Thermal Effects on Compressive Strength of Local Limestone and Claystone

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Abstract–Limestone and claystone are widely used in rocky mountainous areas as building materials in Kurdistan region, in particular, the city of Koya. The outbreak of fire in buildings will have a great impact on strengths of building materials. The property performance of these local materials is understudied. This research investigates the impact of high temperature on the physicomechanical properties of limestone and claystone from Fatha Formation in Koya in Kurdistan region of Iraq. For this purpose, cores were taken from intact rocks; their ends were cut by a mechanical saw to obtain a cylindrical shape and immersed in water for 24 h, and then, subjected to physicomechanical tests of specific gravity, water absorption, porosity, and uniaxial compressive strength. For determining the residual compressive strength, the specimens were taken out from water, surface dried, and exposed to high temperatures of 450, and 650°C for 2 h using an electric oven. The results showed that claystone specimens show more stable mechanical properties than the limestone after exposure to high temperatures, and the high temperature causes lightening in color and significant cracks in both materials.

***Index Terms*—Compressive strength, Claystone, Limestone, Kurdistan Region, Thermal effects.**

I. Introduction

Limestone and claystone have many applications in the construction sector. Using these materials as building units in houses, bridges, tunnels, and the renovation of historic structures are some of these applications. The superior physicomechanical properties for these materials and their low cost qualify them for use in the mentioned applications.

Fire causes destructive damages to structures. Cracks, crusts, and spalling were observed in building stone when exposed to fire. It is considered that cracks refer to discontinuities formed by thermal gradients within the rock blocks during the fire (Koca, et al., 2006). Besides the undesired esthetic considerations, cracks may cause serious structural problems to the structures. It is important to investigate the effect of fire and high temperature on the building units, including natural stones. The walls of structures built by these materials must necessarily withstand high temperatures in the fire which causes serious deterioration in strength and stability (Hajpál, 2002).

Ozguven and Ozcelik (2013) investigated the effect of different degrees of temperatures (room temperature, 200°C, 400°C, 600°C, 800°C, and 1000°C) on marble and limestone using the aspects of change in color and whiteness, polish reception, daily physical changes, and pH. Ozguven and Ozcelik (2013) found that, when natural stones are heated to temperatures above 800°C their structure damages and/or changes, the material becomes pours and cracks occur. They also mentioned that natural stones that face this amount of heat under atmospheric conditions generally, crack, fragmentize, spall, and disperse. Sengun (2014) investigated the influence of temperatures ranging from 105°C to 600°C on the physical and mechanical properties of six carbonate rock samples (two marbles, two low-porous limestones, and two high-porous limestones). It was found that the values of bulk density, P-wave velocity, uniaxial compressive strength and modulus of elasticity, tensile strength, and shore hardness decreased to different extents, whereas apparent porosity increased under the influence of heat up to 600°C. The results indicated a maximum decrease of 62–82% in modulus of elasticity and the least reduction of 1.2–2.7% in bulk density of carbonate rocks. Moreover, the uniaxial compressive strength, tensile strength, P-wave velocity, and shore hardness values decreased by 27–51%, 28–75%, 36–69%, and 10–40%, respectively. Besides, increase in apparent porosity values of tested rocks with very high porosity was decreased, whereas the apparent porosity values of low-porous rocks increased up to 10 times of the initial value.

The calcinations of calcite from 800°C may constitute the main effect generated by fire, as both the transformation from

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calcite to calcium hydroxide and the subsequent hydration of calcium hydroxide involve important volume changes that may alter the internal structure of the stone.

$CaCO_3 + HEAT \rightarrow CaO + CO_2\uparrow$

$CaO + H_2O \rightarrow Ca(OH)_2 + HEAT$

The most catastrophic changes occurred in limestone cores, beginning to take place above 600°C due to calcination processes. This process has been previously reported as a result of high temperature in testing stones containing calcite (Török and Hajpál, 2005; Gomez-Heras, et al., 2006).

It is known that fire and high temperatures cause degradation of natural building stones. There are insufficient studies focusing on the effect of high temperature on physicomechanical properties of limestone and claystone building units. The aim of this research is to investigate the effect of high temperature on the limestone and claystone cores were taken from Fatha Formation in Koya/Iraqi Kurdistan region to show the rocks residual strength after the fire.

II. Geological Setting of the Study Area

Koya tectonic lies in the High Folded Zone of Unstable Shelf, and geographically, it is located in the North East (NE) of Iraq. Haibat Sultan anticline is considered as one of the main folds belongs to this zone which is double plunging and asymmetrical anticline. The fold is extending in the direction of North West–South East trends parallel to the Zagros Fault Thrust Zone. The Iraqi Shelf can be divided into five tectono-physiographic zones. The zones are characterized by varying degrees of Neogene structural deformations. Currently, it is generally bounded by major faults which represent deep-seated structural elements. These zones from NE to South West (SW) are as follows: The Thrust Zone, the Folded Zone, the Mesopotamian Basin, and Salman Zone and the Rutbah-Jezira Zone (Buday and Jassim, 1987; Jassim and Goff, 2006). The Fatha Formation is one of the most widespread and economically important formations in Iraqi Kurdistan region. It forms the continuous belt at the foot SW limb of Haibat Sultan Mountain in Koya area. It is from Middle Miocene age, comprises of anhydrite, gypsum, and salt interbedded with limestone and marl (Buday, 1980). The thickness of the formation is greatly variable. In the central parts of the basin, the thickness is up to 900 m, whereas the thickness of the formation in the studied area ranges between 60 and 200 m (Youkhana and Sissakian, 1986; Omer, 2009). The formation represents the deposits of a relatively strong sinking basin, which often had been separated from the open sea by rising ridges (Buday, 1980). It consists of cyclic deposits of claystone and limestone with gypsum beds in lower cycles. Claystone, reddish-brown in color, is of fine particles and highly fractured, which represents the main consistent of the formation (Omer, 2009). In the studied area, the formation comprises cycles of claystone, siltstone, and sandstone with gypsum and limestone bed. Claystones, reddish-brown in color, are fine to very fine grained, and limestone of white-to-grayish color is characterized by the development of joints and fractures. The bedded exist in moderate to well thick beds, which represents the main constituents of the formation. The locations of study samples are bounded by the Universal Transverse Mercator (UTM) grid 3995011 and 3995423 North, 0468319 and 0468973 East, as summarized in Table I and Fig. 1.

III. Materials and Methods

Eighteen outcrop samples of limestone and claystone were taken from three locations of Fatha Formation. The samples were cut by the use of core cutting machine. After cutting, the two circular surfaces of the specimens were straitened by an electrical saw. All the samples were cut to have a cylindrical shape of 7.5 cm in diameter and 14 cm height. The core samples were coded and immersed in a water tank for 24 h (Fig. 2).

To determine the effect of high temperature, the most common method is putting the samples in an oven (Hajpál and Török, 2004; Gomez-Heras, et al., 2009), and to explain the effect of temperatures, different degrees of temperature must be selected. The purpose of this operation is to clearly show what kind of changes occurs at different high temperatures. Samples were exposed to the heat separately starting from gradually 450°C and 650°C and then compared with reference samples at room temperature of 25°C. The oven was brought to the desired high temperature, and then, the samples were taken out from water tank, surface dried, and put in the oven for 2 h. After heating, the samples were taken out from the oven, cooled to room temperature, and then, subjected to compression test with the non-heated samples. The compressive strength tests were carried out according to the Standards American Society for Testing and Materials (2014).

IV. Physicomechanical Properties Results and Discussions

The average values of the specific gravity, water absorption, and porosity determined from the laboratory for limestone and claystone are 2.67, 0.281, and 0.75 and 2.36, 6.28, and 14.97, respectively, as shown in Table II. The results of uniaxial compressive strength and relative compressive strength for limestone and Claystone are shown in Figs. 3 and 4, respectively. The strength trend curves for the both materials before and after exposure to high temperatures are shown in Fig. 5. For relative compressive strength, the strength of non-heated limestone samples was taken as 100%, and the strength for other specimens was related to this strength. The

TABLE I
Locations of Outcrops Samples in the Study Area by Universal Transverse Mercator (UTM)

Location number	Formation	Coordinates (North and East line)
1	Fatha	3995325 N and 0468422 E
2	Fatha	3993775 N and 0469925 E
3	Fatha	3992230 N and 0469575 E

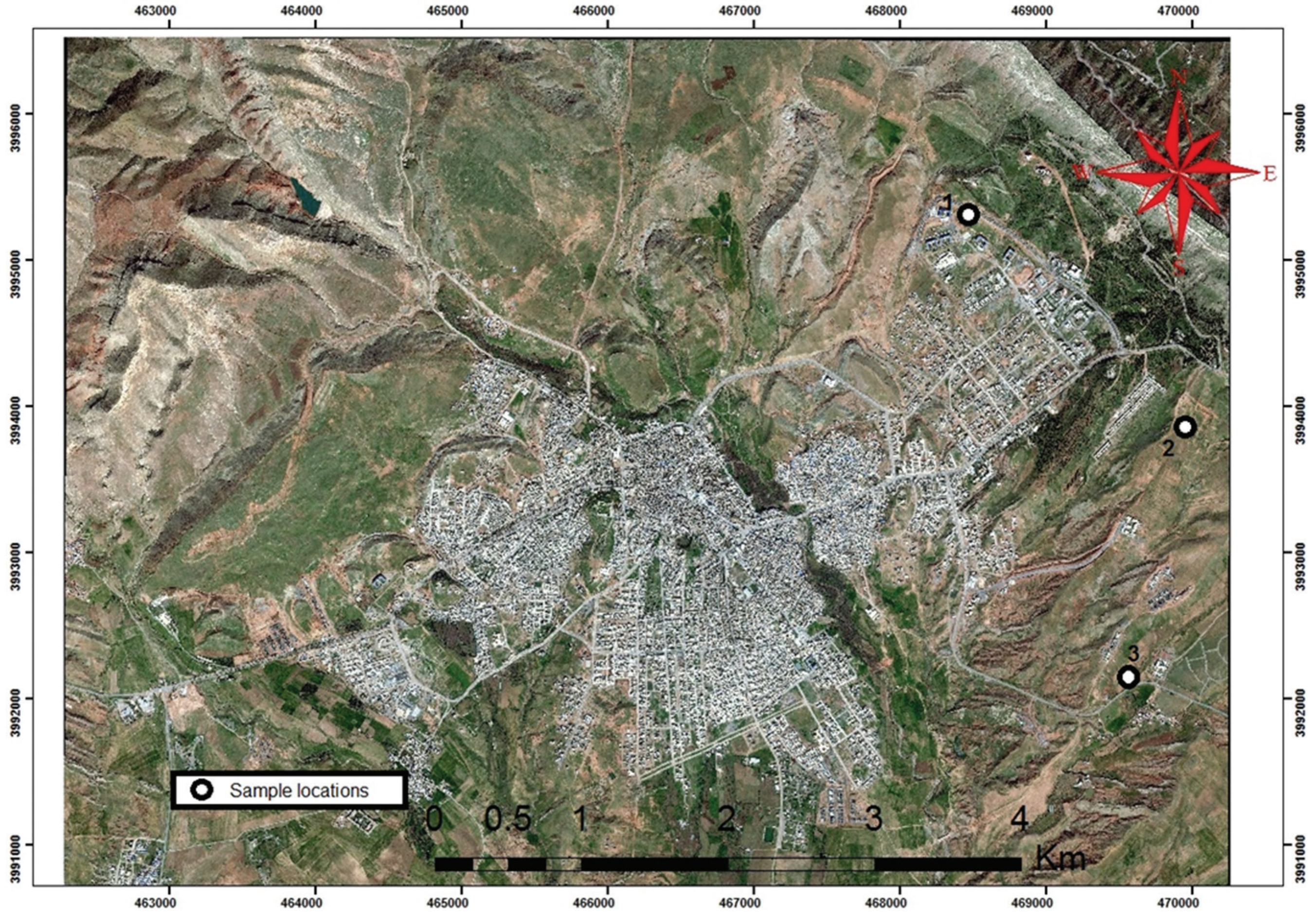

Fig. 1. Satellite Image of Koya City which Indicates the Sample Locations.

Fig. 2. Eighteen Different Core Samples Collection from Limestone and Claystone of Fatha Formation for Laboratory Tests.

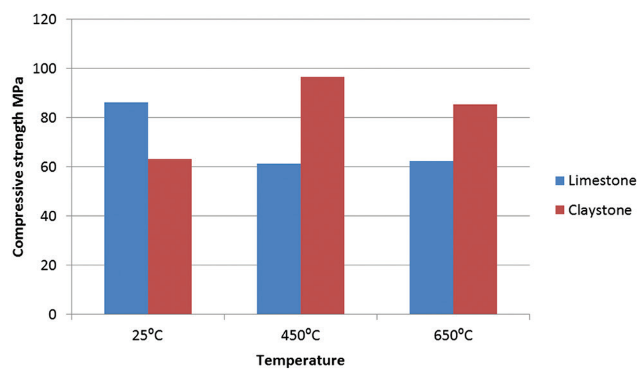

Fig. 3. Compressive Strength for Limestone and Claystone Cores Exposed to Different Temperatures.

average value of compressive strength for limestone ranges from 61.37 to 86.32 MPa, whereas for claystone, the value ranges from 63.17 to 96.6 MPa. The uniaxial compressive strength of limestone and claystone falls within the range of high strength according to New Zealand Geotechnical Society, 2005. Field description of soil and rock, New Zealand: Publication of NZ Geotechnical Society. Statistical models were generated to relate the mechanical properties with the physical properties.

A. Specific Gravity

The specific gravity of a rock is one of its basic properties. It is influenced primarily by the density of the minerals, their content, and amount of void space inside the rock (Bell and Lindsay, 1999). In general, the specific gravity is said to be high when its value exceeds 2.8, and it is usually considered as low when that value is <2.3. The specific gravity stated that the specimens did not show significant changes through laboratory tests. The results of the analyses revealed that the specific gravity displayed little variation and average range from 2.67 to 2.36 for limestone and claystone, respectively, this indicates that limestone has medium value, but claystone has a low value of specific gravity.

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TABLE II
RESULTS OF SPECIFIC GRAVITY, WATER ABSORPTION, AND POROSITY OF SAMPLES

Symbol number	Lithology	Saturated surface dry (g)	Weight in water (g)	Dry weight (g)	Specific gravity	Water absorption (W %)	Porosity (n %)
A1	Limestone	751	471	747	2.66	0.535	1.43
A2	Limestone	846	533	845	2.69	0.118	0.32
A3	Limestone	820	516	818	2.69	0.244	0.66
A4	Limestone	907	572	905	2.70	0.220	0.59
A5	Limestone	822	516	820	2.67	0.243	0.65
A6	Limestone	791	491	789	2.63	0.250	0.66
A7	Limestone	853	538	852	2.70	0.117	0.32
A8	Limestone	779	489	776	2.68	0.386	1.03
A9	Limestone	713	447	710	2.67	0.422	1.13
Average					2.67	0.281	0.75
B1	Claystone	736	447	697	2.41	5.595	13.50
B2	Claystone	840	500	783	2.30	7.279	16.76
B3	Claystone	740	451	703	2.43	5.263	12.80
B4	Claystone	749	450	706	2.36	6.090	14.38
B5	Claystone	646	390	606	2.36	6.600	15.63
B6	Claystone	626	376	585	2.34	7.008	16.40
B7	Claystone	721	433	676	2.34	6.656	15.63
B8	Claystone	694	420	654	2.38	6.116	14.59
B9	Claystone	734	441	693	2.36	5.916	15.02
Average					2.36	6.280	14.97

B. Effect of High Temperature on the Compressive Strength of Limestone

The compressive strength and relative compressive strength for limestone cores exposed to different temperatures are shown in Figs. 3 and 4, respectively. Exposing limestone cores to 450°C resulted in a significant decrease in compressive strength, the strength dropped from 86.32 to 61.37 MPa. The compressive strength of cores after exposure to 650°C was 62.27 MPa which remained approximately similar to that of 450°C. The exposure to high temperature resulted in approximately 30% strength reduction with respect to non-heated samples. This phenomenon is highly due to the transformation of calcium carbonate to lime CaO by the effect of high temperature as stated by Koca, et al., 2006. Moreover, the vapor pressure due to temperature rise inside the specimens causes micro and visible cracks which in turn reduce the compressive strength.

Limestone is consisting of calcite. Calcite is a mineral not much harder than a fingernail. Consequently, limestone is soft. It can easily be dissolved when it exposed to a temperature up to 400°C and changes to free lime as found by Yong and Thomas (1999) and Egger (2006).

C. Effect of High Temperature on the Compressive Strength of Claystone

The compressive strength and relative compressive strength results for claystone cores after exposure to high temperatures are shown in Figs. 3 and 4, respectively. The claystone specimens after exposure to 450°C exhibited a considerable increase in compressive strength. The strength rise was around 40%, and it rises from 63.17 MPa for non-heated specimens to 96.6 MPa for those exposed to 450°C. The compressive strength of 85.4 MPa was recorded for claystone cores after exposure to 650°C; this recorded value is around 13% less than the strength of specimens exposed to 450°C but greater than the none-heated specimens by nearly 25%.

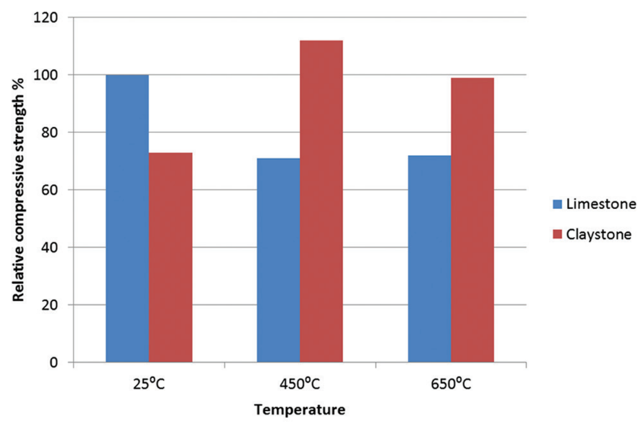

Fig. 4. Relative Compressive Strength for Limestone and Claystone Cores Exposed to Different Temperatures.

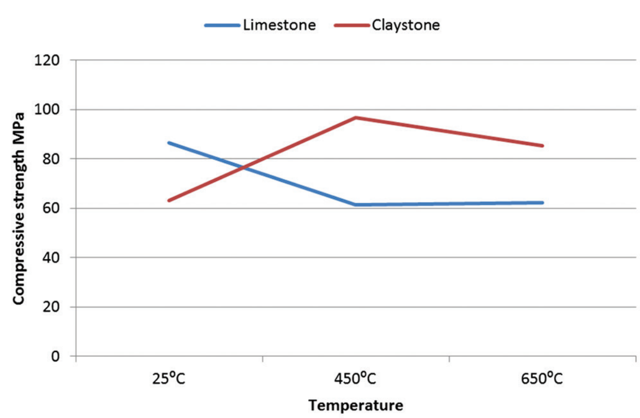

Fig. 5. The Strength Trend Curves for Limestone and Claystone before and after Exposure to High Temperature.

The increase of strength for claystone after exposure to high temperature is attributed to strengthening the bonds of silicate minerals in claystone when exposes to high temperatures. Although some visible cracks were detected on the specimens due to high temperature, the strength was higher than that of non-heated specimen. This phenomenon indicates how strong the bonds become between the silicate minerals after exposure to 650°C. Moreover, the mineralogical decomposition may not occur in claystone, and the high temperature made the particles to be packed together tightly, which by turn resulted in increasing the strength after exposure to high temperature.

D. Porosity Effect on the Compressive Strength

During the process of diagenesis, sediments undergo physical, chemical, mechanical, and mineralogical changes by increasing in temperature and pressure. This increase in temperature and pressure causes loose grained sediments that become tightly packed reducing porosity, essentially squeezing water out of the sediment. Porosity is further reduced by the precipitation of minerals into the remaining pore spaces as stated by Boggs, (2006). A comprehensive study of porosity can provide valuable information to determine whether a given type of natural stone is susceptible to thermal stresses or not (Malaga-Starzec, et al., 2006). The compressive strength results for the limestone and claystone cores after exposure to different temperatures as a function of porosity as shown in Table II.

From the porosity results, it can be observed that limestone samples showed lower porosity than claystone ones. This phenomenon is due to the susceptibility of high clay mineral in claystone for decomposition caused by moisture, an action that increases porosity for the rocks.

The exposure to high temperature causes the moisture in the samples to evaporate. This phenomenon produces a buildup vapor pressure in the samples and causes them to crack which in turn reduces compressive strength. Hence, the higher compressive strength that recorded for claystone than limestone after exposure to high temperature is due to the vapor release caused by the high porosity of the claystone which produces escape path for the vapor pressure to release.

E. Water Absorption at Atmospheric Pressure

Water absorption is a measure of the effective porosity of a stone. The total water absorption value under atmospheric pressure conditions indicates how much water a rock can absorb over 24 h when placed at 3–5 cm below the water level. The water uptake in relation to the dry weight of the sample is mainly influenced by porosity, pore size distribution, and the mineralogical composition of the rock. The water absorption is dependent on the clay minerals present in the rock material which indirectly affects the strength (Mohamad, et al., 2013). Due to the dominance of quartz and clay mineral, the specific gravity of claystone is about 2.36, whereas calcitic limestone rocks have a specific gravity of 2.67. The claystone shows initially over a short period of time very fast water absorption; this can be explained by the information provided by its pore size distribution, that is, the range of pore sizes that allow water to access the open porosity (14.97%). The limestone shows long period of time with slow water absorption to access the ineffective porosity (0.75%).

The results values were obtained are 0.281% and 6.28% for limestone and claystone, respectively. Water absorption values increase in direct proportion with exposure to the temperature of natural stones. Temperature variation and water absorption are parallel characteristics of limestone and claystone samples. This feature, physically, enables to determine the cracks of natural stones.

F. Optical Detections

The exposure to high temperatures caused obvious cracks in both limestone and claystone samples (Fig. 6). Limestone samples were more susceptible for cracking than the claystone ones. By increasing the exposure temperature, the color of the samples became lighter. This phenomenon is highly due to losing moisture and the decomposition of materials at high temperatures.

Fig. 7 shows the limestone sample after exposure to a high temperature of 650°C. From the figure, the decomposition of calcite to free lime can be concluded.

Fig. 6. Cracks in Limestone (a) and Claystone (b) Generated due to High Temperature.

Fig. 7. Limestone Core Sample show Deformation due to Exposure to 650°C.

V. Conclusions

The following conclusions can be drawn from this research:

1. Limestone loses up to 30% of its strength after exposure to high temperatures of 450°C and 650°C.
2. Claystone shows up to 40% strength increase after exposure to 450°C, whereas around 25% increase in strength was recorded after exposure to 650°C.
3. Exposure to high temperatures results in the occurrence of significant cracks and lightening in color for both limestone and claystone building units. Limestone is more susceptible for cracking than the claystone.
4. The strength reduction for limestone after exposure to 450°C is highly due to its low porosity which in turn leads in producing excessive builds up vapor pressure in the samples, whereas in the Claystone samples, the vapor pressure can be released attributable to the high porosity for the rocks.

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