Properties of Cement Pastes Modified with TiO₂ Nanoparticles” *Materials* 2016, 9, www.mdpi.com/journal/materials pp 1-15.
- [8] Turuallo, G., and Mallisa” Sustainable cementitious materials: The effect of fly ash percentage as a part replacement of portland cement composite (PCC) and curing temperature on the early age strength of fly ash concrete” *MATEC Web of Conferences* <https://doi.org/10.1051/mateconf/2019225810015801001> SCESCM 2018 1-6.
- [9] Narmluk M., and Nawa T.” Effect of Curing Temperature on Pozzolanic Reaction of Fly Ash in Blended Cement Paste” *International Journal of Chemical Engineering and Applications*, Vol. 5, No. 1, February 2014.
- [10] Iraqi Specification, No. 5/1984., “Portland cement”.
- [11] Attapulgitte Technical Evaluation Report, Handling/Processing/ Compiled by the Technical Services Branch for the USDA National Organic Program February, Technical Evaluation Report Vol.1, 2010, pp. 1-10.
- [12] ASTM C618-03, “Standard Specification For Coal Fly Ash And Raw Or Calcined Natural Pozzolan For Use In Concrete”, Annual Book of ASTM Standard, Vol. 04-02, February, 2003, p. 3.
- [13] Al –Amide, S. H. M., “Some Properties Of Concrete Containing Fired Local Attapulgitte Clay” M.Sc. Thesis, University of Technology, December, 2012, pp.1-87.
- [14] Mohammed E.A.H., Mohamad.S., Khaled T.,Alzubaidi A., “Study the Effect of Mineral Filler on the Mechanical Properties of Hot Mix Asphalt”, *IOP Conf. Series: Materials Science and Engineering* 870 (2020) 012086.
- [15] BABOR D., PLIAN D. and JUDELE L., “Environmental Impact of Concrete”,*Bulletin of the Polytechnic Institute of Jassy, CONSTRUCTIONS. ARCHITECTURE Section LV (LIX)(4)*, January 2009.
- [16] ASTM C-305-20 Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency <https://doi.org/10.1520/C0305-20>.
- [17] C109/C109M-13 (2013) Standard test method for compressive strength of hydraulic cement mortars. <https://doi.org/10.1520/c0109>.
- [18] HamdyEl-Diadamony, Ahmed A.Amer, Tarek M.Sokkary, SamirEl-Hoseny, ‘Hydration and characteristics of metakaolin pozzolanic cement pastes”, *Volume 14, Issue 2, August 2018, Pages 150-158* <https://doi.org/10.1016/j.hbrcj.2015.05.005>.
- [19] Bahafid S., Ghabezloo S., Duc M., Faure P., and Sulem J.” Effect of the hydration temperature on the microstructure of Class G cement: C-S-H composition and density” *Cement and Concrete Research* Volume 95, May 2017, Pages 270-281.

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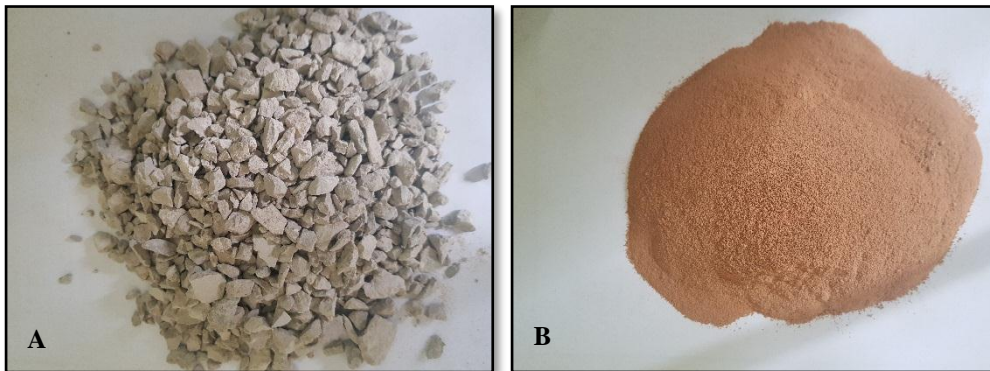


Fig.1. The Attapulgite , A: After crashing the rocks to small parts by hammer and before burning, and B : After burning and grinding.

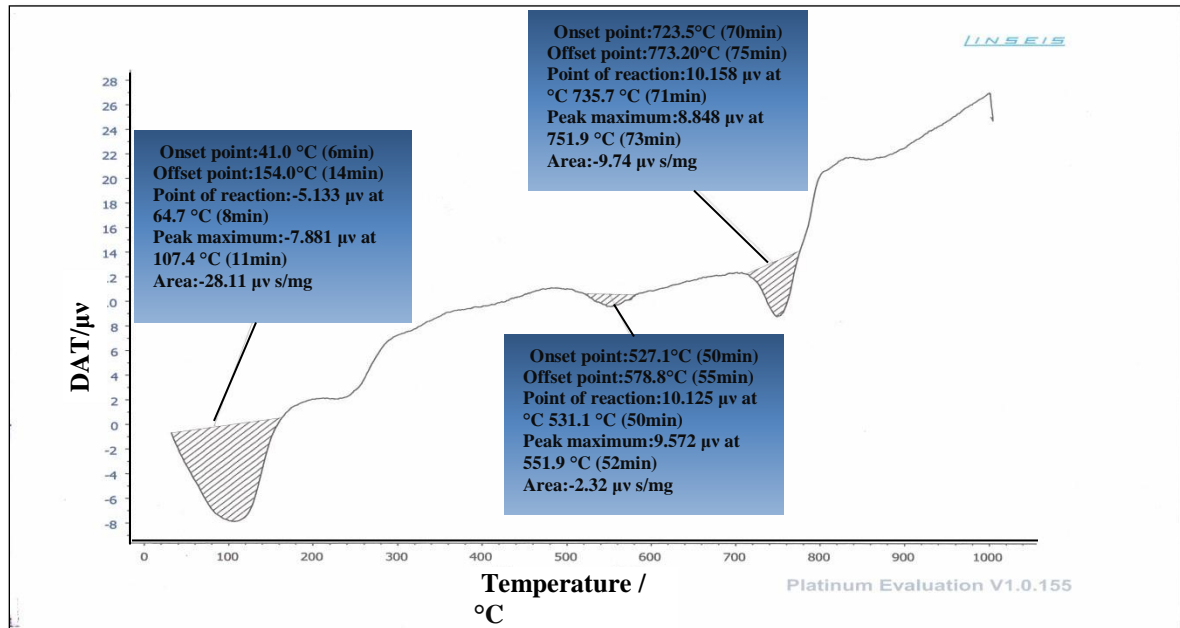


Fig.2. Thermal analysis of Attapulgite.

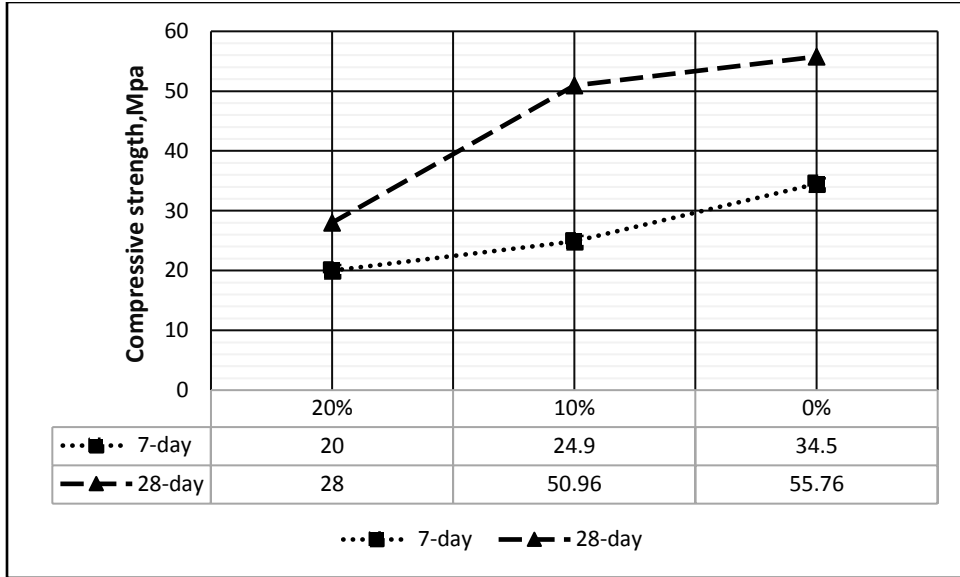


Fig.3. Compressive strength of samples with 0%,10% and 20% of Attapulgit HRA replacement.

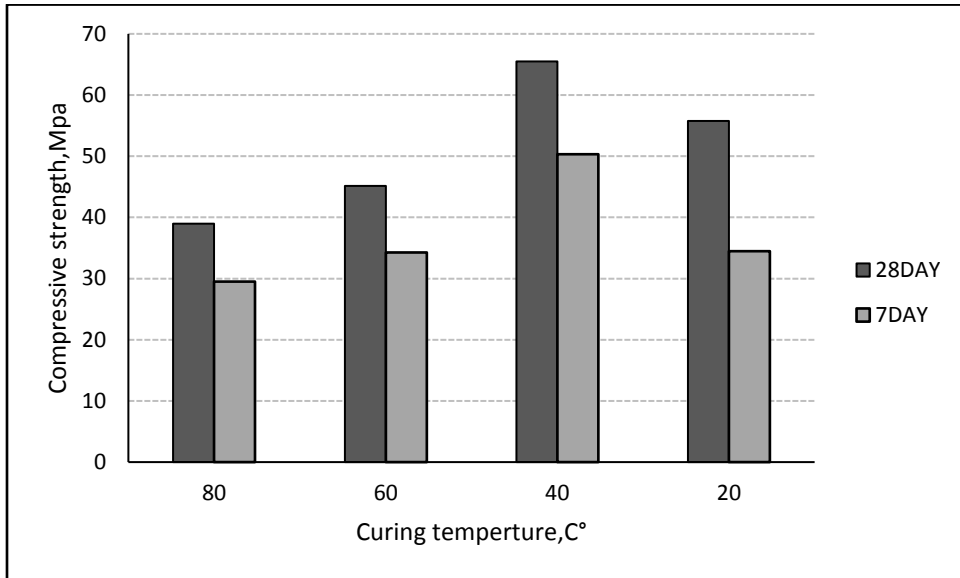


Fig.4. Compressive strength and curing temperature on samples without HRA.

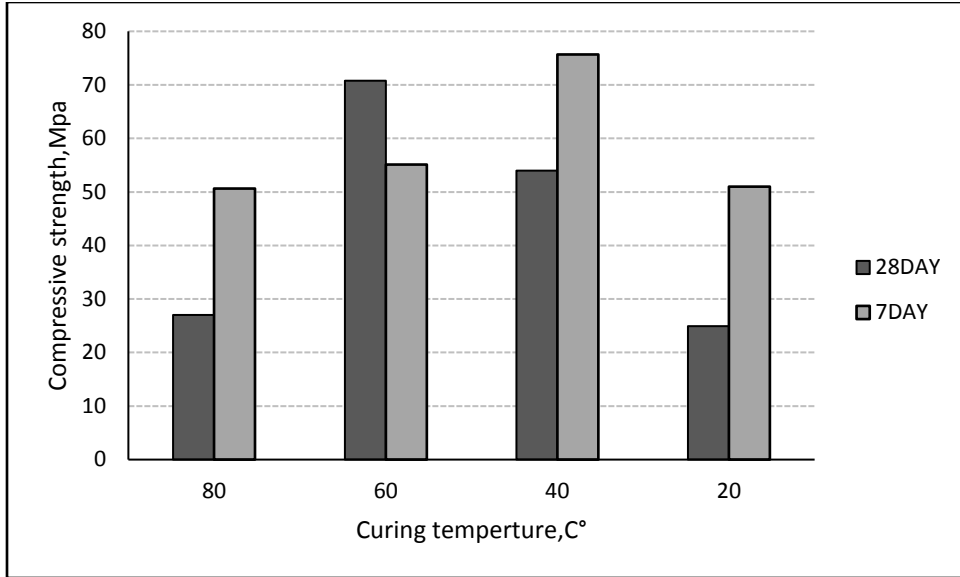


Fig. 5. Compressive strength and curing temperature on samples contain 10% HRA.

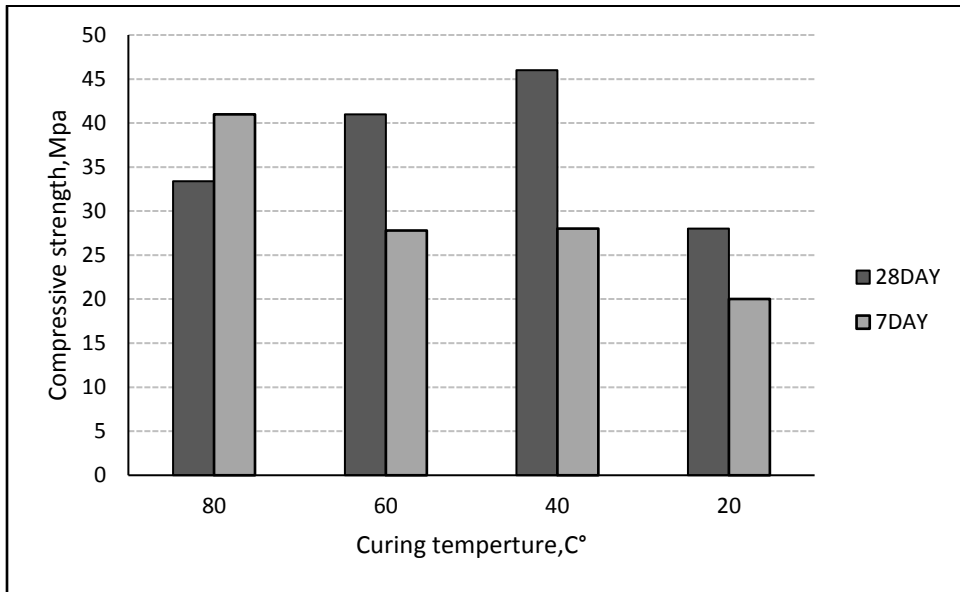


Fig.6. Compressive strength and curing temperature on samples contain 20% HRA.

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Table 1: Chemical composition properties of the cement.

Oxide composition	Percentage by weight	Limit of Iraqi Specification No. 5/1984[7]
Lime CaO	61.89	-
Silica SiO ₂	21.37	-
Alumina Al ₂ O ₃	4.60	-
Iron Oxide Fe ₂ O ₃	3.35	-
Sulphate SO ₃	2.42	≤ 2.8 %
Magnesia MgO	3.05	≤ 5.0 %
Potash K ₂ O	0.36	-
Soda Na ₂ O	0.27	-
Loss on ignition L.O.I.	2.16	≤ 4.0 %
Insoluble residue I.R.	0.60	≤ 1.5 %
Bogue's equations		
Tricalcium Silicate C ₃ S	46.88	-
Dicalcium Silicate C ₂ S	26.17	-
Tricalcium Aluminate C ₃ A	6.53	-
Tetracalcium Aluminate - Ferrite C ₄ AF	10.18	-

Table 2: Physical properties of the cement.

Physical properties	Test results	Limits of Iraqi Specification No. 5/1984
Specific surface area (Blaine method) (m ² / kg)	321	≥ 230
Soundness (Le – chatelier method)(mm)	0.5	< 10
Setting time (Vicat's method)	1:55	≥ 45 min
Initial setting (hrs : min)	2.24	≤ 10 hrs
Final setting (hrs : min)		
Compressive strength (MPa)	17.6	≥ 15
3 days	23.3	≥ 23
7 days		

Table 3: Chemical and physical properties Attapulgitte HRA, the limits of pozzolanic material class N.

Oxids	Content,%	Limits of ASTM C618
SiO ₂	45.87	
Fe ₂ O ₃	4.68	≥70
Al ₂ O ₃	19.47	
MgO	4.00	
L.O.I	12.35	
SO ₃	1.1	
Specific gravity	12.05	-

Table 4: Mix proportion of paste

Mix ID	Attapulgitte HRA,%	Cement, kg/m ³	Attapulgitte HRA,kg/m ³	Water/Cement
M0	0%	500	-	0.36
M10	10%	450	50	0.36
M20	20%	400	100	0.36

Table 5: Compressive strength results.

Mix ID	Attapulгите HRA, %	20C°		40C°		60C°		80C°	
		7- day	28-day	7- day	28-day	7- day	28-day	7- day	28-day
M0	0%	34.5	55.76	50.3	65.5	34.24	45.16	29.5	38.96
M10	10%	24.9	50.96	54	75.7	70.8	55.1	27	50.64
M20	20%	20	28	28	46	27.8	41	41	33.4