

Effects of Mineral Addition on Rheological Properties of Mortar

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Abstract

The evolution of the science of the chemistry of admixtures besides the integration of various mineral additions in granular mixtures has metamorphosed the field of the concrete material. Thus, the produced mixtures are directly affected by the chosen combination of additions of mineral and organic. The disorders of industrial experience have often been associated with the dosages and interactions between the various additions used. The proposed study investigates the impact of the combination of different types of mineral and organic additions on the rheological properties of mortars.

In this research work, mortars were made by varying the rate of substitution of cement by three types of mineral additions (limestone fillers, marble powder, natural pozzolan), in association with different dosages of a 3rd generation superplasticizer (polycarboxylates), in order to study the evolution of shear thresholds and viscosities. In addition, a coaxial vane-type rheometer is used to study the rheological characterization of the mortars. The obtained experimental results revealed the effectiveness of the incorporation of inert mineral additions combined with polycarboxylates on the rheological parameters..

Keywords: Rheology; Mortar; Pozzolan; Marble powder; limestone fillers; polycarboxylates; Shear threshold; viscosity.

1. Introduction

Cementitious mixtures have withstood an important evolution in terms of rheology for several decades due to the advances in the physico-chemistry of materials. The chemical admixture and the mineral additions play costly and essential assets in obtaining the adequate rheological characteristics to the technical and aesthetic quality needed from the constructions.

In order to avoid the agglomeration of cement particles while releasing the water trapped by these grains, superplasticizers are adsorbed on the fine elements of cementitious materials. Thereafter, the fluidity of the mixture while reducing the quantity of mixing water is improved.

For a long time, several generations of superplasticizers have been developed and used,

recently, in the civil engineering sector, polycarboxylates are the most used and effective [1]. These comb-shaped copolymers and carriers of anionic functions and side chains guarantee the dispersion effect through an electrostatic equilibrium that is obtained by neutralization of the positive electric charges existing on the surface of the cement grains and the addition. The identical charges create a force known as a repulsive force that causes strong dispersion of the particles and thereafter prevents coagulation [2]. Although the main chain plays a dominant role in adsorption and electrostatic repulsion phenomena, the side chains are decisive for steric repulsion phenomena [3] preventing Van interactions der Waals to develop an attractive force between the particles [4].

Furthermore, the adsorption of polycarboxylates is not uniform on the surface of cement grains [5]. certainly, there exists an interaction between the polycarboxylates and positively charged phases of cement grains. Therefore, they are adsorbed essentially on the ettringite crystals constituting the aluminate phase, in addition to struggling with the sulfate ions existent in the cement paste in the form of gypsum, or alkaline sulfates [6]. If the cementitious materials are represented by the Bingham model, where the main parameters are the shear threshold and the plastic viscosity, therefore the superplasticizers mainly require a crucial reduction in the dynamic and static shear thresholds. And this is due to the facilitated sliding between the solid grains in the presence of the adsorbed boundary layer [7].

Besides, the incorporation of mineral additions in cementitious structures that gives rise to an important improvement in the carbon balance by reducing energy consumption and the quantities of CO₂ released associated with the production of Clinker [8]. Certain specific properties of the

concrete can be improved by partially substituting the cement with the mineral powders obtained from the additions. Also, the physical influence of mineral additions improves the compactness of the mixture. In some cases, the effective water dosage can be reduced [9], or chemically affected by producing additional hydrates through pozzolanic reactions [10].

In the literature the influence of mineral additions is widely studied and investigated. The rheological behavior of cementitious mixtures have been firstly influenced by the nature of the mineral additives used [11].

Several authors have found that the optimal rates of workability improvement of cementitious and granular mixes is related to each type of mineral addition [12; 13, 14, 15]. But the obtained result is affected by the particle size and the form of the mineral additions which has a great influence on the rheological parameters, especially the viscosity [16, 17].

2. Used materials

The mortars studied are made using an ordinary portland cement CEM 42.5. Marble powder (WMP), limestone fillers (LF) and natural pozzolan (NP) were chosen in this study to partially replace cement. The physico-chemical properties of the cement and the various additives used are reported in table 01

The pozzolan used is of volcanic origin. It is recovered in the form of crushed rocks then crushed and sieved. The marble powder used is recovered by sieving waste from the cutting and shaping of marble stones in an industrial manufacturing, while the limestone fillers used come from the crushing of limestone rocks from dolomitic quarries.

Chemical analysis reveals that pozzolan contains higher dosages of silica and alumina compared to other powders. It also has the highest content of magnesium oxide.

The results of the chemical analysis of the other powders also indicate that they are mainly made up of calcium oxide with a mass concentration of 55% for marble powder and around 53% for limestone fillers.

Methylene blue tests revealed that the (MBV) value of limestone and marble fines were 0.12 and 0.40, respectively, indicating that both powders do not contain clay.

SEM analysis of the three additions used shows that they are in the form of non-abrasive powder characterized by a rhombohedral structure (figure 01).



(a)

(b)



(c)

Figure 01. Powder morphologies obtained by scanning electron microscopy ($\times 1000$).
(a) marbre (b) Fillers (c) pouzzolane
(b)

Table: 01 Chemical composition of Marble powder (WMP), limestone fillers (LF) and natural pozzolan (NP)

	Cement	WMP	LF	NP
SiO ₂	27,17	0,48	0.06	37,84
Al ₂ O ₃	5,47	0,10	0.29	14,74
Fe ₂ O ₃	3,17	0,12	0.22	14,1
CaO	57,20	55,17	52.63	5,34
MgO	1,01	0,72	0.84	3,03
SO ₃	2,27	0,46	0.02	0,25
K ₂ O	0,49	0,01	0.01	0,73
Na ₂ O	0,19	0,01	0.08	0,75
Free CaO	1,91		-	
P ₂ O ₅	0,18	0,02	0.02	0,68
TiO ₂	0,43	0,01	0.02	3,37
PAF		43,53	42.77	
Density	3,03	2,7	2.7	2,77
Blaine SS[cm ² /g]	3700	3600	3300	3200
C ₃ S	52,63			
C ₂ S	23,68			
C ₃ A	8,66			
C ₄ AF	10,71			
< 63 μ m (%)	86.25	78.75	74.51	60.4
compactness without SP	0,553	0.580	0.586	0,573
compactness with SP	0,611	0.624	0.647	0,600

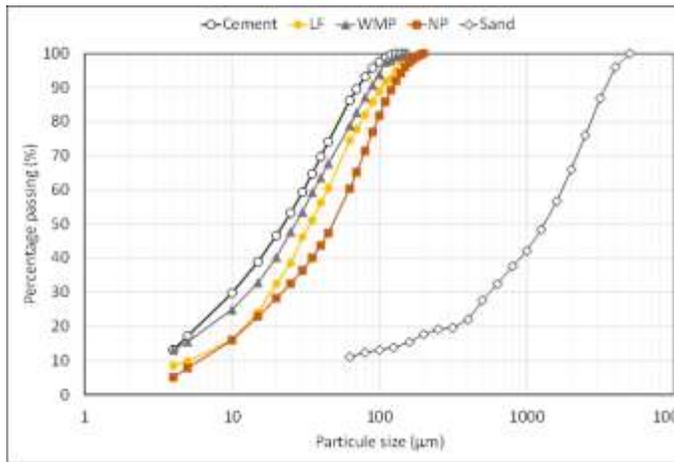


Figure 02. Particle size distributions of materials used.

The sand used is from the big carry of the national company of aggregate. The sand is marketed in granular class 0/4 were obtained from crushed and ground calcareous rocks. It is characterized by an absolute density of 2580 kg/m³, an absorption coefficient of 1.6% and a fineness modulus of 3.08. The grain size analysis of the sand is also represented in figure 02.

The superplasticizer used is of the polycarboxylate type, the main chain of which is composed of carboxylate functions which carries side chains of poly(ethylene oxide). They are characterized by a density of 1.06 and a dry extract content of 22%. The amount of water provided by the polycarboxylate will be taken into account to maintain a constant water/cement (W/L) ratio for all formulations

3. combination studied mixtures :

From a reference mortar with a weight ratio: cement: sand: water equal to 1: 3: 0.50, we partially substituted the cement with the mineral additions chosen for our study (pozzolan, marble powder, fillers limestone) at mass percentages ranging from 0 to 30%. For each formulation, the superplasticizer is used in dosages of 0, 0.2 and

0.4% of the binder mass. In all, 30 combinations listed in table 02 were tested in this study.

4. Méthodes expérimentales

The used method considered the locally sheared material as a Bingham fluid and computed the characteristic shear rate from Couette analogy [19]. The rheological properties of mortar and concrete are closely depending on the constituent's nature and the mixture composition [20]

that is why many rheological tests were executed in this current study, to identify the role of barite in the concrete mixtures. To reduce the amount of concrete batches (reducing costs) and the use of components including cement (reducing the ecological impact). The rheological measures of different CEM mixtures were carried out using an apparatus 'Mortar Rheometer' developed by Soualhi et al. (Figure 3). The main parts of this rheometer are: cylindrical container with a diameter of 10 cm and a height of 13 cm, a steel vane with 5 cm in diameter and 10 cm in height, and the essential component is an agitator directed by a software to record the torques (Figure 3).

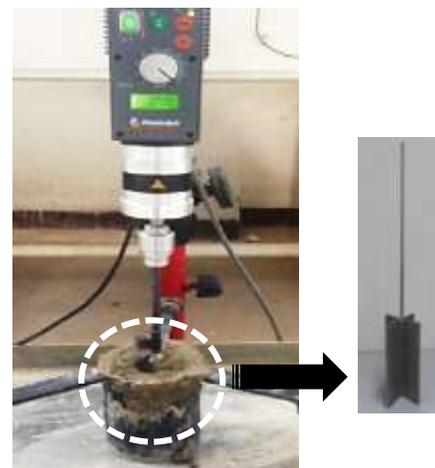


Figure 03: Rhéomètre used

The working principle of rheometer consists in rotating the vane in a cylindrical sample of fresh CEM and then measuring the torques

applied to maintain rotation. A decreasing rotational speed is imposed to the vane of rheometer interrupted by a stabilization phase. The imposed rotation speed Ω (torque M) is recorded every second. The rheological tests' results are obtained in the following form: The main rheological parameters (yield stress τ_0 and plastic viscosity μ) are calculated through the experimental measurements by converting the vane torque and the rotational speed data into shear stress versus shear rate relationships (Figure 4).

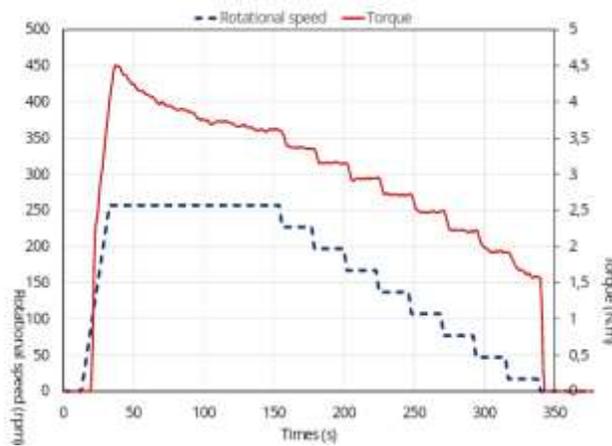


Figure 04: Evolution of torque (T) and rotational speed (N) as a function of time

Mixtures	W/B	Sand	OPC (g)	LF (g)	WM P	NP (g)	SP (%)
Control	0,50	2100	700	0	0	0	0
M1			630	70	0	0	0
M2			560	140	0	0	0
M3			490	210	0	0	0
M4			700	0	0	0	0.2
M5			630	70	0	0	0.2
M6			560	140	0	0	0.2
M7			490	210	0	0	0.2
M8			700	0	0	0	0.4
M9			630	70	0	0	0.4
M10			560	140	0	0	0.4
M11			490	210	0	0	0.4
M12			630	0	70	0	0
M13			560	0	140	0	0
M14			490	0	210	0	0
M15			630	0	70	0	0.2
M16			560	0	140	0	0.2
M17			490	0	210	0	0.2
M18			630	0	70	0	0.4
M19			560	0	140	0	0.4
M20			490	0	210	0	0.4
M21			630	0	0	70	0
M22			560	0	0	140	0
M23			490	0	0	210	0
M24			630	0	0	70	0.2
M25			560	0	0	140	0.2
M26			490	0	0	210	0.2
M27			630	0	0	70	0.4
M28			560	0	0	140	0.4
M29			490	0	0	210	0.4

Table 02: Mixtures studied

5. Results and discussion :

The results shown in Figure 05 confirm that the increase in dosage of superplasticizer is always accompanied by an improvement in workability due to better deflocculation of the binder grains,

which prevents the formation of conglomerates. In addition, the obtained result can be expressed in terms of the reduction in necessary torque maintaining a given speed of rotation. Thus, it leads to a reduction in the shear threshold.

Indeed, the use of Polycarboxylate superplasticizer tends to release the water trapped in the clusters formed by the cement grains by "electrosteric" effect. The released water acts as a lubricant between the particles. Also, it facilitates their sliding on each other to position themselves in an optimal way which leads to the reduction of the rheological parameters.

Thereafter, the superplasticizer causes a slight decrease in plastic viscosity. However, [21] noted that the effect of superplasticizers on viscosity is less significant on mortars due to the absence of gravel in certain phenomena such as thixotropy or destructuring. In addition, chemical reactions can rapidly modify the rheological behavior of a mortar.

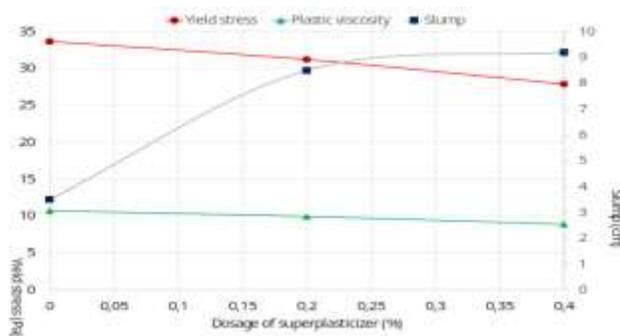


Figure 05 : Rheological parameters of mortars en fonction dosage de superplasticizer

The influence of the three mineral additions studied on the rheological parameters of the mortars is illustrated in figures 06 and 07. The results clearly show that the effect of the additions is not the same.

The partial substitution of cement by inert additions such as limestone fillers or marble powder is interesting from a rheological point of

view. The shear stress and plastic viscosity can be reduced by incorporating two additions with identical chemical composition and morphology. The reason for this result is the fact that there exists a difference between the absolute densities of the additions and that of the cement (fillers and marble: 2.7; cement 3.03). Therefore, an increase in the volume of the binder is generated by a partial mass substitution of the cement with fillers or marble powder. In addition, the paste plays the role of lubricant leading to the improvement in the rheological parameters. Besides, a certain rate of substitution of the cement by these inert additions, the friction between the particles of the binder becomes greater. Consequently increases the rheological parameters of the mixtures.

It also observed that the Blaine-specific surface has more marble powder compared to fillers, which leads to an increase in rheological properties because the demand for water is greater. Our conclusions agree with those of certain researchers [22]. Whilst declaring that for a constant water dosage, the paste becomes more viscous with the increase in the specific surface of the powder. Hence, it leads to the amplification in the shear threshold and the viscosity of the mixture.

However, it is evident that the difference in densities between the cement and the additions cannot be the only reason for the decrease in the values of the rheological parameters since the use of pozzolan with a density of 2.77 also may increase the volume of the paste but with different effect on the rheological parameters of the mortars. It is clear that other intrinsic characteristics of the mineral additions have more significant effect on the rheological parameters.

Chemical composition of natural pozzolana especially its high silica and alumina levels compared to other powders (table 01) is very reactive, involving a greater absorption of the

quantity of water which results to higher rheological parameters.

Moreover, the morphological study of the three additions has shown that the grains of these powders have almost the same shapes (figure 01). Therefore, we noticed on the basis of the particle size analysis that the pozzolana contains a quantity of coarse elements ($> 63 \mu\text{m}$) greater than that contained in the marble powder and limestone fillers. This result, also has been proven by microscopic tests allowing to know the higher shear thresholds and viscosities obtained for mortars with pozzolan. Which are surely the result of greater friction in their pastes.

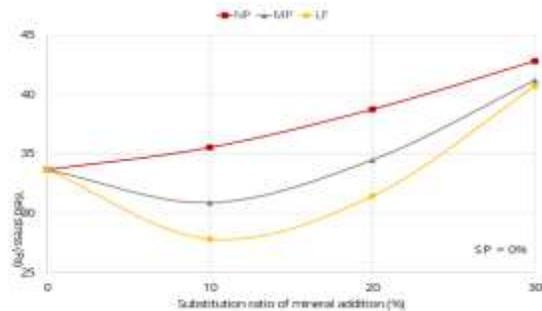


Figure 06 : Evolution of the shear threshold according to the mineral addition

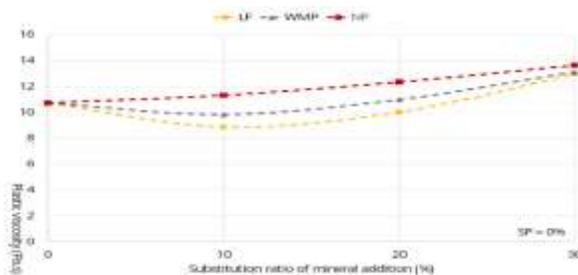


Figure 07 : Evolution of the plastic viscosity according to the mineral addition

The use of superplasticizers has lowered the rheological parameters of all mortars regardless of the type of mineral addition used.

In the presence of superplasticizer in the cementitious matrix, the rheological parameters may vary according to the affinity between superplasticizer and mineral additions and the reactivity of the particles. Thus, the limestone

fillers and the marble powder improves the packing density, which proves their beneficial effects on the granular arrangement. And thus, allowing more free water to be left to fluidify the mixture leading to smaller rheological parameters. On the other hand, the use of pozzolan combined with a superplasticizer has rather a less significant effect on compactness (Table 1).

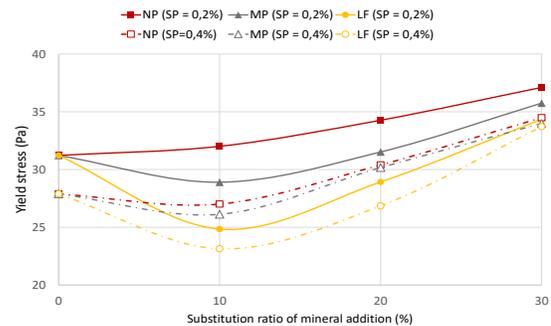


Figure 08 : Evolution of yield stress en fonction of mineral addition

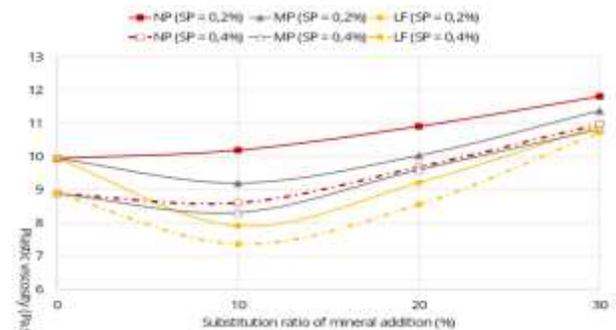


Figure 09 : Evolution of viscosity en fonction of mineral addition

Moreover, according to some researchers, pozzolan prevents the adsorption of the superplasticizer on the cement grains due to its alkali and aluminate content (table 01) [23,24]. In this case, the superplasticizer dosage should be increased in order to reduce the rheological parameters. This is also the reason why the difference between the effect of the different additions on the rheological parameters is

reduced when the dosage of superplasticizers is increased from 0.2 to 0.4% (figures 06 and 07).

The obtained result has been described by previous work which showed that pozzolana requires more superplasticizers to be saturated compared to cement and limestone fillers [25].

From the obtained results, it is observed that a substitution of 20% of cement by limestone fillers or marble powder can be rheologically effective, while a rate of 10% to 20% is recommended for pozzolan depending on the superplasticizer dosage.

6. Conclusion

From the results obtained in this study the following conclusions can be drawn

- The polycarboxylate significantly reduces the sill of shear and lightly the plastic viscosity.
- The substitution of up to 20% cement by inert additions makes it possible to reduce the rheological parameters of the mixture. but, the substitution of cement by reactive additions at a rate greater than 10% generates an increase in the sill shear of the plastic viscosity..
- The combination between the polycarboxylate and inert mineral additions offer the best rheological performance to the mixtures. On the other hand, the polycarboxylate seems to lose its effectiveness when it is used with a high rate of natural pozzolan.

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