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Potential use of Algerian natural diatomite as pozzolanic and insulator material in cement mortar

Bouksani Omar*[1], Abba Fatiha[1], Belkacemi Marwa[1], Kharchi Fattoum[2], Benhadji Maissan[3] and Belhamel Farid[4]

[1]Faculty of Technology, University Yahia Fares of Medea, Algeria
[2]Built Environment Research Laboratory (LBE), Civil Engineering Faculty (USTHB), Algeria
[3]University Saad Dahlab, Blida, Algeria
[4]National Center of Studies and Integrated Research on Building Engineering (CNERIB), Algeria

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ABSTRACT

The aim of this article is to valorize the use of Algerian diatomite as natural pozzolan and insulator material in mortars manufacturing. The diatomite was characterized using X-ray fluorescence, X-ray diffraction, and SEM analysis. The pozzolanic activity of the diatomite was assessed by strength activity index according to ASTM C618 standard specification. The mechanical strength and thermal conductivity were investigated on mortar mixtures prepared by replacing cement by 10, 20, 30 and 40% of diatomite. The experimental results indicated that: X-ray fluorescence, X-ray diffraction and pozzolanic activity index tests confirmed the suitability to use the Algerian diatomite as natural pozzolans. The compressive and flexural strengths of mortars containing up to 20% diatomite decrease slightly compared than mortar without diatomite. Finally, the decreasing of thermal conductivity indicates an improvement on thermal insulation of diatomite mortars.

Key words: *Diatomite, Pozzolan, Strength activity index, Compressive strength, Thermal conductivity.*

Introduction

The world cement industry is responsible for about 7-8% amount of the global carbon dioxide emissions (Olivier *et al.*, 2016). Many minerals have identified as materials capable to reduce these emissions. Diatomite is one among them which can be used as additive in clinker fabrication or as a partial replacement of cement in mortars or concretes.

Diatomite is a sedimentary rock formed by the accumulation of skeletal remains of diatoms. It is lightweight and friable material. Diatomite possesses porous microstructure and presents low thermal conductivity. Moreover, the diatomite it is characterized by a significant content of silica, high specific surface area and by dominant amorphous phase. Used as partial replacement of cement, diatomite can act by two manners. First, due to its high fineness, diatomite acts by a physical effect by filling the voids between the cement particles which reduce the porosity of the material. Second, diatomite can act by chemical effect through its pozzolanic reaction, which occurs between the amorphous silica and portlandite to form new C-S-H gels, which fill the existing pores. Many researchers have investigated the effect of partial replacement of cement by diatomite in pastes, mortars, and concretes. It was observed contradictory results on mechanical strengths, which depend on the diatomite content and its properties. On the one hand, a significant increase in compressive strength has been found (Stamatakis, *et al*., 2003; Macedo, *et al*., 2020). In cement pastes, Stamatakis *et al*. (2003) indicated that 15% diatomite increase the compressive strength by 19.6%, whereas, Fragoulis *et al.* (1997) reported that 10% diatomite increase the strength by 11.6%. They related this improvement to the substantial amount of silicon dioxide contained in diatomite, its high specific surface area and particularly to its rich content of amorphous silica (or rich content of reactive silica).

In mortar, Pokony *et al*. (2019) have found that 20% diatomite can developed the compressive strength of mortar by 14%. In concrete, Pokony *et al*. (2016) and Macedo *et al*. (2020) have found that the concrete containing until 10% diatomite has a higher compressive strength than concrete without diatomite. On the other hand, significant reduce in compressive strength has been reported. This was related to the high water absorption capacity of diatomite which increases the water to binder ratio, as reported on pastes (Fragoulis, *et al.,* 2005; Zahalkova *et al*., 2016; Stanek, 2016).

In addition and despite the high amount of silica, it was found that compressive strength of mortar decreased when the diatomite content increases. Yilmaz *et al*. (2008); Rovnakova *et al*. (2015) found serious decrease in compressive strength for 20% diatomite, while Saidi *et al*. (2020) relate same decreases for 16% diatomite. Similar tendency in reduce of strength was also observed in mortars, particularly when diatomite contained less of silica (Abrão *et al.,* 2019) or when the used diatomite has a coarse particle (Degirmenci and Yilmaz, 2007).

In contrast, some studies have not registered a significant variation in mechanical strengths, when water demand was no excessive. Stamatakis *et al*. (2003) have observed a slight increase in compressive strength for pastes with 20% diatomite, whereas Kastis *et al*. (2006) reported a slight decrease, for pastes with 10% diatomite. They attributed this to the low amounts of silicon dioxide and reactive silica of used diatomite. Also, Yilmaz *et al*. (2008) indicate that the compressive strength of mortar containing 10% diatomite was comparable to the control mortar. This result was attributed to the some crystalline phases of diatomite, despite its high amount of silica. It appears from the literature that the mechanical strengths varied according to the properties of the diatomite and that the thermal properties and the pozzolanic activity are rarely studied.

This article aims to explore the Algerian diatomite, available in mascara region with reserves of 6 million tons (Sonarem 1979) to be used as partial replacement of cement in mortars. Chemical composition, mineralogical characteristics and microstructure morphology of diatomite are identified respectively by XRF, XRD and MEB analysis. As well as, the pozzolanic activity of diatomite is investigated using the strength activity index. After that, this paper describes the mechanical strengths and thermal conductivity of mortars with different amount of diatomite.

Experimental Details

Materials

In this study, diatomite obtained from Algerian northwest part near Mascara state (Fig. 1) and a Portland cement type CEM I 42.5 R were the cementitious materials used. The diatomite was heated at 200 °C, and it was milled to obtain particle size less of 80 μm. Details of chemical and physical properties of cement are given in Table 1. Standard sand and distilled water were used to prepared mortars.

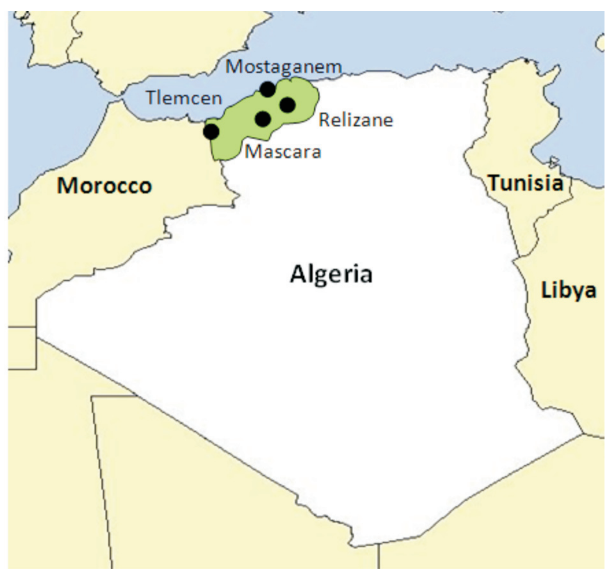

Fig. 1. Geographical *location of diatomite quarry in Algeria*

Experimental program

Characterization of diatomite

The characterization of the diatomite included

Table 1. Chemical and physical properties of cement CEM I 42.5 R

Oxydes (%)		Mineralogical composition	(%)
SiO_2	20.85	C_3S	61.5
CaO	63.13	C_2S	14.5
Al_2O_3	4.78	C_3A	4.1
Fe_2O_3	4.61	C_4AF	13
SO_3	2.63	***Physical characteristics***	
MgO	1.11	Specific gravity (g/cm^3)	3.1
K_2O	0.55	Blaine fineness (cm^2/g)	3250
Na_2O	0.13	Initial setting time (min)	140

physical, chemical, mineralogical and morphological analyses. Chemical composition was obtained by X-ray Fluorescence (XRF), type Rigaku ZSX Primus II. Amorphous and crystalline phases were identified by mineralogical composition using X-ray diffraction (XRD). Morphological analysis to obtain article shape and size was performed with Environmental Scanning-electron Microscope (eSEM-FEI Quanta 250).

Characterization of mortars

The characterization of mortars containing diatomite was carried out in two steps. The first intends to investigate the pozzolanic activity of diatomite by quantifying its strength activity index according to ASTM C618 (2015) standard. From this standard, the strength activity index represents the ratio of the compressive strength of mortar containing 20% diatomite to the control mortar without diatomite (at 7 and 28 days).

The second step consists to assess the effect of partial replacement of cement with diatomite on mechanical strengths and thermal conductivity of mortars. Mechanical strengths were tested at the age of 3, 7, 14, 28 and 90 days, by determination of flexural and compressive strengths according to EN 196-1 (2005). Thermal conductivity was performed by parallel hot-wire method, according EN 993-15 (2005).

Mortars compositions

In the first step, the strength activity index was studied by preparing control mortar and mortar containing diatomite with the same workability. Control mortar was prepared by using 450 g Portland cement (CEM I 42.5R), 1350 g standard sand and 225 g water. Mortars with diatomite were prepared by 1350 g sand and the cement was replaced with 20%. For the mixing water, it is not pre-defined, but it was added progressively until obtained workability similar to that of control mortar.

In the second step, the mechanical strength and thermal conductivity were undertaken by preparing five mixtures (Table 2). These include one control mortar mixture prepared without diatomite. The remaining four mixtures were prepared by replacing cement by 10, 20, 30 and 40% of diatomite. All of these mortars were manufactured with a sand/binder ratio of 3. Whereas, the water amounts of mortars containing diatomite were adjusted to achieve an equivalent workability of the control mortar.

Mortar mixing sequences and casting

Different mixing sequences were carried out. Control mortar without diatomite was mixed according EN 196-1 [18]. Mortars used for the mechanical strengths and thermal conductivity were prepared with variable water to binder ratio. The mixing procedures for these mortars meet the following sequences: cement and diatomite were first dry mixed for 1 minute in a five-Litre mortar mixer. Then, the sand was added gradually over 1min period. Once, the dry mixture became homogeneous, water was added progressively and mixed until obtained workability similar to that of control mortar.

Once the mixing sequence completed, the fresh mortar mixtures were cast in the 40x40x160 mm prismatic molds. Sealed with humid jute sheets for 24 h at 20 °C, the specimens were demolded and cured in water at 23 ± 2 °C, until the testing day.

Table 2. Mortar Mixture proportions for mechanical and thermal properties

Designation	Materials (g)				W/B
	Diatomite	Cement	Water	Sand	
DT 0	0	450	225	1350	0.5
DT 10	45	405	236.7		0.53
DT 20	90	360	252		0.56
DT 30	135	315	288		0.64
DT 40	179	271	501.2		0.67

Results and Discussion

Diatomite Characterization

Physical properties and chemical composition of diatomite are presented in Table 4. From the physical properties, the soft, friable aspect and the bulk density of 0.7 g/cm^3 indicate the diatomite as a light weight material. The retained on 45 µm sieve of 10.5% indicate a high degree of fineness. In fact, the Blaine fineness and BET surface area of the diatomite are 5876 cm^2/g and 19m^2/g respectively. From the Chemical composition, the diatomite has a high amount of silicon dioxide (SiO_2) estimated at 77.9% and few quantities (5.67%) of calcite ($CaCO_3$).

Mineralogical and morphological properties of diatomite are shown by XRD pattern (Fig. 2) and SEM (Fig. 3) respectively. From Figure 2, it can be seen that the mineralogy of diatomite has a dominant amorphous silica phase with two crystalline peaks related to quartz and calcite (Fig. 2). From Figure 3, it is obviously that the morphology of diatomite exhibit highly porous structure with different types of morphologies. There are essentially tubular skeletons, disks, rods with various diameters, from 38 to 80 µm. Its skeletons present a network of almost regular circular or hexagonal micropores 240 nm to 1um in diameter.

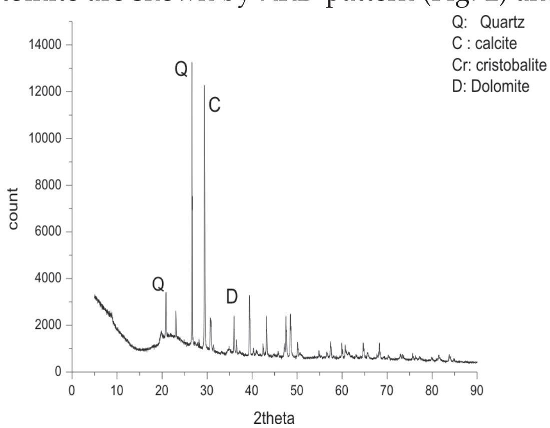

Fig. 2. XRD pattern of diatomite

Pozzolanic activity assessing

The pozzolanic activity of diatomite was assessed by comparing its chemical and physical composition and its strength activity index (SAI) with ASTM C618 standard:

Chemical and physical requirement compositions

Table 5 summarizes the comparison between the chemical composition and physical properties of diatomite with the specifications of ASTM C618 standard. It is found that the diatomite has the chemical properties of natural pozzolanic material. Indeed, the sum content of acidic oxides (silica+ alumina+ iron) reaching the value of 82.64% is more than the minimum requirement of 70% prescribed in ASTM C618 standard. Also, the percentages of Sulfur trioxide (SO_3), Alkalis and the loss on ignition

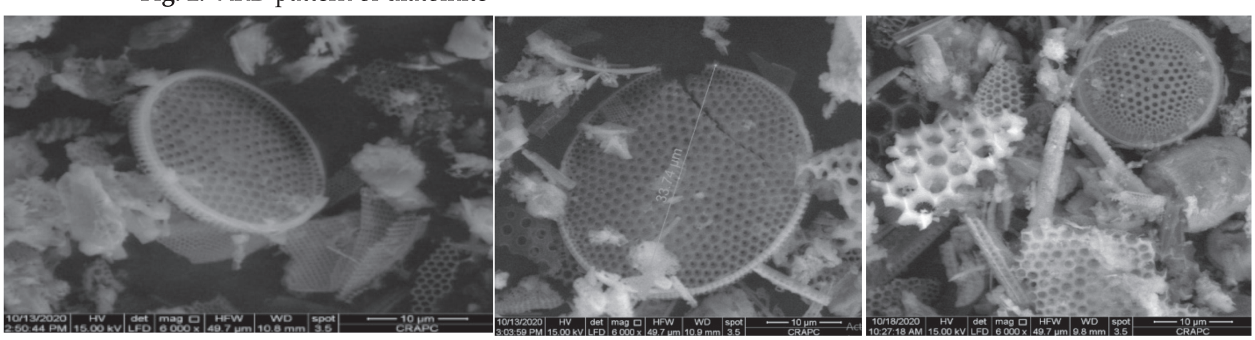

Fig. 3. Scanning electron microscope (SEM) image of diatomite

Table 4. Chemical composition and Physical properties of diatomite

Oxydes (%)		Physical properties	
SiO_2	77.9	Color	White
CaO	5.67	Aspect	Friable
Al_2O_3	3.45	PH	7.8
Fe_2O_3	1.29	Specific gravity (g/cm^3)	2.2
SO_3	0.64	Bulk Density (g/cm^3)	0.7
TiO_2	0.2	Blaine fineness (cm^2/g)	5876

(LOI) are well less than the maximum requirements as specified by these standards.

It can also be seen that the diatomite meets the physical properties of natural pozzolanic materials, since it is characterized by high amount of fine particles. It is found 10.5% particles retained on 45ìm sieve, which is less than the 34% limit defined by ASTM C618. Furthermore, the water requirement for diatomite mortar measured as 112% of control mortar is less than the 115% limit of ASTM C618 prescribed for natural pozzolans.

Strength activity index of diatomite

Figure 4 shows the strength activity index (SAI) of diatomite. It can be concluded that the diatomite satisfied the strength activity requirements for natural pozzolan (Class N), since its strength activity index values of 78.4 and 81.4% at 7 and 28 days, respectively are more than the limit value of 75% prescribed by ASTM C618.

Effect of diatomite on Compressive Strength

The compressive strength development of mortars containing various amount of diatomite is shown in Fig. 5. As compared to the mortar without diatomite, the strength decreases slightly, when diatomite replaces cement until 20%. For example, the 28-day compressive strength of mortars with 10 and 20% diatomite replacement reaches 40.9 and 38.2 MPa, respectively, while the mortar without diatomite achieved 44.7 MPa. This decrease in strength results from the supplementary mixing water required to achieve the same workability of mortar without diatomite.

However, increasing diatomite content more than 30% exhibited a sudden drop in compressive strength. This drop in strength can be attributed to the low cement content. Moreover, this can be attributed to the high water demand due to the porous structure of diatomite as shown by SEM in Fig. 3 and to its high specific surface area.

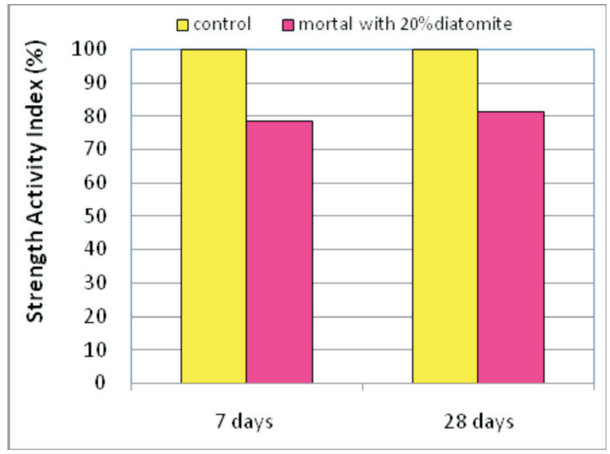

Fig. 4. Strength activity index of diatomite

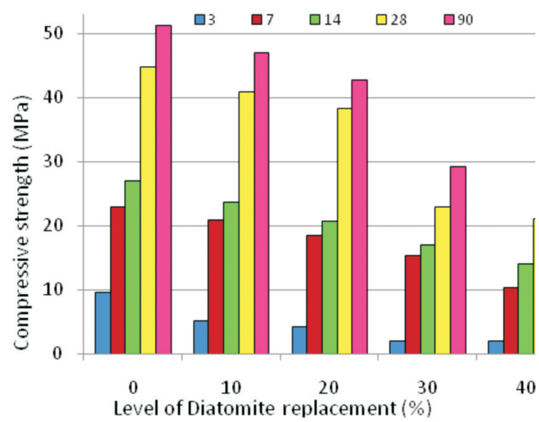

Fig. 5. Compressive strength development of mortars containing various amount of diatomite

Globally and based on the 28-day compressive strength, it is observed that replacing cement by 30 and 40% diatomite can achieve the strength of 23

Table 5. Chemical and Physical properties of diatomite with ASTM C618 Class N

Chemical Composition		Limit requirementsASTM C618 class N
SiO_2 + Al_2O_3 + Fe_2O_3	82.64	Minimum of 70%
SO_3	0.64	Maximum of 4%
Na_2O	1.23	Maximum of 1.5%
Na_2Oeq	1.67	————
Loss of ignition	4.24	Maximum of 10%
MgO	1.88	———-
P_2O_5	0.15	————
Physical properties		
Retained on 45 µm sieve (%)	10.5	Maximum of 34%
Requirements water (%)	112	115%

and 21 Mpa respectively. Indeed, the mortar with 40% diatomite can be considered as masonry mortar, since its compressive strength exhibits comparable with the minimum value (17.2 MPa) required by the ASTM C270 (2001).

Effect of diatomite on Flexural Strength

The development of flexural strength of mortars containing various amount of diatomite is shown in Figure 6. A Similar trend to the compressive strength was observed for the flexural strength. It can be seen that the flexural strength decreased progressively as the replacement level of diatomite increased until 20%. Whereas, if replacement level of diatomite is more than 30%, the flexural strength decreases considerably.

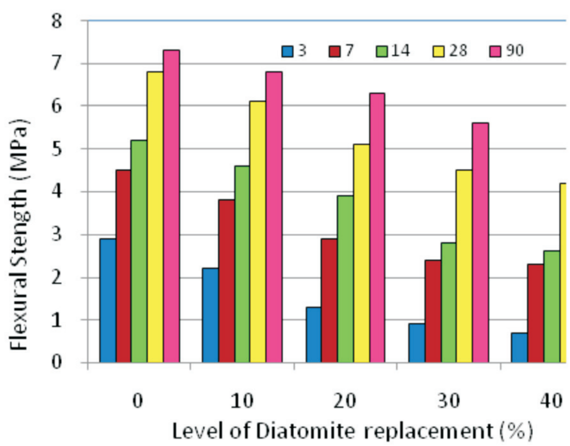

Fig. 6. Flexural strength development of mortars containing various amount of diatomite

Effect of diatomite on Density

Density of mortars containing different amount of diatomite, measured at 28 days, is presented in Fig. 8. Measurement revealed that the density of mortar decreases with increasing diatomite content, especially above 20% diatomite. It is observed that the density of control mortar was 2100 kg/m^3, while those containing 30 and 40% diatomite were 1944 and 1892 Kg/m^3, respectively.

This decrease can be attributed to the low specific gravity of diatomite (2.2 g/cm^3 given in Table 4) compared to Portland cement (3.1 g/cm^3) which leads to reduce the mass per unit volume of the mortar.

Effect of diatomite on Thermal conductivity

Thermal conductivities of mortars with and without

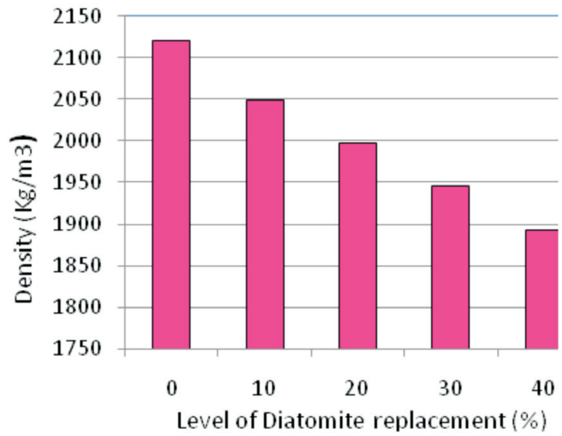

Fig. 8. Density development of mortars containing various amount of diatomite

diatomite are presented in Fig. 9. Results illustrated that the thermal conductivity significantly decreased with increasing content of diatomite. Thermal conductivity of mortars without diatomite is 1.63 W/m.K, whereas mortars containing 20, 30, and 40% diatomite, their thermal conductivity reach 0.58, 0.65 and 0.46 W/m.K, respectively. This decrease in thermal conductivity means that the diatomite tends to improve the thermal insulation due its porous structure as shown through the scanning electron microscope (SEM) images in Fig. 3.

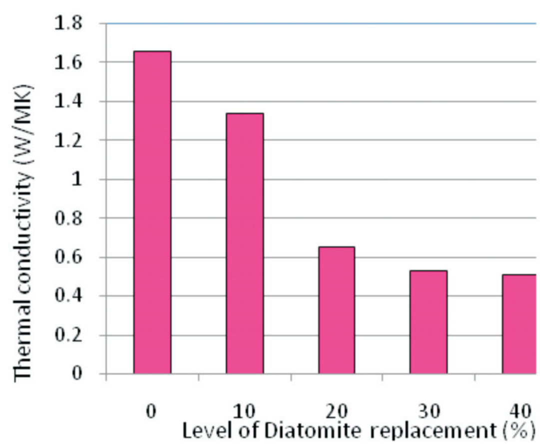

Fig. 9. Thermal conductivity of mortars with various level of diatomite replacement

Conclusion

This paper investigated the physical, chemical, mineralogical and morphological characteristics of Algerian diatomite. Mechanical strength, density and thermal conductivity of mortars containing diatomite as replacement of cement were also presented.

The following conclusions can be drawn:

-From the characteristics results, Algerian diatomite can be suitable as a mineral additive material. Since it is characterized by a high silica content (SiO_2=77.9%), a high specific surface area and a predominant amorphous phase. Furthermore, diatomite can be considered as natural pozzolanic material, since its strength activity index is compliant with ASTM C618 recommendation.

- From the mechanical strengths, a slight decrease in compressive strength is observed for mortars containing up to 20% diatomite compared to the mortar without diatomite. However, the compressive strength drops significantly when the diatomite content increases more than 30%. Whereas these mortars with low strengths can be considering as masonry mortars, according ASTM C270 standard.

- From the density and the thermal conductivity results, a high content of diatomite provides a lightweight mortar with a good thermal insulation.

Through the soft, friable aspect and the pozzolanic properties of diatomite, the experimental results suggest that the diatomite can be used as partial replacement of cement. This contributes to produce less quantity of cement which reduces both the impact on the environment and the cost of cement fabrication.

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