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RHEOLOGICAL AND MECHANICAL BEHAVIOR STUDY OF ECO-FRIENDLY CEMENT MORTAR MADE WITH MARBLE POWDER

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The work aim is to investigate the rheological and mechanical behavior of an eco-friendly mortar made with marble powder. Marble is have used as sand (total substitution of natural sand) and as an additional material (partial substitution of cement). Firstly, rheological tests were carried out on the cement pastes in order to study the effect of cement substitution by marble powder on the rheological behavior. Secondly, our study is devoted to evaluate the mechanical performances (flexural strength, compressive strength, mechanical behavior and ultrasonic pulse velocity) of a fluid mortar such as the case of the self-compacting mortars elaborated with the marble powder as an addition a material and as sand. The mechanical test results show that compressive strength and mechanical behavior of an ecological cement mortar made with marble waste as natural sand improved significantly. However, marble-based mortars with *100%* of marble sand have given a mechanical strength similar to that obtained by control cement mortar (*100%* natural sand). It was also noted that it an ecological cement mortar made with *30%* of marble powder as an addition a supplementary material can be obtained. This leads to a reduction in cement consumption and a reduction in CO_2 gas emissions caused by cement production.

Key words: powder marble wastes, mortar, sand, rheological properties, viscosity, mechanical strength, mechanical behavior and strength gain.

1. Introduction

Marble is a commonly utilized building material in the field of civil engineering, and its processing results in a substantial volume of waste generated in quarries and processing facilities. Over *30%* of marble is typically discarded as waste in the form of powder or fine aggregates. Numerous studies have demonstrated the potential for reusing this type of waste in various applications, particularly in the construction materials sector. Extensive research has been conducted on incorporating marble waste into concrete formulations. Multiple investigations have explored the feasibility of utilizing marble dust waste as a mineral additive in concrete [1-9]. These studies generally found that marble dust had no adverse effects on the workability of fresh concrete but did have a detrimental impact on the mechanical properties of hardened concrete.
Researchers have also examined the use of waste marble as an alternative to fine aggregates or as an additional material [10-17]. Binici and colleagues delved into the durability of concrete containing granite and marble as coarse aggregates [18, 19], demonstrating that substituting marble, granite, and ground blast furnace slag could lead to more durable concrete. Other research has investigated the properties of concretes and mortars incorporating marble powder waste. The results revealed that substituting *10%* of natural sand with marble powder yielded the highest strength. Additionally, Gameiro *et al.* explored the characteristics of concretes

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created with marble waste by substituting aggregates at rates of *0%*, *20%*, *50%*, and *100%*, reporting that concrete incorporating marble aggregates exhibited superior properties compared to those with fine aggregates. More recently, Singh *et al.* [21, 22] provided an overview of research focused on partially replacing cement and sand with marble waste in concrete.

Regarding the use of marble waste in self-compacting concretes, studies evaluated the use *30%* of waste marble as fines in concrete formulation. Results have shown that the fresh and hardened characteristics of concrete were improved. Hameed *et al.* [23] have evaluated the physical and mechanical characteristics of concrete by using the marble powder. With *15%* of marble powder self-compacting concretes can be obtained having best characteristics, namely: the water absorption and permeability as well as fast chloride penetration and compressive strength. The utilization of limestone and marble powder in the concretes production was also studied [24]. These authors' findings revealed that marble powder improved the compressive strength at a rate that should not be underestimated. They reported that the highest compressive strength was observed in mixes that contained *10%* marble [24, 25]. However, it should be noted that other researchers have found that using marble powder (especially sand substitution) leads to improvements in the mechanical characteristics of the concrete [3, 26]. Munir *et al.* [27] studied the characteristics of concrete elaborated with cement replacement by waste marble powder up to *40%*. Singh *et al.* [22, 28] have reported that up to *15%* or *20%* of powder marble can be used (as cement substitution) in concrete production.

In this regard, our study aims to reclaim waste marble for reuse as sand in the formulation of fluid concrete. The experimental investigation was conducted in two phases. Initially, it involved the examination of the effect of cement substitution with marble powder (*0%*, *10%*, *20%*, *30%* and *50%* wt.) on the rheological behavior of the studied cement pastes, the other phase is devoted to evaluation of the mechanical performances (flexural strength, compressive strength, mechanical behavior and ultrasonic pulse velocity) of self-compacting mortars elaborated with the marble powder as cementitious materials (*0%*, *10%*, *20%*, *30%* and *50%* wt. of cement) and as sand (*0%*, *30%*, *50%*, *70%* and *100%* wt. of natural sand).

2. Experimental program

2.1. Raw materials

Portland cement, limestone filler, polycarboxylate-based superplasticizer, natural sand (*0/3mm*), marble powder (PM), are used as raw materials in this study. Portland cement is the CEM II 42.5 usually used in construction. The marble powder used is recovered from crushed wastes of the white marble quarry. River sand was used in mortar formulation, after being sieved to remove coarse grains, then washed and dried in an oven at *105°C* for *24h*. The chemical composition of the binder (cement and marble) is given in Tab.1. Physical characteristics of sand and marble is given in Tab.2. They are in accordance with the european standard EN 12620. Finally, to ensure the fluidity required for self-compacting mortars, a super-plasticizer conforming to EN-934-2 was used (see in Tab.2, which shows the physical properties of the superplasticizer applied).

Table 1. Chemical composition of binder (cement and marble powder).

chemical compound	portland cement (PC) [%]	marble powder (PM) [%]
$CaCO_3$	-	*99.05*
CaO	*63.02*	*55.06*
P.C	-	*45.26*
MgO	*1.87*	*1.01*
SiO_2	*20.7*	*0.15*
Al_2O_3	*4.75*	*0.08*
Fe_2O_3	*3.75*	*0.07*
SO_3	*1.98*	-
Na_2O	*0.90*	-

Table 2. Physical characteristics of natural sand, marble powder and superplasticizer.

properties	natural sand	marble powder	superplasticizer
true density	*2.750*	*2.73*	*1.06 ± 0.01*
apparent density	*2.66*	*2.63*	
sand equivalent (%)	*85*	*77*	
fineness modulus	*2.8*	*2.2*	
porosity	*2.18*	*3.72*	
compactness	*97.80*	*96.25*	
absorption (by weight) (%)	*0.71*	*0.41*	
saturation (%)	*0.75*	*0.87*	
pH	-	-	*4.5 – 6.5*
content of Na_2o eq	-	-	*≤ 1 %*
content of Cl^-	-	-	*≤ 0.1 %*
dry extract	-	-	*28.01 ± 2 %*

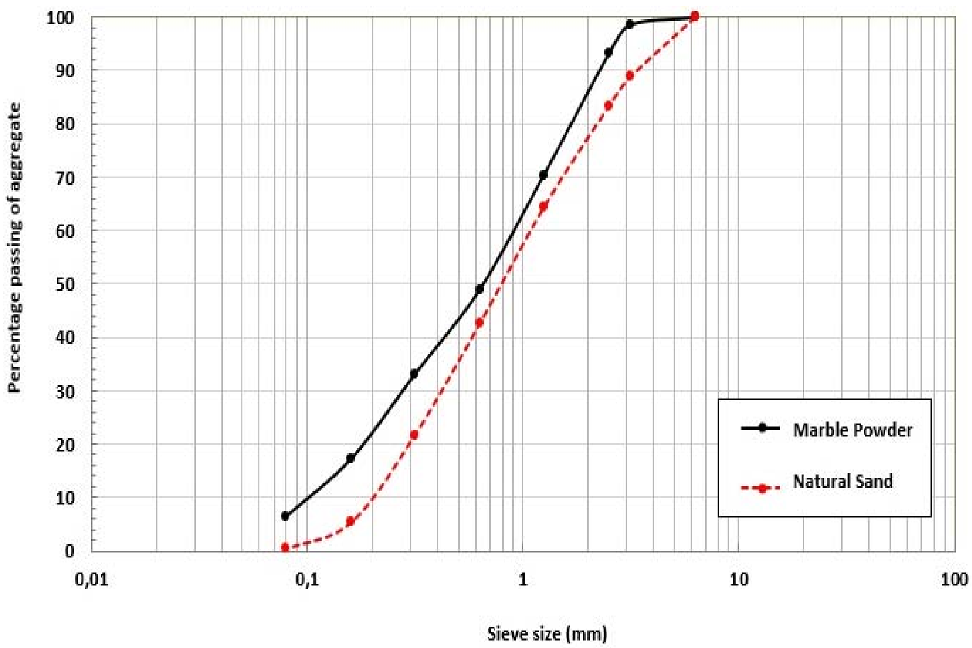


Fig.1. Particle-size distribution of the natural sand and marble powder

According to grain size distribution, the fineness modulus values of sand and marble powder are acceptable for a mortar, and natural sand can improve the mortar workability. Marble can be used to obtain a good workability and strength without segregation risks.

2.2. Mix design

The experimental study was conducted in two parts. First, the work involves studying the substitution effect of portland cement with marble powder (PM), the rheological behavior of cement pastes with fixed superplasticizer dosage and fixed water/binder ratio (W/B=*0.35*). Table 4 gives mix design of the reference cementitious paste (without marble powder). The other pastes were formulated by substituting cement with marble powder at (*0%*, *10%*, *20%*, *30%* and *50%* wt.). For the second phase of our study, fluid mortars, namely: self-compacting mortars (SCM), were elaborated according to the formulation given in Tab.5. The dosages of each mortar component have been obtained using the concrete-equivalent mortar design method [29-30]. By replacing the sand with marble at various dosages (*30%*, *50%*, *70%* and *100%* by weight of natural sand), the other mortar variants are obtained.

Table 4. Reference cementitious paste used for rheological studies.

compounds	cement paste
cement* [*g*]	*100*
limestone fillers [*g*]	*10*
water [*g*]	*38*
superplasticizer [%]	*1.3*
W/B	*0.35*

*Cement is substituted by marble powder ratio at *10%*, *20%*, *30%* and *50%* wt.

Table 5. Details of mortar mixes $\left[kg/m^3\right]$

		water	limestone fillers	cement	powder marble	naturel sand
cement substitution	**MC0**	*256*	*66*	*662*	*0*	*1373*
	MC10	*256*	*66*	*600*	*66.2*	*1373*
	MC20	*256*	*66*	*529.6*	*132.4*	*1373*
	MC30	*256*	*66*	*463.4*	*198.6*	*1373*
	MC50	*256*	*66*	*331*	*331*	*1373*
sand substitution	**MS0**	*256*	*66*	*662*	*0*	*1373*
	MS30	*256*	*66*	*662*	*412*	*961*
	MS50	*256*	*66*	*662*	*686.5*	*686.5*
	MS70	*256*	*66*	*662*	*961*	*412*
	MS100	*256*	*66*	*662*	*1373*	*0*

2.3. Test methods

a) Rheological study

All rheological tests were made by using an AR2000 rheometer from TA Instruments (Figs 2 and 3). All rheological tests were performed according to a chosen protocol which is applied on series of cement pastes samples (Fig.2). Rheological study was performed according to an adequate protocol previously used for the cement pastes by the authors [29]. It is necessary to pre-shear at $350\ S^{-1}$ for *60* seconds to homogenize the pastes and to obtain reproducible rheological measurements (laboratory conditions $T = 20 \pm 1^{\circ}C$). The measurement was carried out under the imposed shear rate of $600\ S^{-1}$.

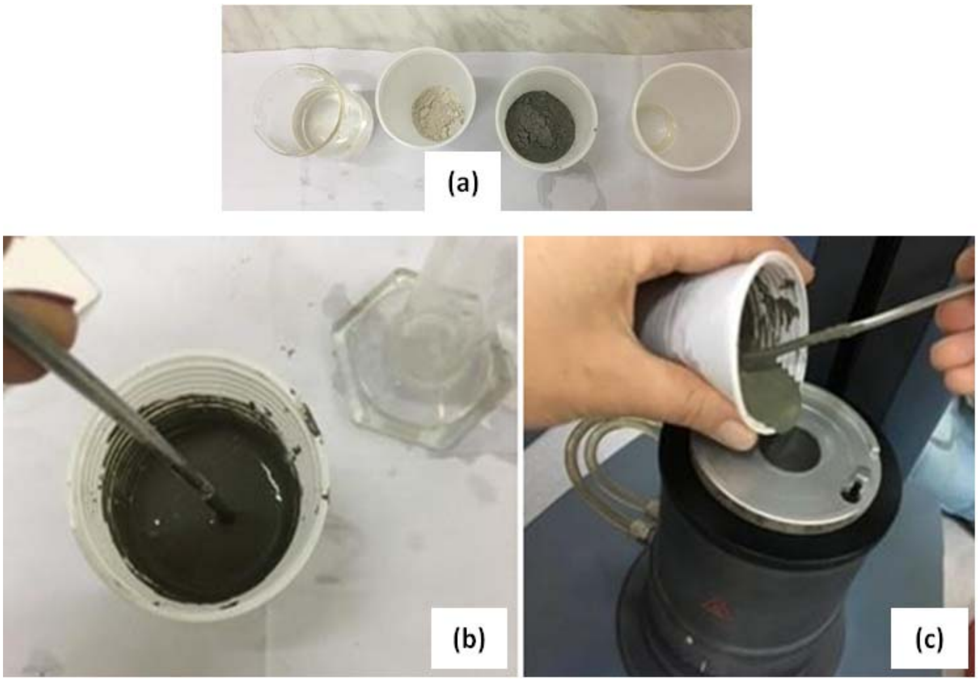


Fig.2. Preparing of cement pastes: (a) raw materials (b) mixing of cement pastes (c) preparing of rheological tests.

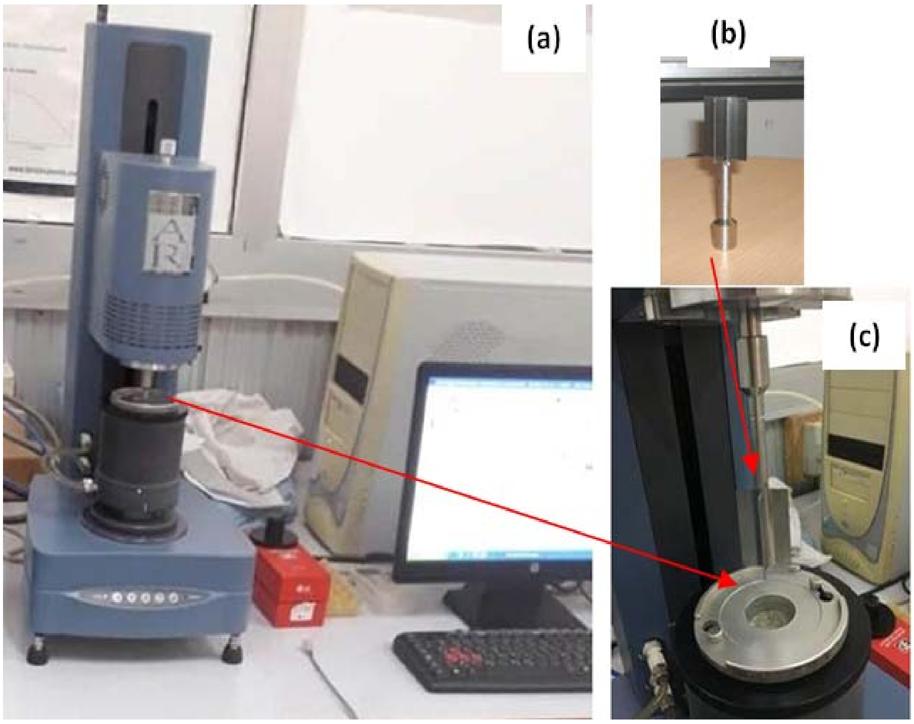


Fig.3. Rheological equipment: (a) AR2000 rheometer, (b) rotor valve geometry used, (c) cement paste sample.

b) Fresh properties

The fluidity of prepared mortar was measured after each preparation using the mini-cone slump test, by measuring the flow diameter of the mortar after slump test according to RFNARC [31].

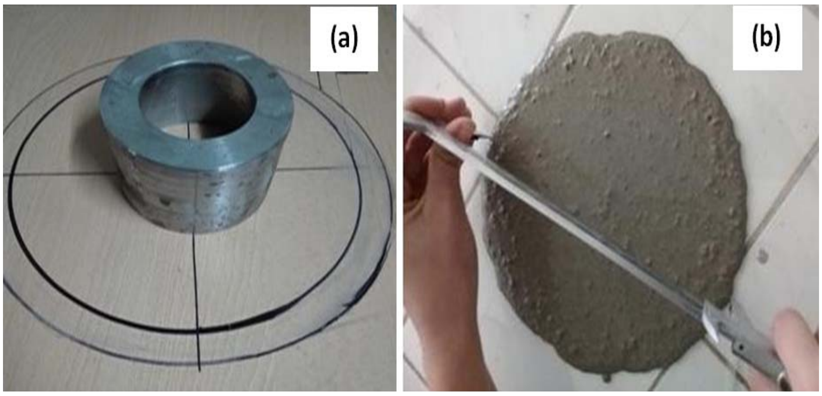


Fig.4. Slump test: (a) mini-cone (b) fluidity of mortar.

c) Mechanical properties

Prismatic specimens $40\times40\times160\ mm^3$ and cubic specimens $50\times50\times50\ mm^3$ were produced for each mortar mixture in order to assess their mechanical properties in the hardened state. These assessments were conducted using a strength testing machine. At various time intervals during storage in water at a controlled temperature of $21\pm1°C$, the specimens were subjected to tests to determine their bending strength, uniaxial compression strength, and mechanical behavior (as depicted in Fig.5a and 5b). The cubic specimens were specifically utilized to assess the compressive strength and mechanical behavior, while the prismatic samples were employed for evaluating flexural strength and conducting ultrasonic tests (as indicated in Fig.5c). Three-point bending and uniaxial compression tests were performed at *2*, *7*, and *28* days on the specimens stored in water, following the guidelines outlined in ASTM C348 and ASTM C349 [32-33]. To evaluate the mechanical behavior of the mortars, cubic samples $50\times50\times50\ mm^3$ were subjected to a uniaxial compression test.

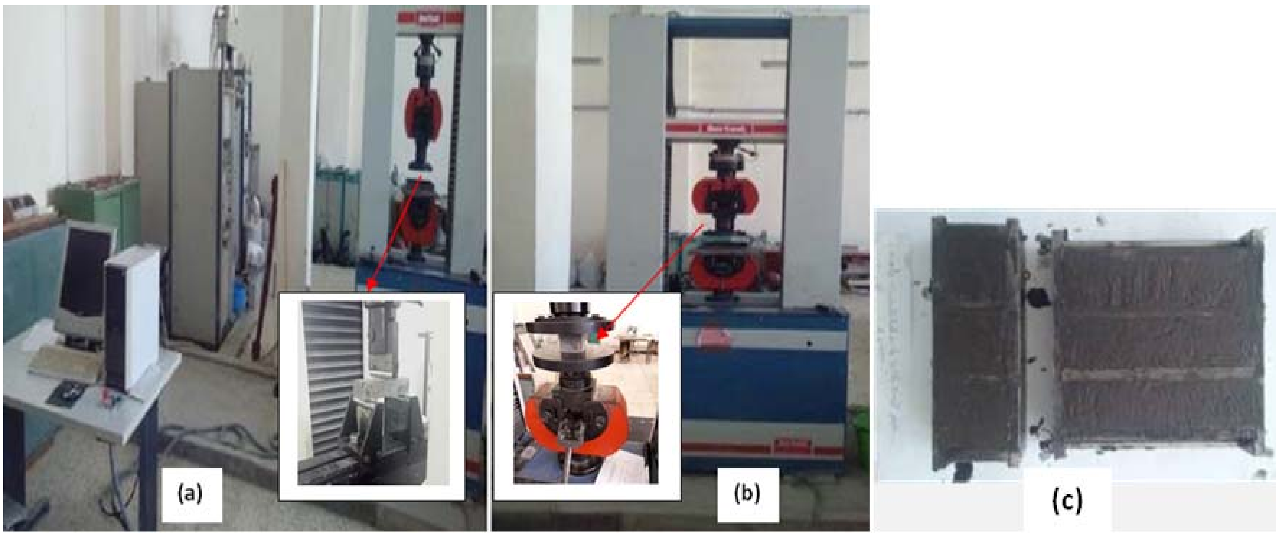


Fig.5. Mechanical testing: (a) flexural tests, (b) compressive and behavior mechanical, (c) cubic and prismatic specimens.

Fig.6. Ultrasonic pulse velocity tests carried out on prismatic samples.

3. Results and discussions

3.1.Rheological properties of studied cement pastes

Figures 7 and 8 give the variations in shear stress $\left(\tau_p\right)$ and plastic viscosity $\left(\eta_p\right)$ of cement pastes at different levels of marble powder (PM) substitution, as a function of shear rate. It is evident that both shear stress and plastic viscosity increase as the level of cement substitution with marble powder (PM) rises, up to a *20%* substitution rate. Beyond this point, a decrease of these parameters is observed at a *30%* substitution level of cement with marble powder. This trend can be attributed to the specific surface characteristics of marble powder particles, which increase the demand for water to facilitate surface wetting. Beyond the *30%* substitution mark, marble powder contributes to enhanced fluidity by reducing the viscosity of the cement paste [29, 34, 35], as demonstrated in the figure. Notably, the cementitious pastes exhibit greater fluidity and improved flow characteristics at substitution rates of *30%* and *50%* compared to those at *10%* and *20%*. Several studies have previously reported the influence of specific surface area on rheological parameters [30, 34-36].

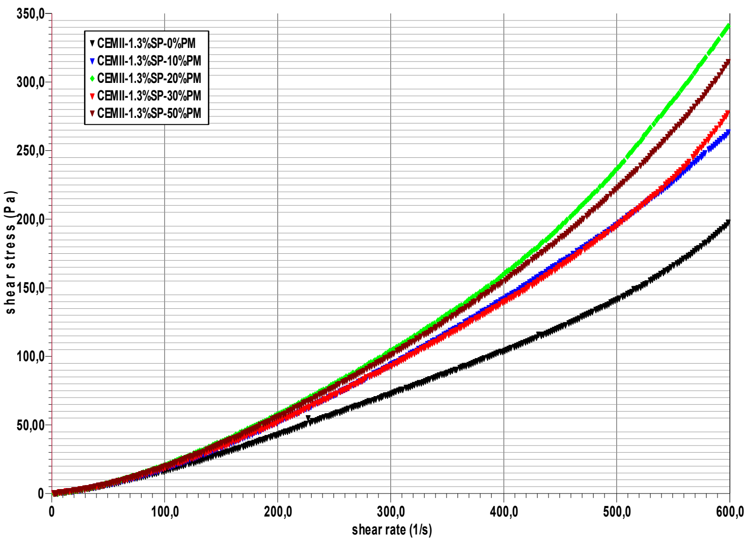


Fig.7. Share stress as a function of share rate: at different cement substitution rate by marble powder.

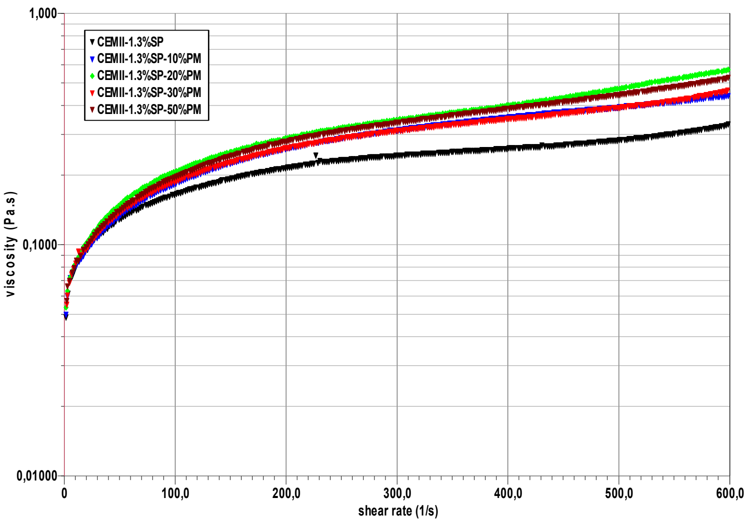


Fig.8. Plastic viscosity as a function of share rate: at different cement substitution rate by marble powder.

To study the rheological behavior of the cement pastes studied, the rheometry software TA Data Analysis of the AR2000 rheometer enabled us to identify the rheological behavior of all the cementitious pastes studied (see Fig.9). Indeed, by calibrating the models already existing in the software, the rheological behavior of these pastes could be identified. According to the results obtained, all studied cement pastes have a behavior of a plastic Bingham fluid identical to that of the Herschel-Buckley rheological model described by the Eq.(3.1), which is consistent with what exists in the literature [29, 35-37].

$$\tau_p = \tau_0 + K\dot{\gamma}^n , \tag{3.1}$$

τ_p : is shear stress; τ_0 : is yield stress; $\dot{\gamma}$: is shear rate; K: is the consistency index; n: is the flow index.

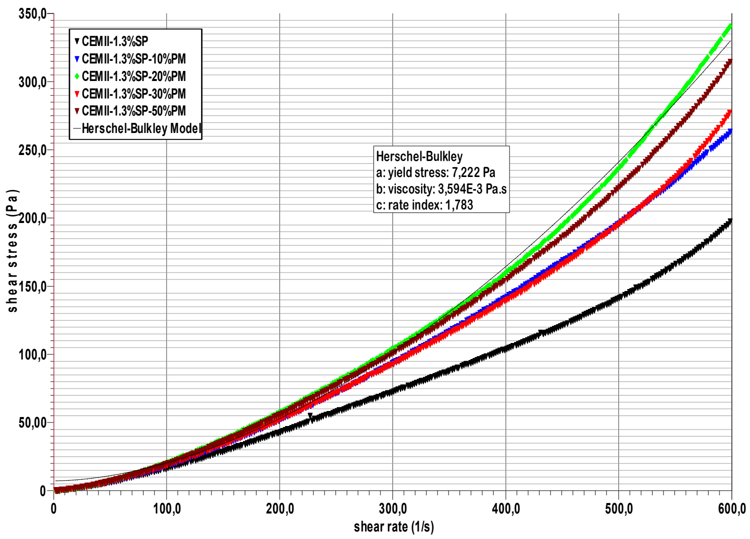


Fig.9. Rheological behavior of cement pastes studied.

3.2. Fresh and hardened properties of mortars studied

a). Fluidity of mortars

The variation in mortar fluidity for both cases of marble powder substitution, namely cement and sand, is graphically represented in Fig.10.

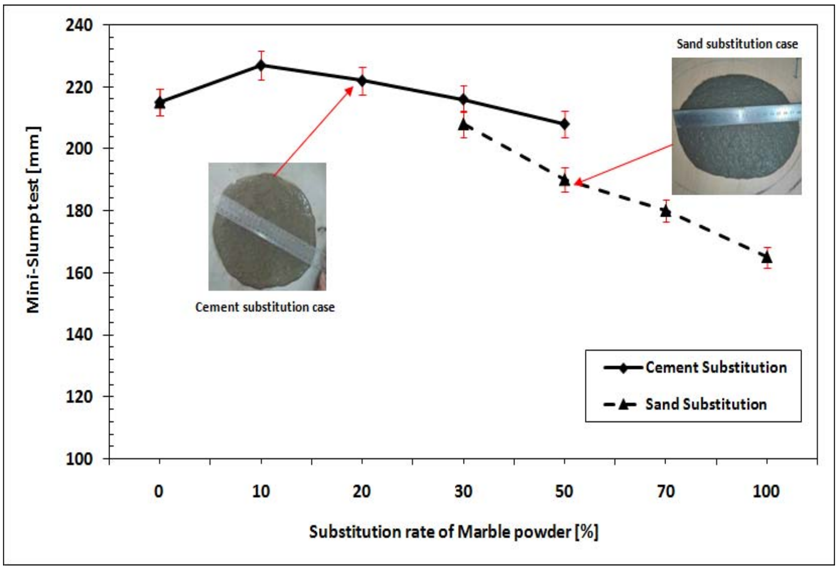


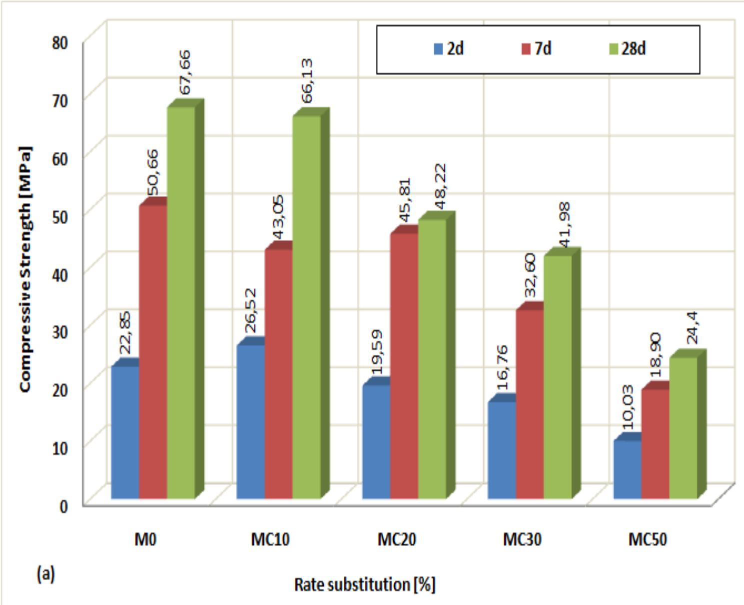
Fig.10. Fluidity of mortars based on marble powder (cement and sand substitution).

Regarding the substitution of cement with marble powder, it is worth highlighting that there is an increase in fluidity observed in the MC10 mortar, followed by the MC20 mortar. Additionally, it is evident that the fluidity of the mortar diminishes as the rate of substitution with marble powder increases. In fact, up to a *50%* substitution rate, the reduction in the cake's diameter obtained is not particularly significant. Furthermore, the mortar meets the criteria for self-compacting concretes in terms of both fluidity and ease of handling. Baboo *et al.* [38] noted a slight improvement in concrete workability when replacing cement with marble powder up to *20%*. However, other authors have reported that while flow ability decreases, it still falls within the medium range as per IS specifications. Many researchers have also observed that the most substantial enhancement in fluidity occurs up to a *10%* marble powder substitution [39-40].

However, for the case of sand substitution with marble powder, the results show that the fluidity of the mortars has increased for mortars at *30%* sand substitution, compared to the control mortar. As soon as this threshold is exceeded, a decrease in the mortar fluidity is noted with the increase of the sand substitution rate. Certainly, up to a *50%* substitution, the reduction is not significant, and the mortar satisfies the criteria for self-compacting mortars regarding fluidity and ease of application. This can be explained by the effect of specific surface of powder marble on mortar fluidity which has been reported by several authors [37-39]. Once the sand substitution with marble powder exceeds *70%*, the self-compacting mortars experience a slight reduction in their fluidity, although they still fall within the self-compacting mortar classification. It is worth mentioning that this decrease in fluidity can be compensated for by increasing the superplasticizer dosage.

b) Compressive and flexural strength

Figure 11a reveals that the compressive strength rises with increasing curing time across all the self-compacting mortars. Notably, it was observed that the strength of mortars remains consistent for M0 and MC10 compositions. However, when the marble powder content reaches *20%* in MC20, a slight decline in the compressive strength becomes apparent. The strength for MC30 and MC50 mortars decreases compared to the control mortar, regardless of the curing age of mortars. It should also be noted that the highest value of the compressive strength recorded at *28* days is around *74 MPa* for the control mortar, followed by the MC10 mortar at *66 MPa*.



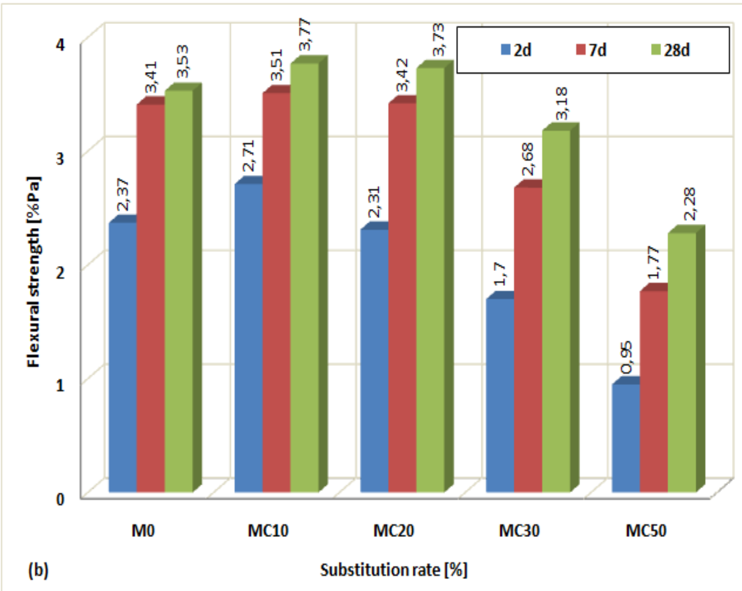


Fig.11. Mechanical strength development of studied self-compacting mortars based on marble powder: (a) compressive, (b) flexural; cement substitution case.

For other mortar mixtures, strength values remain in the category of self-compacting mortars, except for the MC50 mortar where the recorded strength is *24 MPa*, that this value is suitable for construction. It has been reported that up to *10%* marble powder used as cement, can give a better mortar in terms of fluidity and compressive strength. The substitution level of cement with marble powder can be further extended up to *20%*, as *28*-day compressive strength of MC20 mortar was a good value of strength with *48 MPa*. The same observation with a level of *20%* replacement of cement by marble powder was made in [17, 40-41].

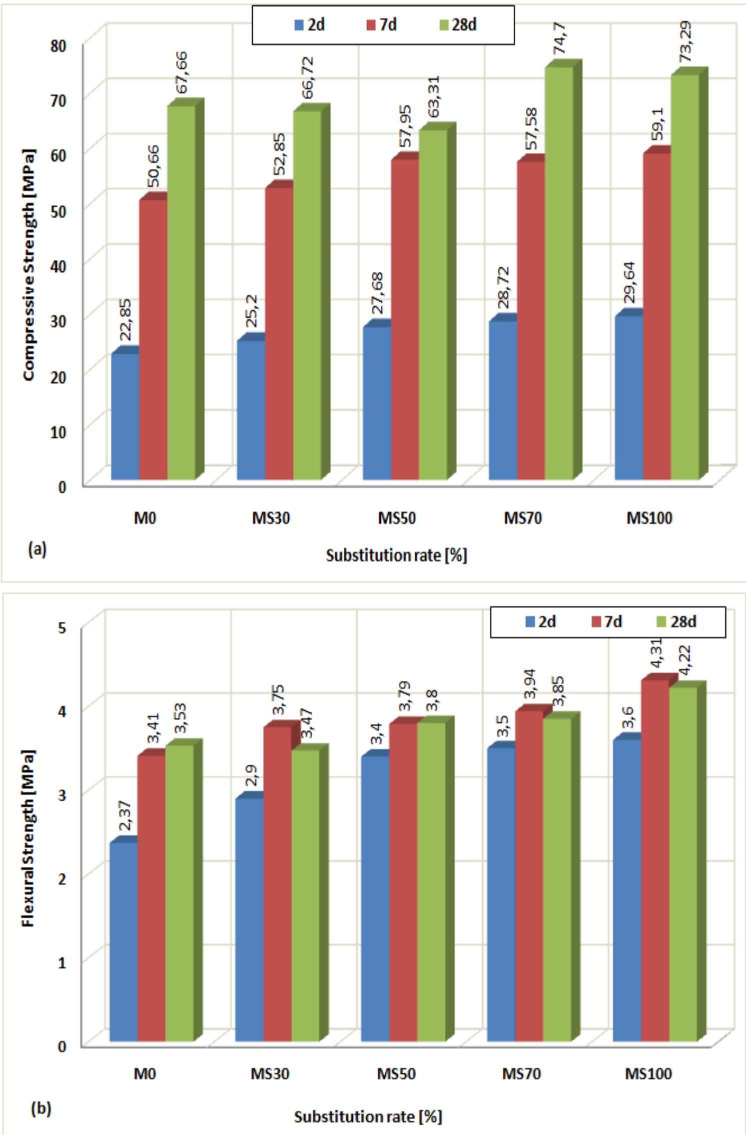


Fig.12. Mechanical strength development of studied self-compacting mortars based on marble powder: (a) compressive, (b) flexural; sand substitution case.

Furthermore, it is noteworthy that the flexural strength exhibited improvements in both MC10 and MC20 mortars when compared to the control mortar, as depicted in Fig.11b. However, after reaching a *20%* substitution level of cement with marble powder, a reduction in flexural strength was observed. It was also observed that up to a *30%* cement substitution with marble powder, the flexural strength of the mortar remained satisfactory and closely approached that achieved by the control mortar.

The effect of marble powder as sand replacement on the compressive and flexural strength of studied self-compacting mortars is given in Figs 12a and 12b.

Previous research focused on incorporating marble powder as a fine aggregate in mortars or concretes has consistently demonstrated the highly advantageous impact of using marble powder in lieu of sand, particularly regarding mechanical performance and material durability [11, 12]. Our findings clearly underscore the effectiveness of marble powder when employed as a sand replacement. Indeed, we observed a noticeable enhancement in both compressive and flexural strength across all curing periods when marble powder was used as a complete sand substitute, with a remarkable *10%* improvement in compressive strength compared to the control mortar.

Furthermore, a self-compacting mortar formulated with total sand substitution by marble powder can achieve an impressive compressive strength of approximately *74 MPa* at *28* days, alongside a flexural strength of approximately *4.22 MPa*. In comparison with results obtained from existing literature [39-42], it can be concluded that this marble powder-based mortar demonstrate a significant potential as a sustainable alternative to traditional cement mortars in construction applications.

3.3. Mechanical behavior of studied mortars

The mechanical characteristics of each mortar mixture were determined through experimental analysis, as illustrated by the stress-strain curve in Fig.13.

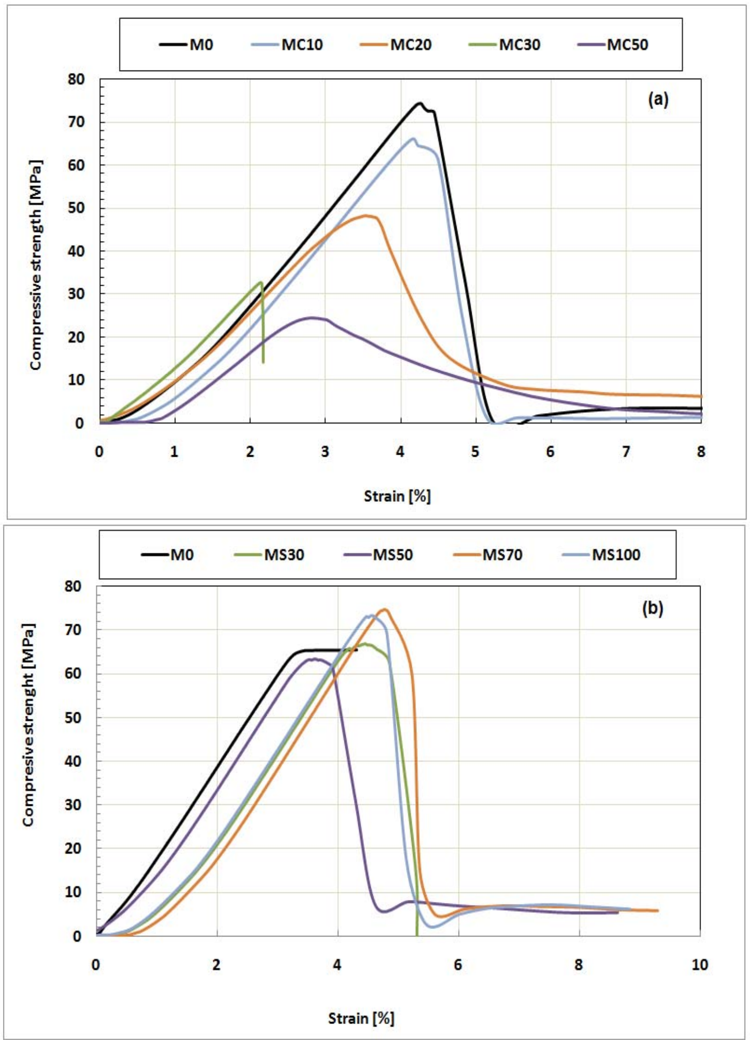


Fig.13. Stress-strain behavior of powder marble-based mortars at *28*-day, (a) cement substitution, (b) sand substitution.

All the mortars under investigation exhibited a brittle behavior, characterized by an initial elastic range followed by rupture at a maximum load. However, it is worth noting that a mortar (MC10), featuring a *10%* substitution of cement with marble powder, displayed identical mechanical behavior to the reference mortar MC0, as depicted in Fig.13a. On the other hand, as the cement substitution with marble powder increased up to *30%*, the mortar's mechanical behavior continued to exhibit brittleness, albeit with a reduction in maximum load and less deformation when compared to the MC0 mortar, as shown in Fig.10a [43-46].

In the context of replacing natural sand with marble powder, the findings related to mechanical behavior are quite intriguing. In fact, when substituting sand with marble powder up to *70%*, the mortar's mechanical behavior remains brittle and mirrors that of the control mortar, as evidenced in Fig.13b. A marginal enhancement in maximum load is noticeable, accompanied by reduced deformation compared to the control mortar, as reported in previous studies [42].

3.4. Ultrasonic testing of studied mortars

Ultrasonic pulse velocity (UPV) was analyzed across all mortar samples to assess mortar density and to gauge the influence of marble powder. Figure 14 showcases the sound velocity results in relation to the substitution of cement and sand with marble powder, along with curing time at *28* days. The results show a slight decrease in the sound velocity of the mortars beyond *30%* substitution of cement with marble powder compared to the reference mortar specimens. However, it was noted that a clear improvement in mortar compactness is reflected by an increase in sound velocity when sand is fully substituted with marble powder. It should also be noted that the good adhesion of the marble grains with the cement paste favors well the compactness of the mortars (see Fig.14).

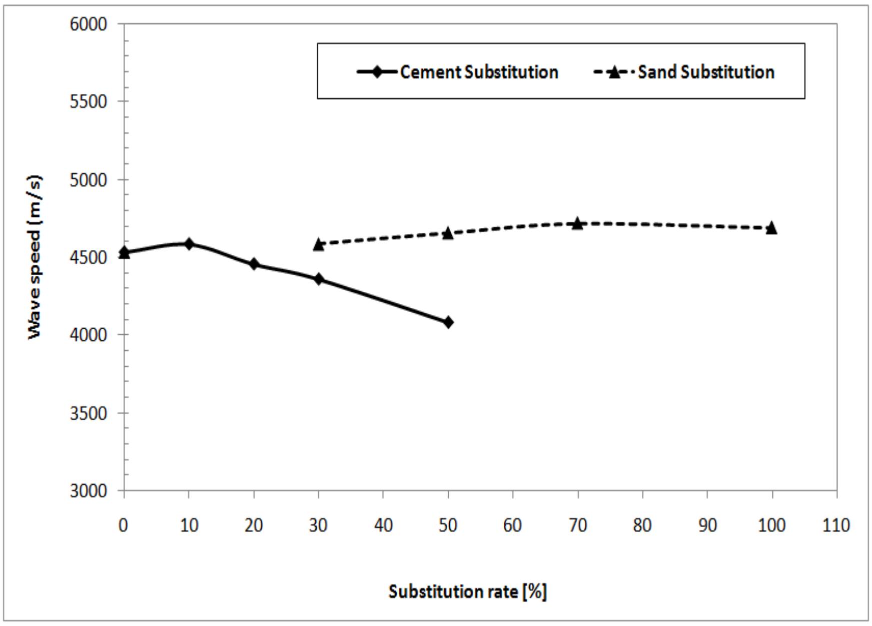


Fig.14. Ultrasonic pulse velocity evolution of powder marble-based mortars measured at *28*-days.

4. Conclusion

The study focused on the use of marble powder as an additional material and a fine aggregate in the manufacturing of fluid mortar. The rheological and mechanical behavior of self-compacting mortars made with powder marble (in two substitution cases: cement and natural sand) were reviewed and compared. it can be concluded that;

Beyond *30%* of cement substitution, the marble powder has improved the fluidity by decreasing the shear stress and viscosity of the cement paste. The effect of the specific surface of powder marble on rheological parameters has been observed. Beyond *70%* sand substitution with marble powder, the self-compacting mortars lose a little their fluidity, but they remain in the category of self-compacting mortars.

Up to *70%* replacement of natural sand with marble powder, a self-compacting mortar with acceptable fresh characteristics can be obtained. Indeed, an improvement of compressive and flexural strength at all curing age was observed for all self-compacting mortars up to *100%* sand substitution. However, a *20%* reduction in cement consumption translates into fluid mortars with acceptable characteristics for construction.

At a *30%* substitution rate of cement with marble powder, the mechanical performance of the mortar continues to display brittleness. Nevertheless, there is a noticeable decrease in the maximum load and reduced deformation compared to the control mortar. In the case of sand substitution with marble powder, the findings regarding mechanical behavior are particularly noteworthy. Remarkably, up to a *70%* substitution rate of sand with marble powder, the mortar's mechanical behavior remains brittle and mirrors that of the control mortar.

Acknowledgements

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Nomenclature

PC – Portland cement
PM – marble powder
SCM – self-compacting mortar
SCC – self-compacting concrete
W/B – water/binder ratio
MC – mortar with cement substitution
MS – mortar with sand substitution
τ_p – shear stress
$\dot{\gamma}^n$ – shear rate
η_p – plastic viscosity
τ_0 – yield stress
K – consistency index
n – flow index

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