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Effect of partial substitution of clinker by pure limestone: Study of its influence on the physical-chemical properties and the mechanical performance

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Abstract

The aim of this work is to valorize a natural mineral source such as the pure limestone (P.Lime) of Bani Qais-Amran - Yemen, by introducing it in the production of cement to achieve the following objectives:

- Minimize the emission of CO_2 into the atmosphere which is the main cause of the greenhouse effect;
- Develop a new durable hydraulic Binder;
- Improve physical-chemical of cements as well as the mechanical performance of concrete;
- Gain a percentage of energy and raw materials consumed.

We have prepared the new durable cements from the clinker and the P.Lime. Partially substituting the clinker by this natural material in powder form which was finely at different percentages ranging from 0 to 40% by weight of the clinker with a pitch of 5%. The influence of the incorporation of the P.Lime in cement on the physical-chemical characteristics of cement such as the specific surface area, the density was studied. The effect of addition of P.Lime in cement on the physical properties of fresh cement paste was also studied to know the setting time and the water content. Also, the influence of the addition of the P.Lime on the compressive strength of mechanical of cement at hardened state has been studied.The obtained results by the different physical-chemical analysis showed that the addition of P.Lime in cement increased the fineness by the specific surface. In more, the density decreases. We have remarked that, the setting time decreases when the percentage of the P.Lime is increased. Of the same, the compressive strengths of mechanical at 2, 7 and 28 days have improved with the increase the amount of P.Lime.

Keywords: : New Hydraulic binders (NHB), pure limestone (P.Lime), durable cement, physical properties, compressive strengths.

1. INTRODUCTION

In the Yemen, there are many mineral materials and natural rocks in a large quantity and good quality. Among these materials, we find the natural Pozzolan (PN), the pure sandstones (PS) and the pure limestone (P.Lime). This last one is located in several regions in Yemen, such as, Hadramout, Mahra, Sana'a, Amran, Etc. [1-5]. According to statistical estimates of the Ministry of oil and minerals in the Yemen, these regions contain reserves of about 3.6 billion m^3 of P.Lime [1]. Depending on the results of the chemical analysis of P.Lime, which showed that the extent of its purity, where the percentage of calcium oxide ranging from 51.50 to 55.60%, silica going from 0.03 to 4.28%, and the iron between 0.02 to 0.72% [1]. As the physical analysis has shown that the absolute density of P.Lime ranging from 2.4 to 2.7 g/cm^3, the degree of whiteness between 81.70 to 95.15% and the specific surface area between 2685 cm^2/g to 4488 c cm^2/g [1]. This gives the material the possibility of being used in many industrial applications, such as the agriculture which acts directly on soil like a fertilizer fertility [6] and the construction applications that are the ceramics, the glass, the steel, the metals, the rubber, the plastics and other chemical industries. The pure limestone is used as adding in the production of cement and various types of the concrete by partially substituting the clinker with this will reducing the energy and the raw materials consumed during the production of clinker on one hand and minimize the emissions of carbon dioxide into the atmosphere that the main cause of the greenhouse on the other hand. The incorporation of mineral additions P.Lime remains until now an important technique for the improvement of the properties of the concrete, such as the fluidity, the strength, the durability, [7-10] Etc. These mineral addition effects are significantly the theology of cementitious materials to fresh state, which is directly connected with the development of resistance [11,12], sustainability of materials hardened [13-15]. Many works have studied the influence of fillers of the P. Lime as an aggregate on the mechanical performance of concrete [16-20]. In this work, we have achieved in the laboratory of cement and quality control of Amran cement plant (Yemen) in collaboration with the laboratory of Agro resources polymers and process engineering of the faculty of science, Ibn Tofail University (Kenitra-Morocco), we study the possibility of developing a new durable hydraulic binder at the base of the pure limestone (P.Lime) of Bani Qais-Amran (Yemen). While substituting partially the clinker by this natural material in powder form and finely crush at different percentages ranging from 0% to 40% in weight of clinker with a step of 5%. We studied the influence of P.Lime on the physical-chemical characteristics of cement such as (the fineness by specific surface area (Blaine) and the density). We, also, studied the effect of addition of P.Lime in the physical properties of fresh cement paste to know the setting time and the moisture on one hand. The influence of the addition of the P.Lime on the mechanical resistance in the hardened state was studied on the other hand.

2. MATERIALS AND METHODS

2.1. Materials

Before the evaluate the effect of the addition of pure limestone (P.Lime), from the field of Bani Qais-Amran in Yemen on the physical chemical characteristics of cement and the mechanical properties of mortar and/or concrete in the fresh and hardened state, we proceed studying the characteristics of the materials used in our work to understand the phenomena that occur during the hydration of the mixture.

2.1.1. Cement

The type of cement, used in this work, is (CMI / 42.5) from the factory of AMRAN in Yemen with 95% of clinker and 5% of gypsum. The chemical composition determined by X-Ray Fluorescence (XRF), mineralogical and the physical properties is legendary in tables (1), (2) and (3):

Table 1. Elementary chemical compositions of clinker, gypsum and cement

Content (%)	CaO	SiO_2	Al_2O_3	Fe_2O_3	Mgo	SO_3	K_2O	Na_2O	CL
Clinker	62.76	21	5.84	3	1.96	0.9	1.21	0.2	0.02
Gypsum	33.4	0.7	0.36	0.09	0.63	47.2	0.03	0.1	0.01
Cement	61.29	19.99	5.57	2.85	1.89	3.22	1.15	0.2	0.02

Table 2. Mineralogical composition of clinker

Chemical Name	Chemical Formula	Oxide Formula	Cement Notation	Mineral Name	Content(%)
Tricalcium Silicate	Ca_3SiO_5	$3CaO.SiO_2$	C_3S	Alite	47.7
Dicalcium Silicate	Ca_2SiO_4	$2CaO.SiO_2$	C_2S	Belite	25.1
TricalciumAluminate	$Ca_3Al_2O_6$	$3CaO.Al_2O_3$	C_3A	Aluminate	10.4
TetracalciumAluminoferrite	Ca_2AlFeO_5	$4CaO.Al_2O_3.Fe_2O_3$	C_4AF	Ferrite	9.1

Table 3. Physical properties of the Clinker and cement

	Physical properties	Units	Values
Clinker	Specific surface area	cm^2/g	3360
	Density	g/cm^3	3.17
Cement	Specific surface area	cm^2/g	3240
	Density	g/cm^3	3.14

2.1.2. Le Chatelier apparatus:

These apparatus allows to measure the density of cement by moving an inert liquid with cement within a graduated container according to the specification of the norm EN196-3.

2.1.3. Devise Blaine:

This device allows measuring the fineness of cement by the method of permeability (specific Blaine Surface), according to the norm10.1.005 NM.

2.1.4. Standard mixer:

It’s a device with a 5 liter of capacity and a blade of mixing that can run at 2 speeds (say slow, quick 285RP/M and 140RP/M) which allow preparing the mixture of paste of cement and mortar of good homogeneity conferring to the specification of the standard EN 196-1 [21].

2.1.5. The device of VICAT automatic and manual:

These are devices that help to determine the optimum amount of water using the standardized consistency and measurement start and stop times using a 40 mm in height truncated mold and a sliding rod equipped at its end with a needle 1.13 mm in diameter, according to the specification of the standard EN196-2 [22].

2.1.6. Compression testing machine:

It’s an automatic hydraulic press that allows determining the mechanical resistance of compression of the mortar and/or concrete conferring to the standard EN 197-1 [23].

2.1.7. Fluorescence of X-rays (FRX) (9800 ARL spectrometer):

It is an elemental analysis technique that can measure up to 83 elements of the periodic table in samples of forms and varied nature. The spectrum of X-Rays emitted by the material is characteristic of the composition of the sample. By analyzing this spectrum, we can deduce the basic compositions, mass concentrations of elements.

2.1.8. Test room:

All tests are conducted in an air-conditioned room with a temperature of 20°C ± 1°C and a relative humidity of more than 90%, according to the specification of the norm EN 196-1 [24].

2.1.9. Sand

To prepare our mortar, we have used standard sand conferring to the standard EN 196-1, delivered by the new French company of Littoral. Its particle size analysis is illustrated in figure (1).

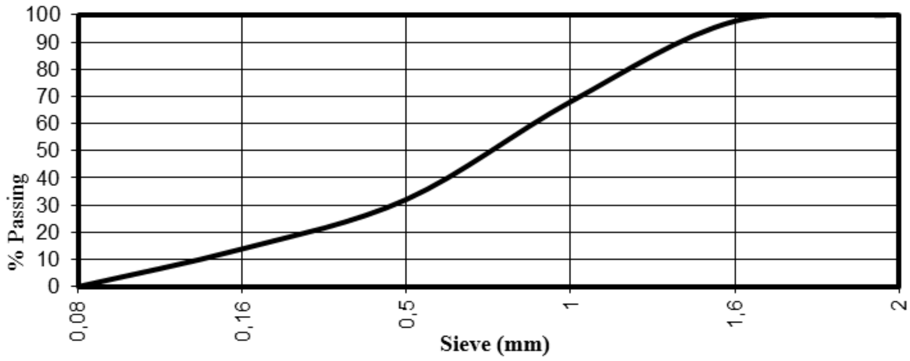


Figure1. Grading Curve of sand

The particle size analysis presented in figure (1) there is that used sand grains are distributed in a systematic way according to the standard EN 196-1 specifications [21].

2.1.10. The pure limestone (P.Lime)

The pure limestone (P.Lime) is an inorganic material that spread in many parts of Yemen, including Hadramout, Sana'a and Amran, etc. This material occupying a volume about of 3.6 billion m^3. The chemical analysis was determined by the X Ray Fluorescence (XRF), mineralogical analysis by X-Ray Diffraction (XRD) and physical analysis of the P.Lime of Bani Qais-Amran (Yemen) after crushing, drying for 12h at 80°C and grinding are shown in tables (4 and 5) and figure (2).

❖ *The physical-chemical characteristics*

The chemical composition of P.Lime after grinding determined by XRF is shown in table. (4).

Table 4. Elementary chemical composition of P.Lime determined by XRF

Content (%)	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	CL	LOI
Pure limestone	54,96	0.621	0.12	0,159	0.411	0,0799	0.0132	0	0.0006	43.64

As the results shown in the table (4) we can see that the P.Lime of Bani Qais-Amran - Yemen contains to 54.96% of lime (CaO), 0.12 of alumina, 0.159 of iron and 0.621% of silica. In order the sum of the percentages is equal to 56.36% and the rest of the sum is the loss of ignition (LOI) [22]. The mineralogical analysis by X-Ray Diffraction (XRD) of the P.Lime of Bani Qais-Amran-Yemen is shown in figure 2.

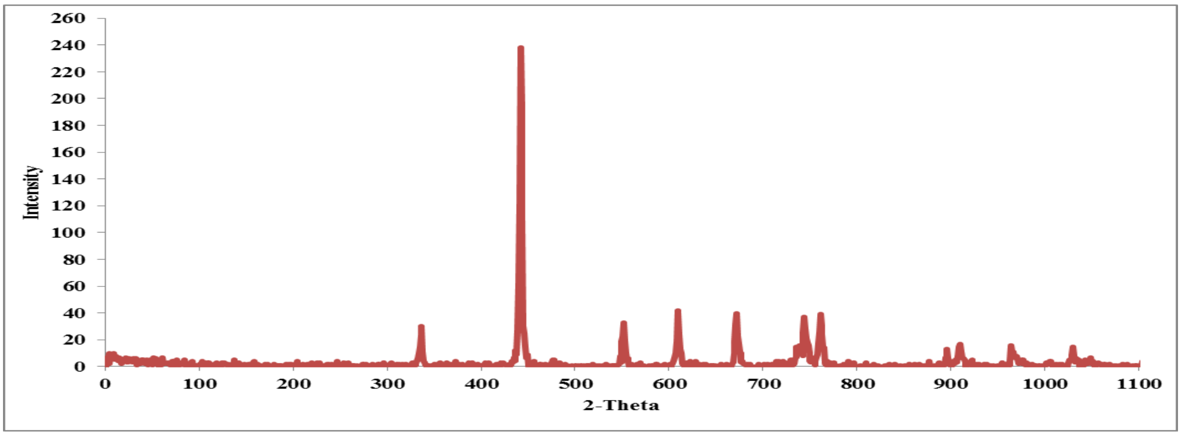
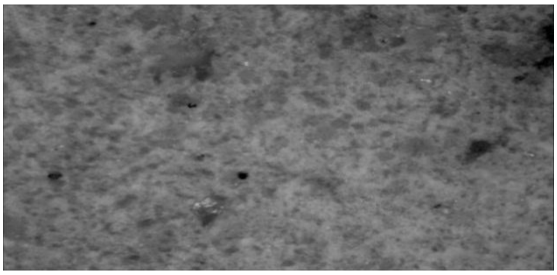


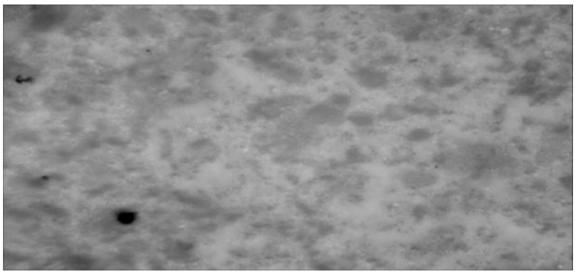
Figure 2. Spectrum of P.Lime by the X-Ray Diffraction

The results of the mineralogical analysis by X-Ray Diffraction (XRD) of the P.Lime in figure (2) reveal the strong presence of the lime, followed by the silica, then the magnesium afterwards the oxide reveals the strong presence of the calcite ($CaCO_3$), afterward by the dolomite ($CaCO_3/MgCO_3$) and then the Magnesium carbonate, ($MgCO_3$) and the Magnesium hydroxide Ca $(OH)_2$/mg $(OH)_2$ [23-25].

The microscope metallography analysis of the P.Lime gives an indication on the rearrangement of the particles of a composite solid powder state is summarized in figure (3).



(a)



(b)

Figure 3: View of the P.Lime by microscopic metallography of 100 x (a) and 200 x (b)

The particles of P.Lime in the form of rosettes, are illustrated in the figure (3) using a metallography microscope to two extensions (100 and 200).

❖ *The physical characteristics*

The physical characteristics of the pure limestone are given in table (5):

Table 5. Physical properties of the P.Lime

Physical characteristics	Units	Values
Blaine specific surface area	cm^2/g	4776
Density	g/cm^3	2.13

2.1.11. The mixing water

Spoil our mixture, we used tap water (wells), its main characteristics are collected in the table (6).

Table 6. The main characteristics of the mixing water

Components	PH	T, D, N	CO_3^{-2}	HCO_3^-	Calcium	Magnesium (Mg^{+2})	Conductivity
Units	-	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	ms/cm
Values	7.0	450	216.0	0.0	56.4	52.4	692,0

2.2. Methods

We have studied the influence of the incorporation of P.Lime in the matrix of formulation of cement while partially substituting the clinker with P. Lime at different percentages ranging from 0% to 40% by weight of clinker with a step of 5 % on the physical - chemical properties of cement, such as the fineness by specific surface area, the density, the setting time, the water content and on the mechanical properties of concrete. The different formulations were prepared in a standard mixer EN-196-1 [27] following the procedure indicated by the norm EN-196-3 [26] relative to the normal consistency of pure paste [26-29]. The method includes determining of compressive strength of the prismatic specimens of dimensions 40 mm × 40 mm × 160 mm. These specimens were collected from a paste plastic mortar according to the norm EN 196-1 [27]. The matrix of the formulation containing one part of a cement + addition of CKD and three parts of sand normalized with a report of water and cement varies from 0.50% to 0.54%, according to the percentage of the P.Lime as indicated in the tables (7), (9) and (9). The mortar is prepared by mixing and putting into a mold using a standardized shocked device [26-28]. The mold containing the test sections is kept in a humid atmosphere for 24 hours and then removed from the mold is stored under water pending the moment of the strength tests. At the required age, the samples are removed from their wet preservative medium, they are broken into two halves by bending and each half is subject to the compression test [28].

Table 7. Composition of cement powder state used to prepare 500g of mixture at base of P.Lime

Content (%)		C-T	C P.Lime 5%	C P.Lime10%	C P.Lime 15%	C P.Lime 20%	C P.Lime 25%	C P.Lime 30%	C P.Lime 35%	C P.Lime 40%
Cement	Mass (g)	500	475	450	425	400	375	350	325	300
	%	100	95	90	85	80	75	70	65	60
Pure limestone	Mass (g)	0	25	50	75	100	125	150	175	200
	%	0	5	10	15	20	25	30	35	40

Table 8. Composition of fresh cement paste state at base of P.Lime

Content (%)		P-T	P P.Lime 5%	P P.Lime10%	P P.Lime 15%	P P.Lime 20%	P P.Lime 25%	P P.Lime 30%	P P.Lime 35%	P P.Lime 40%
Cement	Mass (g)	500	475	450	425	400	375	350	325	300
	%	100	95	90	85	80	75	70	65	60
Pure limestone	Mass (g)	0	25	50	75	100	125	150	175	200
	%	0	5	10	15	20	25	30	35	40
Water	Mass (g)	140	143	146	148	150	153	155	157	160
	W/C	0.280	0.286	0.292	0.296	0.300	0.306	0.310	0.314	0.320

Table 9. Composition of mortars in the hardened state used by P.Lime

Content (%)		M-T	M P.Lime 5%	M P.Lime10%	M P.Lime 15%	M P.Lime 20%	M P.Lime 25%	M P.Lime 30%	M P.Lime 35%	M P.Lime 40%
Cement	Mass (g)	450	427.5	405	382,5	360	337.5	315	292.5	270
	%	100	95	90	85	80	75	70	65	60
Pure limestone	Mass (g)	0	25	50	75	100	125	150	175	200
	%	0	5	10	15	20	25	30	35	40
Sand	Mass (g)	1350	1350	1350	1350	1350	1350	1350	1350	1350
Water	Mass (g)	225	227	231	233	235	238	239	241	243
	W/C	0.500	0.504	0.513	0.518	0,522	0.529	0,531	0.536	0.540

3. RESULTS AND DISCUSSION

3.1. Physical-chemical properties of cement at base of the pure limestone

3.1.1. The density of cement at base of P.Lime

The absolute density of cement is measured by the movement of liquid inert to cement inside a graduated container, it is measured using a device Le Chatelier preferably jolting according to EN 196 - 6 / ASTM C188, NM 10.1.004, was presented in the figure (4) [29].

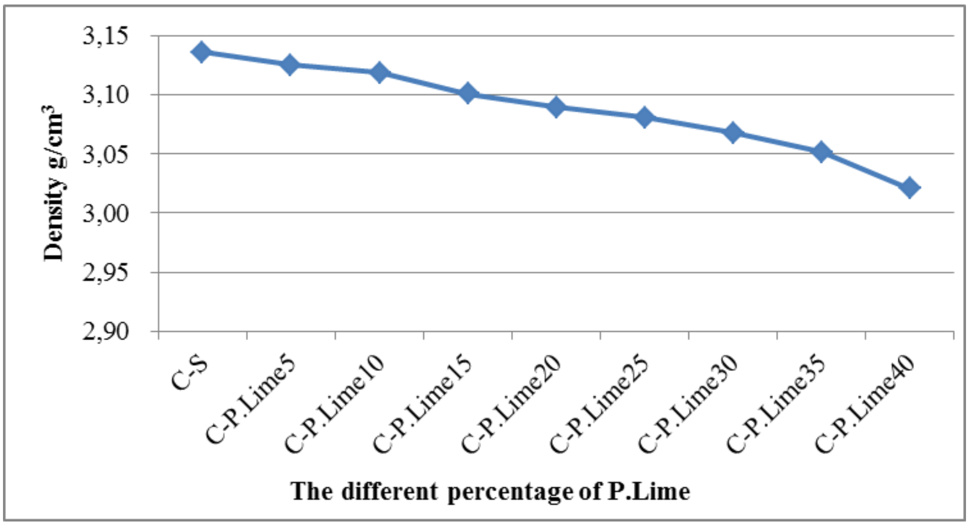


Figure 4. Variation in the density in function to the different percentage of P.Lime

This experimental study shown in the figure (4) reports that there is a decrease in the density of cement with the addition of the P.Lime by cement control report. For example, at 40% of the P.Lime in cement, the density decreases from 3.14 g/cm^3 to 3.02 g/cm^3. This decline is logically explained by the fact that adding that replaces the clinker has a lower real density [30-34].

3.1.2. Fineness by the method of air permeability (method of Blaine specific surface area)

The fineness of the cement is usually expressed by its surface area. It is the total surface grains contained in a mass of powder [35-37]. The specific surface is usually expressed in cm² of surface area of the cement grains per gram of

powder. The fineness is an important feature: when tempering, more cement in contact with the water surface is large and more moisture is fast and complete. The specific surface of our mixture is determined using the apparatus air permeability standard 10.1.005 NM, was distinguished in the figure (5).

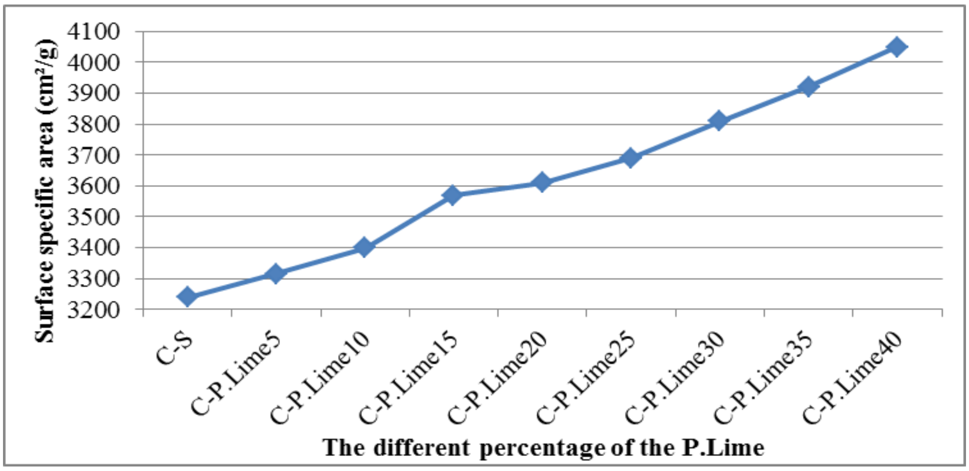


Figure 5. Variation of the surface specific Blaine in function of the different percentage of the P.Lime

According to the figure (5) observed that the SSB of cement increases with the addition of P.Lime, usually caused by the specific surface of P.Lime.

3.2. The influence of the P.Lime on the physical properties of the fresh cement paste state

3.2.1. Water content

The consistency of the cement paste is a feature, which evolves over time. To be able to study the evolution of the consistency in terms of to the different percentages of addition of pure limestone (partial substitution of the clinker by the P.Lime of: 0%, 5%, 10%, 15%, 20%, 25%, 30%, 35% and 40%), it must be possible from a standard consistency which is the same for all studied paste [38-40]. The aim of this test is to determine the optimum amount of mixing water for obtaining a good mortar. This testing is with the Vicat apparatus in accordance of the EN 196-3. It was illustrated in the figure (6).

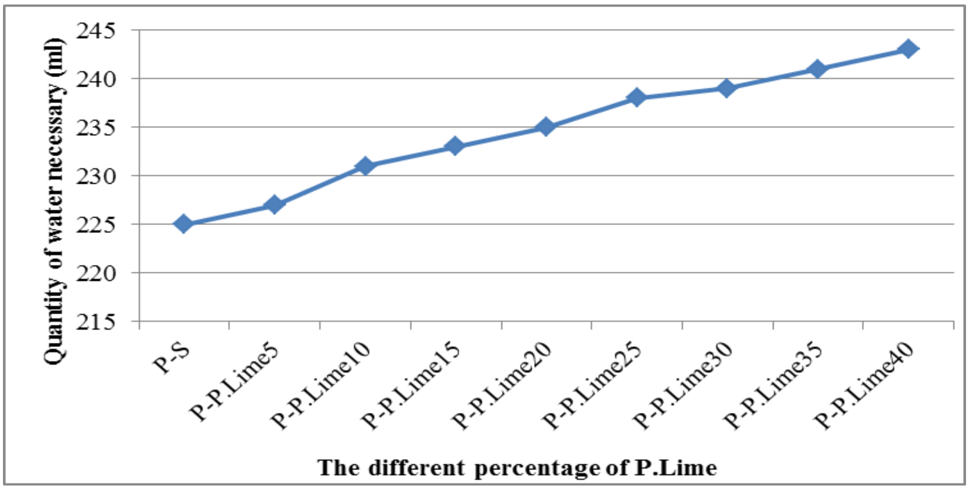


Figure 6. Variation of the necessary quantity of water in function to the different percentage of P.Lime

According to the figure (6) there is the water content of the new binding cement paste at base of P.Lime increases with mass fraction of this. This increase is generally owing to the chemical and mineralogical composition of our addition in the P.Lime which is rich in CaO; on one hand the presence of CaO high rate influence on the phenomenon of hydration since this mineral phase-rich limestone tends to have more demand water and on the other hand to the fineness of the addition.

3.2.2. The setting time

The objective of this test, in this new hydraulic binding base of P.Lime, is to know the initial and the final times of the fresh cement paste made from this material. This experiment was conducted with EN 196-3 using the compliant automatic Vicat apparatus, was noted in the figure (7).

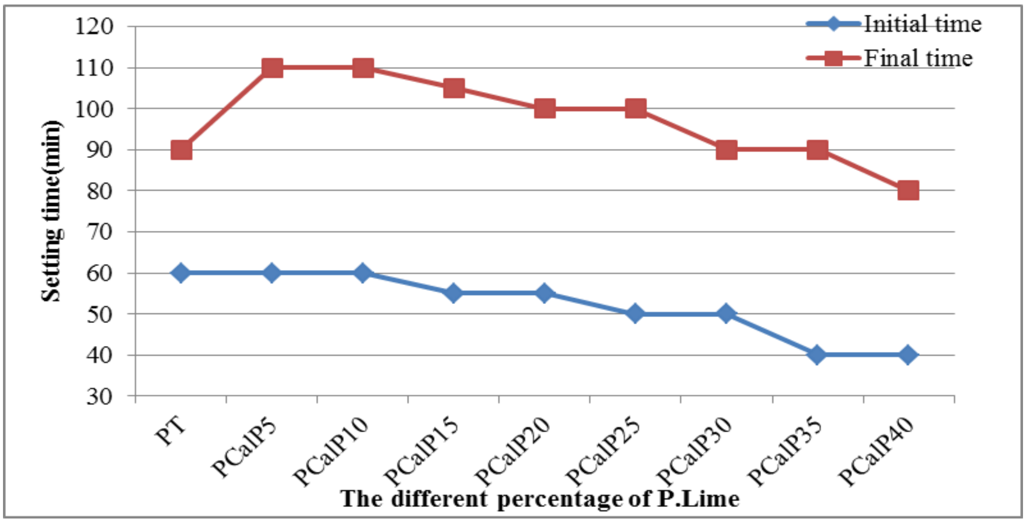


Figure 7. Variation of setting time in function to the different percentage of P.Lime

According the figure (7), that shows the evolution of initial and final time of cement paste based on P.Lime depending on the mass fraction of the addition of the P.Lime, we observed that the presence of the fillers of limestone in cement acts favorably on the physical properties of our new binder basis of P.Lime, such as the initial and final times. This last decreased significantly by the percentage of the P.Lime, which gave our bonded the role of accelerator. This decrease in setting time due generally to the composition chemical and the mineralogical of our addition which is rich in CaO and low Al_2O_3 on one hand and on the other hand, it is grace to the fineness of the fillers of limestone that fills the gaps between the cement particles, which subsequently improves the compactness of the concrete.

3.3. The influence of the P.Lime on the mechanical properties in the hardened state

At last, we also assessed the influence of P.Lime on the mechanical performance of mortar or concrete by using the mechanical resistance of compression. The measurements of the mechanical resistance are normal mortars (4 x 4 x 16) cm^3 sample according to the specification of the standard (NF EN 196-1) [28,41]. The samples of the test are demoted after one day and kept in down-water in the age of crushing. The measurement of the mechanical resistance of compression is performed at 2, 7 and 28 days to observe the gradual evolution of mechanical performance of our new hydraulic binders P.Lime-based on the mass fraction of the P.Lime in %, were presented in the figure (8). According the figure (8) we can deduct the following remarks:

- ❖ The resistance of all the P.Lime mortars increases steadily with age and are no drop.

❖ The compression strength decreases significantly with the increase in the percentage of the adding of P.Lime 2, 7 and 28 days.

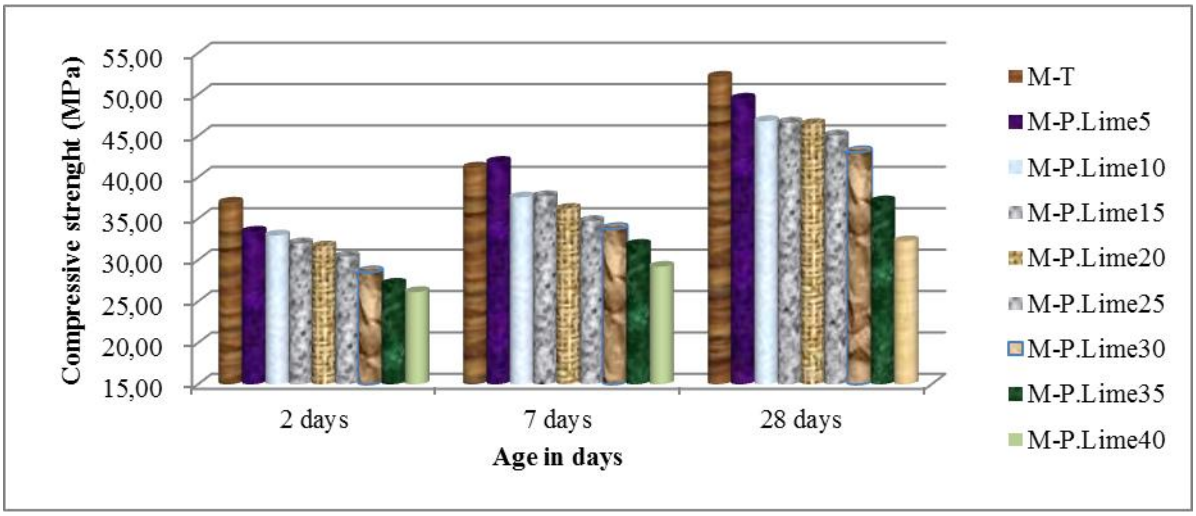


Figure 8. Variation of the compressive strength in function to the age in days

4. CONCLUSION

In this work, we studied the influence of the substitution partial of clinker by the pure limestone on the physico-chemical characteristics of cement, physical properties and compressive strength. The study highlighting on the dependence between the replacement the part of clinker by the pure limestone at different percentages and the properties of cement. It's about, in particular, the fineness by the specific surface area, the density, the free lime, the demand of water, the setting time and the compressive strength. Indeed, the physical-chemical and the mechanical analysis of cement showed that up to 40% of pure limestone, the cement properties are adequate according to international standards. The results obtained by the different methods of physical- chemical analysis showed that the adding of P.Lime in cement increased the fineness by specific surface. More the density is decreased. We have remarked that, the setting time was decreased when the percentage of the P.Lime is increased. Of the same, the compressive strengths of mechanical at 2 /7 and 28 days have improved according to the increase of pure limestone. However, a rate of pure limestone above 40%, will lead to a drop in cement quality which is concretized by low mechanical strengths and a very strong expansion.

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