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Synopsis: A powder obtained as a by-product of marble sawing and shaping was characterized from a chemical and physical point of view in order to use it as mineral addition for mortars and concretes, especially for self-compacting concrete. This marble powder showed a very high Blaine fineness value of about $1.5 \text{ m}^2/\text{g}$, with 90% of particles passing 50 μm -sieve and 50% under 7 μm . For rheological studies, several cement pastes were prepared using marble powder, with and without the addition of an acrylic-based superplasticizer and by varying the water to cementitious materials ratio. In order to evaluate the effects of the marble powder on mechanical behavior, many different mortar mixtures were tested, all prepared with sand to cement ratio of 3:1 at about the same workability. Mixtures were evaluated based upon cement or sand substitution by the marble powder. Results obtained show that 10% substitution of sand by the marble powder provided maximum compressive strength at about the same workability.

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INTRODUCTION

A marble powder, obtained as a by-product of marble sawing and shaping, was characterized from a physical and chemical point of view for evaluating the possibility of using it in mortar and concrete production.

Mineral additions in general influence the performance of fresh concrete and mortar. Therefore, a rheological study was carried out on various cement pastes prepared with marble powder in combination with cement, and eventually, also, with a superplasticizing admixture. In particular, the goal was to investigate the influence of marble powder on rheological properties of cement pastes for predicting the effect of its addition on self-compacting concrete mixtures (1-6).

MATERIALS

Portland Cement

A commercial portland-limestone blended cement type CEM II/A-L 42.5R according to the European Standards EN-197/1 was used. The Blaine fineness of cement was $0.41 \text{ m}^2/\text{g}$ and its specific gravity was 3.05 kg/m^3 .

Aggregate

Natural sand (5 mm maximum size, with 0.9 % passing the $75 \mu\text{m}$ sieve) was used. Its bulk specific gravity (SSD condition) was 2.62 kg/m^3 and its SSD condition water absorption of 3.0 %.

Chemical Admixture

In some cases, a water-reducing admixture was added to mixtures. It was constituted of a carboxylic acrylic ester polymer in the form of 30% aqueous solution.

Mineral Addition

A marble powder was used, which was obtained as a by-product of marble sawing and shaping. Its specific gravity was 2.55 kg/m^3 and the value of Blaine fineness was $1.50 \text{ m}^2/\text{g}$. It can be observed that the marble powder had a high specific surface area; this could mean that its addition should confer more cohesiveness to mortars and concretes (4, 5).

For better physical characterization of the marble powder, its grain size distribution was performed using laser diffraction. From the graph shown in Fig. 1, it can be observed that 50% of particles had a diameter of $7 \mu\text{m}$ ($d_{50} = 7 \mu\text{m}$) and 90% of particles had a diameter lower than $50 \mu\text{m}$ ($d_{90} = 50 \mu\text{m}$).

The marble powder is produced as “slurry”, a mud made of powder and water. Therefore, for its use in concrete it is important to know how much water is contained in the slurry, by drying it and registering the weight loss related to water evaporation. A known weight of slurry was put in an oven to dry at a temperature of $110 \pm 5^\circ\text{C}$. At fixed intervals (1 hour, 4 hours, 24 hours, 48 hours, and 72 hours) the weight loss was registered with the aim to reach a constant weight. The results obtained are reported in Fig. 2. It is evident from the graph that the sample loses water quickly and reaches the constant weight after about 24 hours.

In order to characterize the marble powder from a chemical point of view, thermal analysis and X-ray diffraction were carried out. Thermal analysis carried out show that the examined material contains about 66% of calcium carbonate, CaCO_3 . As a matter of fact, as it can be observed in Fig. 3, a sharp weight loss (corresponding to the flex of the DTA curve) occurs from about 730°C to 900°C while strong heat absorption was detected, since the decomposition reaction of calcite is endothermic. X-ray diffraction analysis, reported in Fig. 4, show the presence of quartz, which could be estimated at about 3%, and ankerite (ferroan dolomite) at about 2%. The remaining part of the marble powder may consist of almost amorphous silica or silicates, coming from natural stones other than marble, whose

low crystallinity (making them mostly undetectable by X-ray diffraction) may be due to mechanical processing (sawing and shaping).

RHEOLOGICAL BEHAVIOR OF CEMENT PASTES

The study of the rheological behavior of cement pastes is an essential step for the evaluation of fresh concrete behavior and for the optimization of self-compacting concretes (7). For this purpose, eight cement pastes were prepared by varying the water to cement ratio (0.4 - 0.5), the amount of marble powder addition (10% and 20% by weight of cement), the basis for adding marble powder (as either cement or sand replacement), and by eventually adding a superplasticizing admixture (at a dosage of 0.5% by weight of very fine materials, i. e., cement plus marble powder). The proportions of these paste mixtures are shown in Table 1.

The rheological behavior of these cement pastes was determined at 15 minutes after ingredients mixing, and then every 20 minutes up to 1 hour.

The apparatus was a rotating rheometer based on coaxial rotary cylinders with a slowly increasing shear rate (D), ranging from 1 to 100 s^{-1} . Bui et al. (7) found that the rate of $1 \div 100 \text{ s}^{-1}$ was the most suitable for rheological model of concretes, while higher rotation rates were considered too fast and rates limited to 50 s^{-1} did not produce consistent results.

The walls of the concentric cylinders were not smooth but roughened in order to reduce (if not completely eliminate) the “slip” phenomenon; i. e., the development of a water-rich layer close to the inner surface of the rotating cylinder, which produces a lubricating effect, making flow easier, and not representative of the bulk material (8).

The rheological behavior was described by means of the Bingham flow model (Fig. 5):

$$\tau = \tau_y + \eta \cdot D$$

where τ is the shear stress [Pa], τ_y is the yield stress [Pa], η is the plastic viscosity [Pa·s], and D is the shear rate [s^{-1}].

The slope of the down-curve (decreasing shear rate) was used to calculate the plastic viscosity, while the intercept at zero shear rate was used to calculate the yield stress.

In Fig. 6, the measured yield stress values are plotted as a function of time. It is evident that the pastes prepared with marble powder, superplasticizing admixture, and water/cement of 0.4 showed the highest values of the yield stress, more than 40 Pa. Quite high values also were obtained for the cement pastes prepared with marble powder and water/cement of 0.5 but in absence of superplasticizer. On the other hand, when the water to cement ratio was 0.5 and the superplasticizing admixture was added, even at the low dosage (0.5% by weight of cement), the yield stress was very low, less than zero, thus implying low cohesiveness of the related mortars and concretes, particularly for self-compacting concretes.

In Fig. 7 the measured plastic viscosity values are plotted as a function of time. Also in this case, the same hierarchy of yield stress values was maintained among the various cement pastes.

For maximum segregation resistance, the yield stress of the paste should be high (1, 2, 3, 7) and the difference in density between the aggregate and the paste should be low. If the density of the aggregate particle is greater than the density of the cement paste, segregation will occur to some extent. However, if the plastic viscosity of the matrix were high enough the velocity of the falling aggregate particle would be so slow that segregation would be avoided (7). On the basis of the results reported in Fig. 6 and Fig. 7, it can be seen that in the presence of a superplasticizing admixture (e. g., for preparing self-compacting concrete), the addition of marble powder is very effective in improving segregation resistance provided that water/cement is lower than 0.5. Otherwise, a viscosity-modifying agent should be added to the mixture for adjusting its rheological behavior (10, 11).

Thixotropy is the property of certain gels, such as cement paste, which are rigid when left standing but increase their fluidity when put into movement. Fig. 8 shows the measured thixotropy values, plotted as a function of time, where thixotropy was calculated as the area included between the

up-curve and the down-curve (Fig. 5). This measure can give an estimate of the energy necessary to move mortars and/or concretes and even an estimate of the lateral formwork pressure that concrete, especially self-compacting concrete, will exert after placing, the lower the formwork pressure the higher the thixotropy value (12). In this case, particularly with increasing time, the difference among the various cement pastes is slight and the value of the energy loss is generally quite low.

MORTAR MIXTURES PROPORTIONS

The mortar mixtures proportions are reported in Table 2. All mortars were prepared with the same ratio of marble powder to cement (ratio of 3:1).

Consistency of fresh mortars was evaluated through the use of a shaking table by measuring the mortar flow. The test was carried out according to the procedure reported in the Italian Norm UNI 7044-72. In this work the flow measure was the same for each mortar and its value was equal to 13. On the other hand, the water content and, consequently, the water to cement ratio are different for the various mortars, keeping constant the fresh mortar fluidity.

Marble powder was used as a 10% replacement of either cement or sand, with or without an acrylic based superplasticizing admixture, which was added at a dosage of 0.5 % by weight of cement.

PREPARATION AND CURING OF SPECIMENS

Prismatic specimens, 40 by 40 by 160 mm in size, were manufactured for mechanical tests; these specimens were cast in stainless steel forms and wet cured at 20°C until the time of test.

COMPRESSION TESTS: RESULTS AND DISCUSSION

Mechanical behavior of mortars prepared without chemical admixture was studied by compression tests at curing times of 3, 7, 28 and 56 days and the results obtained are reported in Fig. 9. It can be noticed that 10% replacement of either cement or sand with marble powder caused a 10 - 20% decrease of the mortar compressive strength, higher for longer curing times. However, marble powder used as the replacement of sand performed better than the case of the marble powder used as the replacement of cement.

In addition, compression tests of mortars prepared with superplasticizing admixture were carried out at curing times of 3, 7, and 28 days. The results obtained are reported in Fig. 10. In this case 10% replacement of either cement or sand with marble powder did not cause any evident loss of strength after 28 days of curing. In fact, the use of marble powder in combination with superplasticizing admixture allowed as compensating the high water absorption of marble powder itself and, consequently, the water to cement ratio could be maintained sufficiently low in order not to compromise the mortar mechanical strength. Again, marble powder used as the replacement of sand performed equal to or better than the case of the marble powder used as the replacement of cement.

CONCLUSIONS

Due to its high fineness of the marble powder, it proved to be very effective in assuring very good cohesiveness of mortar and concrete, even in the presence of superplasticizing admixture, provided that water to cement ratio was adequately low.

In terms of mechanical performance, 10% substitution of sand by the marble powder in the presence of a superplasticizing admixture provided maximum compressive strength at the same workability level.

Next step of this continuing experimental work is preparation of concretes, fluid and self-compacting, by using the marble powder.

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Table 1. Paste mixture proportions.

CEMENT PASTE	W/C	Amount (g)			
		Cement	Water	Marble Powder (MP)	Admixture (ADM)
CEM (0.5)	0.5	100	50	-	-
CEM+MP10 (0.5)	0.5	90	50	10	-
CEM+MP20 (0.5)	0.5	80	50	20	-
CEM+ADM (0.5)	0.5	100	50	-	0.5
CEM+ADM (0.4)	0.4	100	40	-	0.5
CEM+ADM+MP20 (0.5)	0.5	80	50	20	0.5
CEM+ADM+MP20 (0.4)	0.4	80	40	20	0.5
CEM+ADM+MP10 (0.4)	0.4	90	40	10	0.5

Table 2. Mortar mixture proportions.

Mixture	REF	10% CEM	10% SAND	REF+A	10% CEM+A	10% SAND+A
W/C	0.61	0.68	0.59	0.48	0.49	0.53
Mixture proportions, kg/m ³						
Water	275	276	266	220	200	240
Cement	450	405	450	450	405	450
Sand	1350	1350	1215	1350	1350	1215
Marble Powder	-	45	135	-	45	135
Chemical Admixture	-	-	-	2.25	2.02	2.25

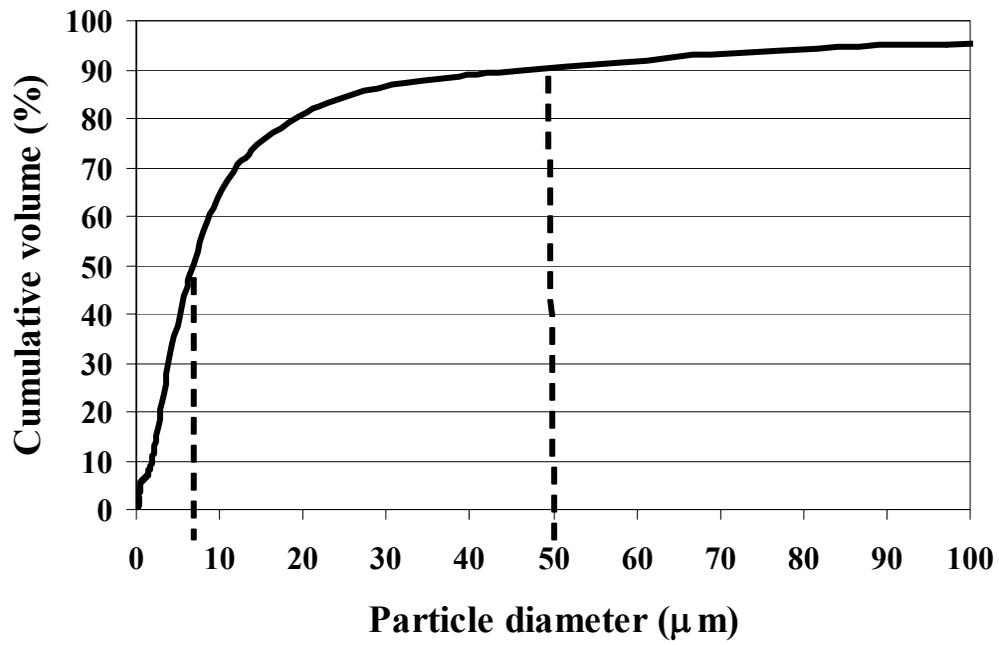


Fig. 1 – Grain size distribution of the marble powder by laser diffraction.

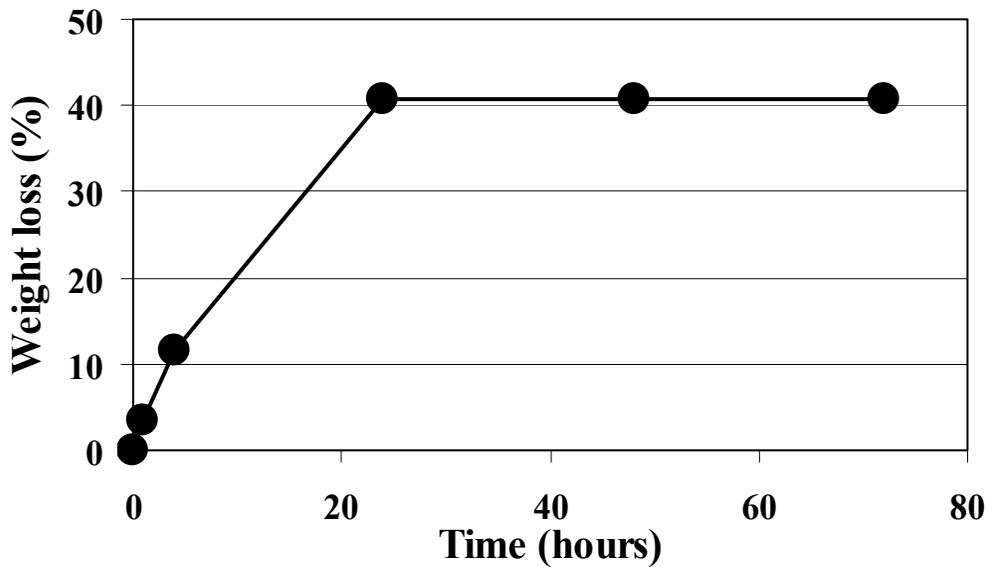


Fig. 2 – Weight loss of the marble powder slurry verses time.

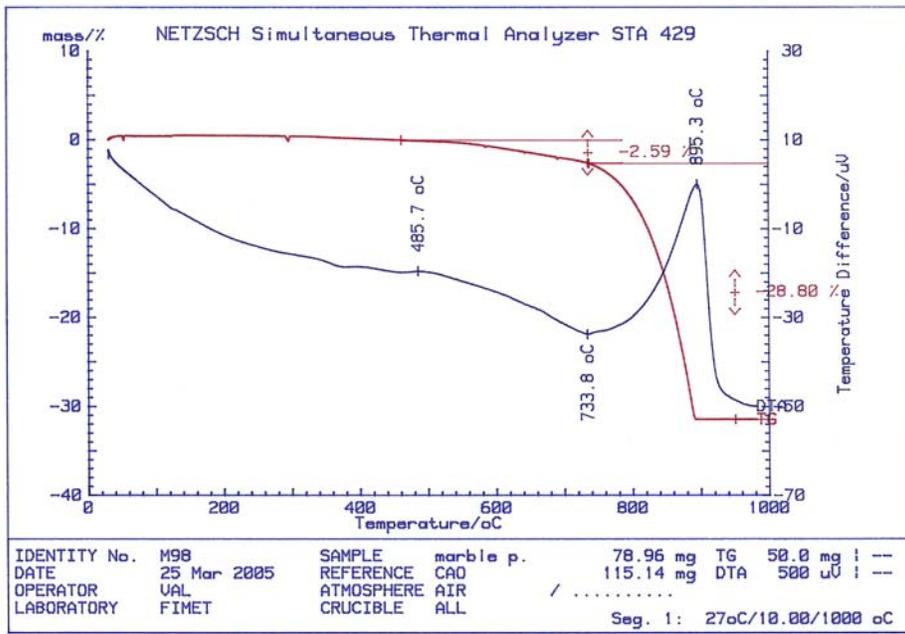


Fig. 3 – Results of the thermogravimetric (TG) and differential thermal analysis (DTA) of the marble powder.

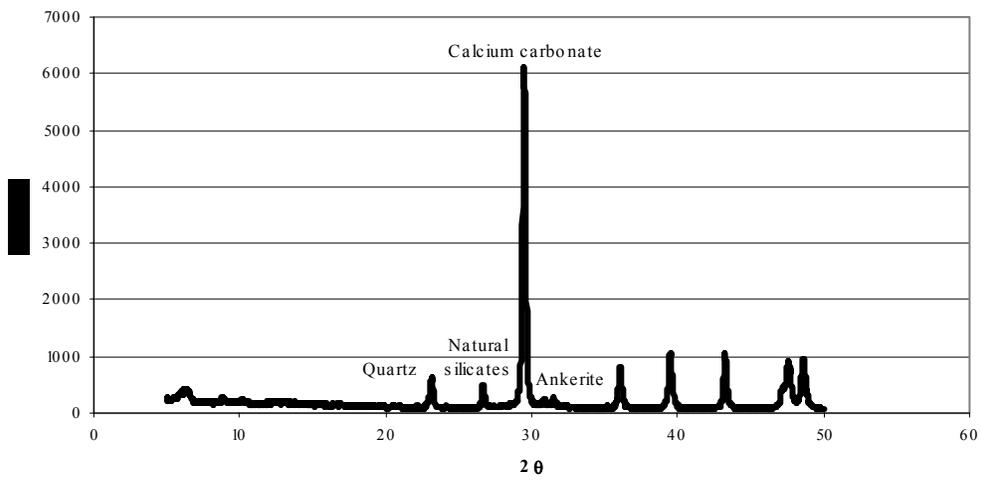


Fig. 4 – X-ray diffraction of the marble powder.

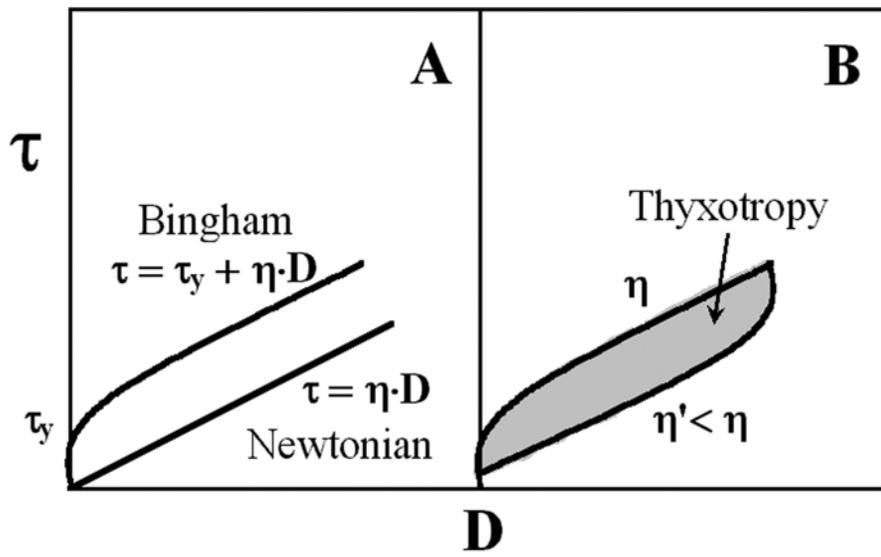


Fig. 5 - Typical shear stress (τ) versus shear rate (D) of Newtonian or Bingham fluid (A); thixotropy measured by the hysteresis area (B).

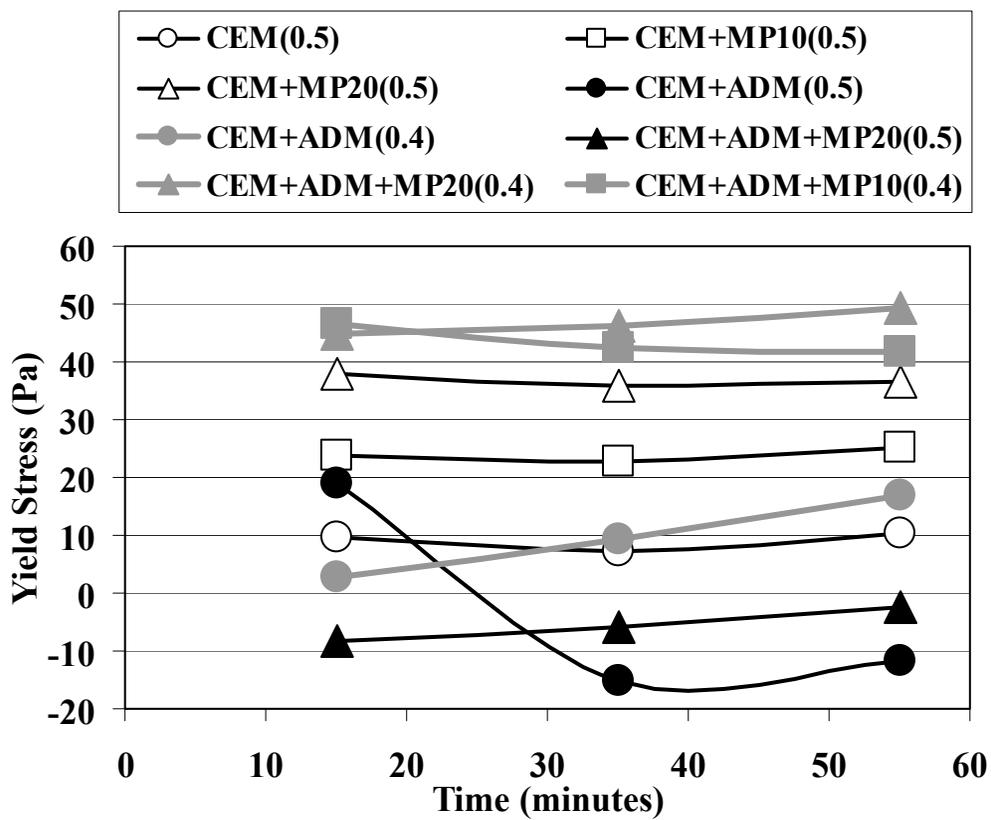


Fig. 6 - Yield Stress values versus time.

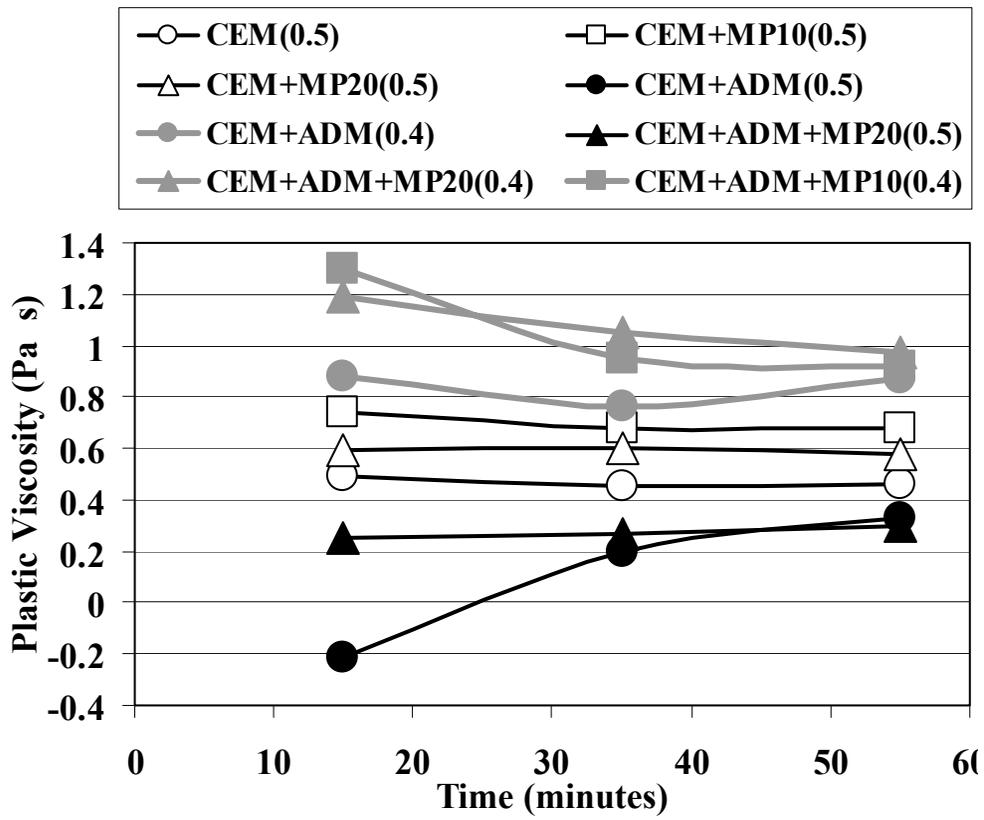


Fig. 7 - Plastic viscosity values verses time.

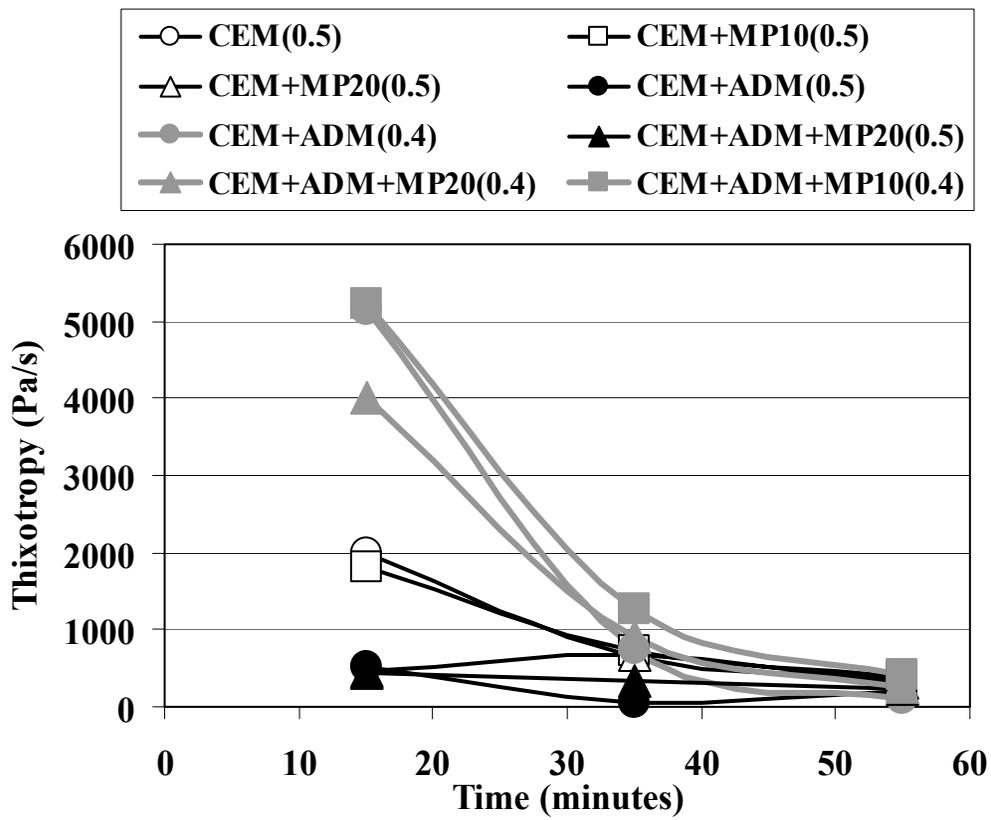


Fig. 8 - Thixotropy values verses time.

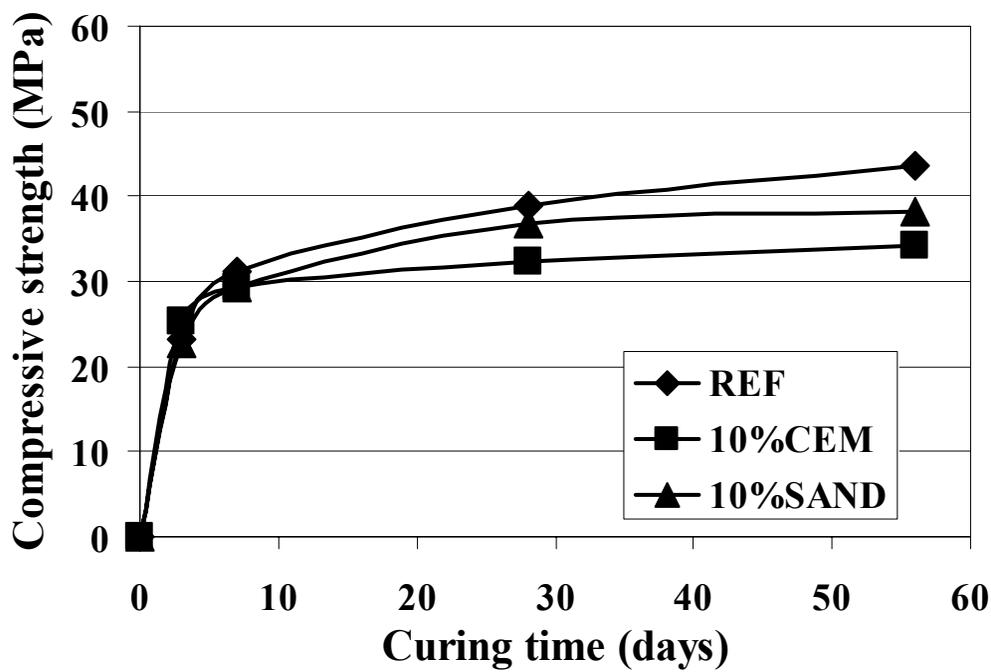


Fig. 9 - Compressive strengths verses curing time for cement mortars without superplasticizer.

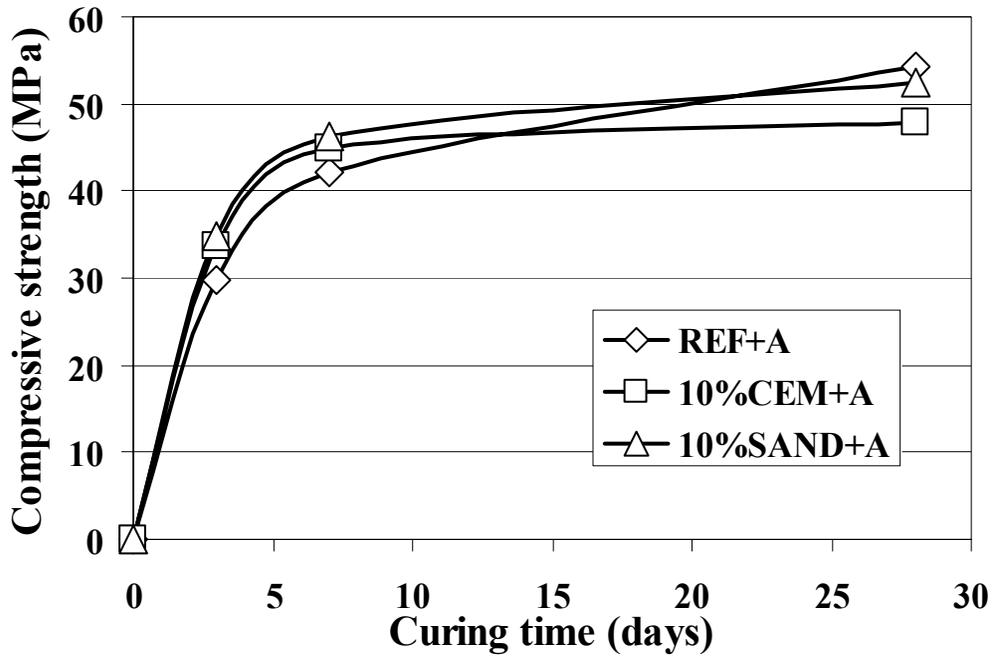


Fig. 10 - Compressive strengths verses curing time for cement mortars with superplasticizer.