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Fresh and hardened properties of cement mortars using marble sludge fines and cement sludge fines

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Abstract

The construction sector could provide solutions for the safe utilization of industrial by-products as construction materials, if proper characterization and control of the materials properties is undertaken. Under this consideration, fines produced from marble cutting and fines produced from concrete truck washing were investigated as fine material for use in cement mortars. Both these by-products are produced in large amounts in the form of sludge. Marble Sludge Fines (MSF) and Cement Sludge Fines (CSF) were characterized in terms of fineness, density, chemical analysis and suitability for use with cement. Mortars with variable rate (10%, 20% and 30%) of cement substitution with MSF or CSF were tested and compared to a reference cement mortar in respect to their fresh and hardened properties. Packing ability and viscosity were measured in fresh mortars, while strength development, water absorption and porosity were measured in hardened mortars. The results confirm the suitability of both as filler material; although MSF performed better regarding fresh mortar properties and CSF showed better results regarding strength development.

Keywords: cement mortars; marble sludge fines; cement sludge fines; industrial by-products.

1. Introduction

By-products from industrial processes are being produced in large amounts around the world and their handling, processing and disposal is taking up considerable effort and increasing financial and environmental cost [1,2]. It is estimated that the amount of waste generated during cutting, sawing and shaping of ornamental marble accounts for 20-25% of the total marble processed [3], resulting in huge amounts of marble waste. Marble waste is either produced as large pieces that are left over in primary cutting, which find various uses as aggregates [4,5]; or as fine material produced in wet condition from sawing and shaping, named marble powder, marble dust or marble sludge fines (MSF), which is being investigated as filler for construction applications [6–8]. Concrete production, on the other hand, is also generating large amounts of waste; depending on local practices, it is estimated that 1~9% of fresh concrete is returned to the concrete batching plant [9]. This accounts for huge amounts of waste as the annual global consumption of concrete exceeds 10 billion tons [10]. The returned fresh concrete has several possible uses, including immediate recycling or use as a source for recycled concrete aggregates [11–13], however, the material adhered to the inside of the barrel is washed and stored in sedimentation tanks, as it has high alkalinity [14]. The alkaline water from the sedimentation tanks finds some use replacing fresh water in concrete production [15–17] and the remaining material, named cement waste slurry or cement sludge fines (CSF) finds little or no use.

Both MSF and CSF consist of fine particles and are produced in wet condition, which makes handling and processing more difficult, since some processing is usually required prior to transportation or use [18].

Their use as fine material, however, could provide an alternative for the construction industry, which consumes considerable amounts of filler material from natural sources [19]. Relevant literature from Corinaldesi et al. [20] reported that marble powder showed the filler effect that improves early age strength in mortars and recommended 10% as optimum cement substitution rate. Hameed et al. [21] found that marble sludge fines used as filler in self-compacting concrete decreased viscosity and improved segregation resistance, suggesting a limited use of 15% by total powder weight. Omar et al. [22] used marble powder as sand replacement in concrete and verified its filler effect, while they also reported a strength increase at 15% sand replacement rate. Aliabdo et al. [23] tested marble dust as cement or as sand replacement at various rates up to 15% wt. and confirmed its compatibility for use with cement, while they reported a decrease in cement mortar and concrete strength when MSF was used as cement replacement and an increase in concrete strength when MSF was used as sand replacement. Ulubeyli et al. [24] used the waste marble in the conventional concrete as binder or fine/coarse aggregate and it positively affected properties of hardened concrete, whereas in self-compacting concrete, the increase of waste marble replacement ratios decreased its mechanical properties. Sardinha et al. [25] studied the durability of concrete with MSF as cement replacement and reported that it generally decreased with increased MSF use, while at low replacement levels (5%) the durability reduction could be offset with the use of superplasticizers. It must be noted though, that the authors used a rather coarse MSF, with only 27.5% passing through the 125 μm sieve. Mashaly et al. [26] used MSF sized below 100 μm as cement replacement in concrete and reported an increase in water demand and also an increase in mechanical strength at replacement rates up to 20%.

The research on CSF properties and utilization is clearly more limited: Zervaki et al. [27] found an improvement in compressive strength of mortars when untreated CSF was used, without any removal of its free water content. However, when it exceeded a certain concentration, it led to loss of workability. Moreover, dry de-agglomerated CSF, when used in mortars as a cement replacement at 2% ratio, caused a slight improvement in compressive strength, due to the filler effect. In higher replacement ratios a slight decrease of compressive strength was detected. Audo et al. [28] tested the incorporation of CSF plants as limestone fillers in mortars and reported a decrease in the workability of the fresh mortar and a variability in the compressive strength of the hardened mortars, between -30% and +17%, compared to the reference. Hossain et al. [29] reported that the concrete slurry waste generated from ready-mixed concrete plants is classified as a corrosive hazardous material and used the accelerated carbonation technique in order to produced hardened products. Correia et al. [30] replaced natural fine aggregates with the fresh concrete waste (FCW). The recycled new concretes were in various water/cement ratios and the results showed that the fresh concrete workability worsened with the increase in FCW content but the water absorption (5–10 wt.%), 7-day compressive strength (26–36 MPa) and 28-day compressive strength (32–44 MPa) remained within the specified ranges.

Following existing literature, the present study attempts to properly introduce MSF and CSF in cement-based mortars as supplementary cementitious materials. The fineness of the two by-products shows that their optimal use would be as cement-replacement fillers, provided that they are suitable for such a use. Hence, material characterization is carried out firstly, followed by testing cement mortars with cement replacement rates of up to 30%. The suitability for use as filler is investigated by a series of physical and analytical tests, including fineness determination and chemical analysis. Regarding mortars, 10%, 20%, and 30% wt. cement replacement rates were chosen for both materials, as these rates are sufficient to describe the effect of filler use with cement. Since the fresh mortar properties were expected to be affected, workability, viscosity and packing ability were determined, while the hardened properties investigated were compressive and flexural strength at various ages, water absorption and porosity. The two by-products show some similarities and the research aims also at comparing their effectiveness as fillers for cement mortar production, while pointing out any potential

advantages or disadvantages from their use.

2. Materials and methods

2.1 Materials characterization

In this study, Portland cement CEM I 42.5 N, produced according to EN-197-1 was used as reference. The industrial by-products (MSF and CSF) were received in wet condition, both containing about 40% water. Although the two by-products could be used as received and, therefore, avoid the cost and energy for drying, it was decided at first to use them in dry condition for two reasons; Firstly, because transportation of construction materials in wet condition is usually more costly and, secondly, because the granulometry of the dried material can be controlled more easily by crushing and sieving. In order to achieve uniformity for testing, MSF and CSF were air-dried at 40°C until constant mass and then they were crushed and sieved in order to obtain fines of grain size less than 0.075 mm. The particle size distribution of all the materials used was then measured using a Malvern Mastersizer 2000 laser particle size analyzer.

In order to determine the suitability of MSF and CSF for use with cement, a series of characterization tests was carried out. The chemical composition of MSF, CSF and of the reference CEM I 42.5 N cement used were determined using Atomic Absorption Spectroscopy (AAS), as total oxides of metals by digestion using a mixture of concentrated acids, AAnalyst 400, Perkin Elmer. Loss on ignition (L.I. %) was determined at 1000 °C. Additionally, their water-soluble salts were determined using Ionic Chromatography, as anions of salts extracted with distilled water and then filtered, Dionex, while simultaneous Differential Thermal – Thermogravimetric Analysis (DTA-TG), SDT 2960 TA Instruments, was used for the determination of the calcium carbonate (CaCO₃) content of MSF and CSF, under a N₂ atmosphere from 10 °C to 1000 °C. The apparent specific density of the fines used was determined using a Le Chatelier flask according to ASTM C188-14 [31], while their mineralogical composition was determined using a PW 1840 Philips diffractometer (XRD). Since the test materials were tested as cement replacement materials, it was decided to measure their effect on setting time and expansion of ordinary cement according to EN 196-3 [32]. For this reason, seven cement pastes of standard consistence were prepared; one using 100% CEM I 42.5 N; three 10%, 20% and 30% MSF w/w as cement replacement; and three using 10%, 20% and 30% CSF w/w as cement replacement. As the replacement of cement with alternative binders may alter water retentivity and workability, the water required for standard consistence was recorded.

2.2 Design of experimental testing on mortars

Test mortars were prepared in the laboratory, by using 1 part binder; 3 parts sand; and 0.5 parts water. All mortar mixtures were prepared with CEM I 42.5 N cement and natural siliceous river sand conforming to ASTM C33/C33M-13 [33]. MSF and CSF were used as cement replacement at rates of 10%, 20%, and 30%, resulting in seven different mortars, according to Table 1.

Table 1. Constituents of test mortars.

Constituents [g]	R (Reference)	M10	M20	M30	C10	C20	C30
CEM I 42.5	450	405	360	315	405	360	315
MSF	0	45	90	135	0	0	0
CSF	0	0	0	0	45	90	135

Water	225	225	225	225	225	225	225
Sand	1350	1350	1350	1350	1350	1350	1350
Superplasticizer (% of total binder)	0.5	0.5	0.6	0.6	0.8	1.0	1.2

The water to binder ratio was selected to be constant at 0.50, in order to properly assess the effect of cement replacement on strength. Therefore, a polycarboxylate-based superplasticizer was used in varying rates, in order to achieve similar consistence for all fresh mixtures. The required consistence was 150 ± 10 mm diameter at the flow table test, according to EN 1015-3 [34]. The amount of superplasticizer required in order to achieve the required consistence served also as an indication of water retentivity of MSF and CSF, as well as a measure of their effect on workability. An ICAR rheometer was used in order to determine the plastic viscosity of the fresh mortars using the Bingham model [35,36].

Since MSF and CSF have similar or finer particle size distribution than cement, their effect on packing of the wet mortar was investigated. The wet packing method proposed by Wong and Kwan [37] was followed, by measuring the weight of the compacted fresh mortar in a reference mould and then by calculating the solid concentration ϕ of the granular material according to equations (1) and (2).

$$V_c = \frac{M}{\rho_w \cdot u_w + \rho_c \cdot R_c + \rho_{msf} \cdot R_{msf} + \rho_{csf} \cdot R_{csf} + \rho_s \cdot R_s} \quad (1)$$

$$\phi = \frac{V_c}{V} \quad (2)$$

in which V_c is the solid volume of the solid materials used, M and V are the mass and volume, respectively, of the wet mortar in the reference mould $\rho_w, \rho_c, \rho_{msf}, \rho_{csf}, \rho_s$ are the densities of water, cement, MSF, CSF and sand, respectively, u_w is the water ratio, equal to the water to cementitious ratio by volume, and R_c, R_{msf}, R_{csf} and R_s are the volumetric ratios of cement, MSF, CSF and sand, respectively.

After mixing, the 40 x 40 x 160 mm specimens were cast, compacted and cured in an environmental chamber, at $20^\circ\text{C} \pm 2^\circ\text{C}$ and $95\% \pm 5\%$ relative humidity until testing. Compressive strength was measured at 3, 7 and 28 days, while flexural strength, porosity, capillary absorption and apparent specific density were measured at 28 days. The porosity measurement was conducted according to RILEM CPC 11.3 [38], in which the specimens were submerged in the water under vacuum and capillary tests were performed according to EN 1015-18 [39].

3. Results and discussion

3.1 MSF and CSF properties

The particle size distribution of cement, MSF, CSF and natural sand can be seen in Figure 1. The natural sand used showed a gradation of 50 to 4000 μm , while the three binders were considerably finer; MSF shows grain sizes of 0.5 to 80 μm , CSF was sized 0.5 to 400 μm , while cement ranges from 3 to 80 μm . Overall, MSF seems to be finer than cement, while CSF is of similar average fineness, but has a broader range of grain sizes. When MSF and CSF are used for cement substitution in mortars, the difference in particle size distribution is expected to have an effect on the workability and packing ability of the produced mortars.

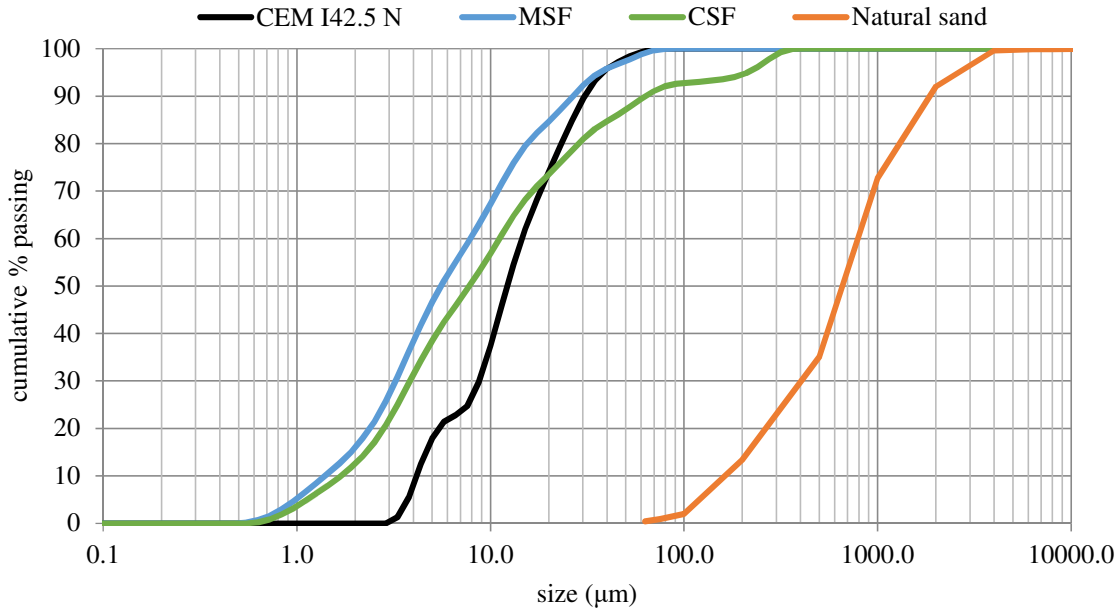


Figure 1. Particle size distribution of the materials used.

The apparent specific density CEM I 42.5 N, MSF and CSF was measured 3140 kg/m³, 2443 kg/m³ and 2556 kg/m³, respectively, according to ASTM C188-14 [31], while the apparent specific density of the natural sand was measured 2636 kg/m³, according to ASTM C128-15 [40]. The chemical analysis of CEM I 42.5 N, MSF and CSF are shown in Table 2. MSF consists mostly of calcium in the form of calcite (Figure 2), which is expected, because it originates from limestone marble sawing. Its chemical composition is similar to that of limestone filler [41] and the high loss of ignition accounts for its carbonate content, which reaches 98.3% wt. (Figure 4).

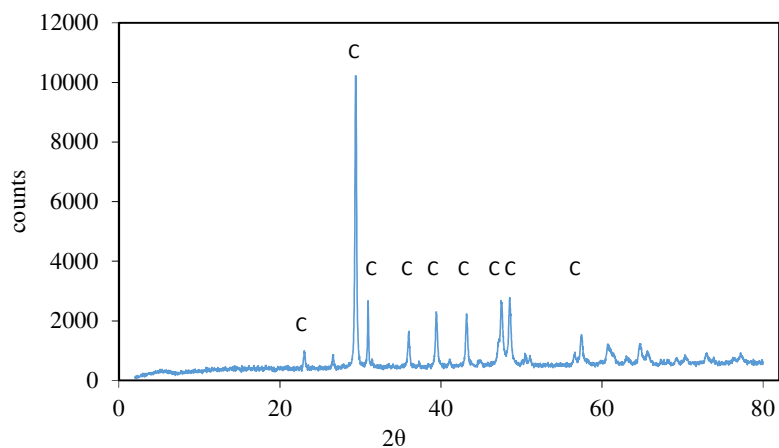
Table 2. Chemical composition of binders (% w.t.)

Constituents	Na ₂ O	K ₂ O	CaO	MgO	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	L.I.%	Cl ⁻	NO ₃ ⁻	SO ₃
CEM I 42.5 N	0.57	1.08	66.80	3.91	2.40	3.74	19.60	1.91	0.03	0.02	1.49
MSF	0.21	0.09	53.02	1.59	0.28	1.14	1.68	41.99	0.03	0.01	0.41
CSF	0.15	0.24	55.07	1.63	1.74	2.80	1.48	36.09	0.03	<0.01	0.34

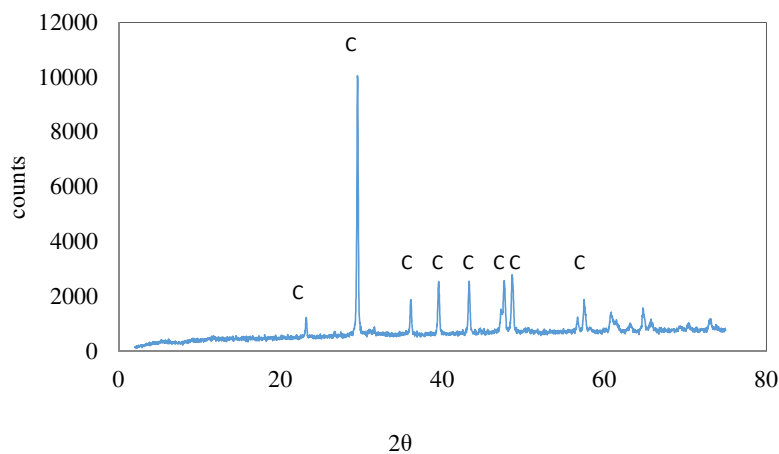
CSF showed a very similar chemical composition to that of MSF and the siliceous content originating from cement hydrates was probably excluded in the sieving process. Crushing and sieving of CSF and MSF was used to release more fine material from brittle agglomerates, rather than reduce the whole fraction below 75 µm, which would be more costly. As a result, the harder compounds in CSF were not crushed and were also probably retained in the 75 µm. Both alternative fine materials had low salts contents and are suitable for cement substitution in this respect. CSF originally consists mostly of hardened cement paste and fine limestone aggregates; therefore, its CaO and SiO₂ contents were not expected to be reactive. The XRD diagram (Figure 3) shows that the mineralogical composition of CSF was mainly calcite. Also, an absence of C₃S and traces of C₂S peaks were observed. The latter may indicate full hydration of the sludge. Also, a strength development test with lime according to ASTM C593-95 [42] showed minimal strength development at 28 days (<0.50 MPa), which implies that there is no significant pozzolanic activity from CSF. The high value of loss on ignition (L.I. %) can be attributed to the bonded water proportion and CO₂ quantity that was emitted from

184 the sample. Indeed, according to the DTA-TG analysis (Figure 5), the loss of volatile compounds (green line of the
 185 diagram) accounted 29.8% for the loss of carbon dioxide, CO_2 , and 0.4741% (or 0.18 w.t.%) for the dihydroxylation
 186 of portlandite, $\text{Ca}(\text{OH})_2$. The presence of a high amount of CaCO_3 (67.77 wt%.) was also verified.

187 The results of the analytical tests imply that both MSF and CSF are fine materials without any hydraulic
 188 or pozzolanic properties. The fact that they can be easily crushed and sieved through the 75 μm implies a
 189 probable use as filler materials. Regarding handling and processing, it would be preferable to use MSF and
 190 CSF in their original wet condition in order to avoid the drying process; however, further research is required
 191 in this direction.



192 Figure 2. XRD diagram of MSF, where C: calcite.
 193



194 Figure 3. XRD diagram of CSF, where C: calcite.
 195
 196
 197
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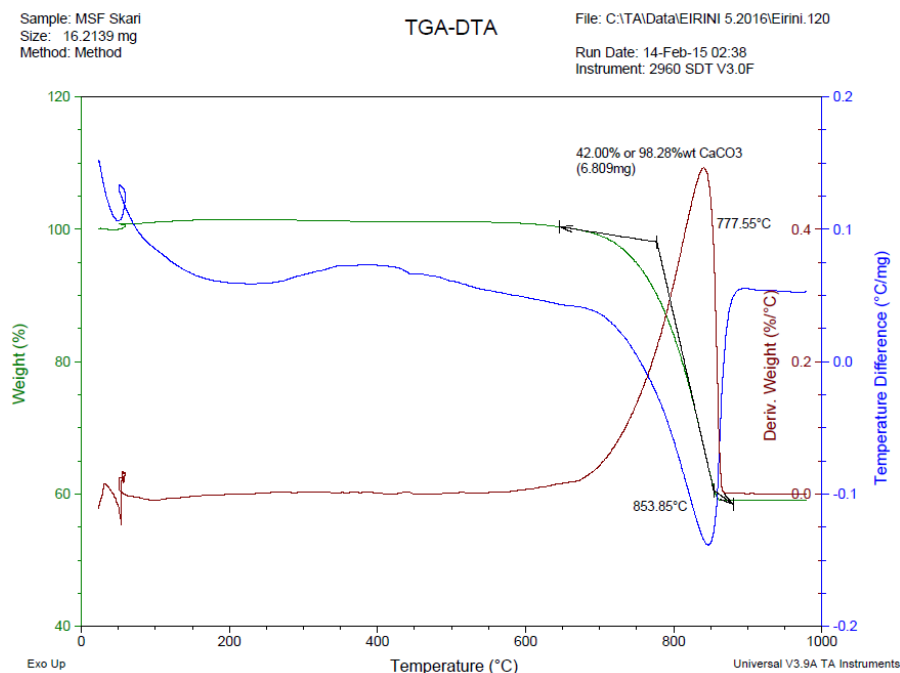


Figure 4. Differential thermal–thermogravimetric analysis (DTA-TGA) analysis of MSF and quantification of calcite content as a percentage by mass of sample.

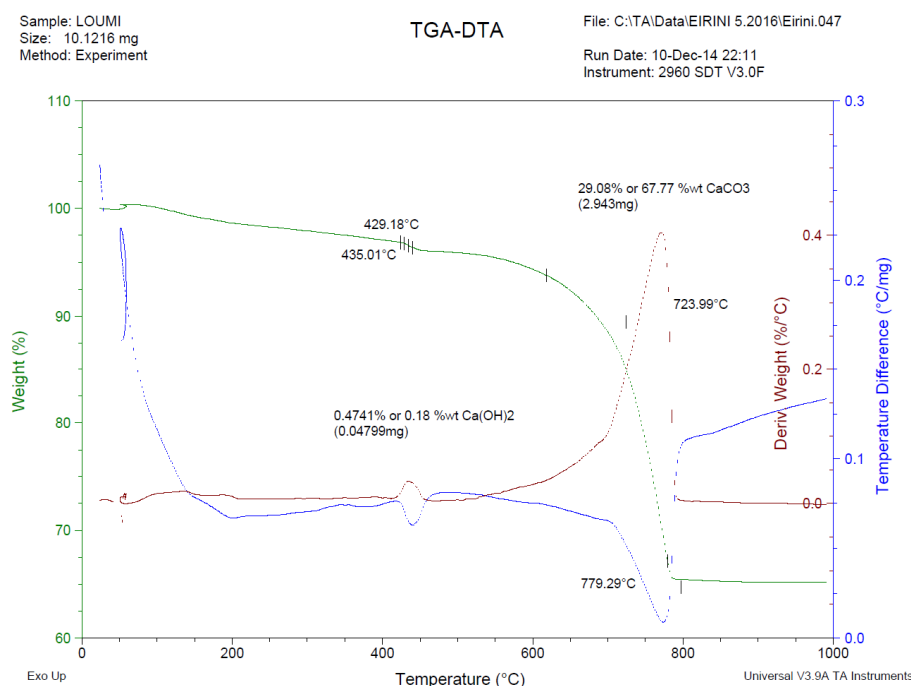


Figure 5. Differential thermal–thermogravimetric analysis (DTA-TGA) analysis of CSF and quantification of calcite content as a percentage by mass of sample.

From the results of Table 3, it seems that cement substitution with MSF does not alter water requirement significantly, while the initial setting time increased slightly at 10% substitution, but the effect diminished at higher replacement rates. Despite an increased value for 20% replacement, which is way below the threshold value of 10 mm according to EN 196-3, MSF use also does not produce considerable expansion in the cement pastes. CSF use on the other hand, seems to increase the water

requirement for standard consistence, which explains the increased superplasticizer used in the mortars with CSF in Table 1, in order to have the same workability of fresh mortars. The initial setting time increased slightly with increased CSF use, but still way below the maximum allowed increase value of 120 min according to EN 206-1 [43], while Le Chatelier soundness remained unchanged with increased CSF use. The variations in initial setting time can be explained by the different coarseness and surface hardness of the grains of MSF and CSF, in respect to the filler effect.

Table 3. Effect of cement replacement with MSF or CSF on setting time and soundness of pastes.

Constituents [% of binder]	Reference	MSF10	MSF20	MSF30	CSF10	CSF20	CSF30
CEM I 42.5	100%	90%	80%	70%	90%	80%	70%
MSF	0	10%	20%	30%	0	0	0
CSF	0	0	0	0	10%	20%	30%
Water/binder for standard consistence	0.296	0.298	0.300	0.296	0.314	0.360	0.376
Initial setting time [min]	187	207	191	182	199	203	204
Le Chatelier soundness [mm]	1.2	0.9	2.7	0.4	1.0	0.5	1.1

3.2 Results from testing mortars

3.2.1 Fresh mortar properties

As it can be seen from Table 1 and already commented in the previous paragraph, CSF use required an increased amount of superplasticizer in order for the mortar to reach the required workability. Regarding plastic viscosity resulting from the Bingham model and packing ability as expressed by solid concentration ϕ , of the fresh mortars, the results are presented in Table 4.

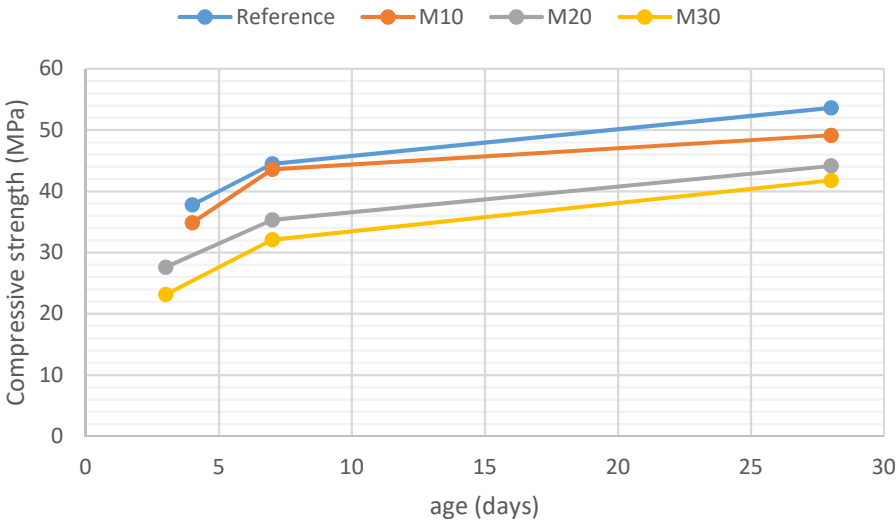
Table 4. Viscosity and packing ability measurements in fresh test mortars.

Test mortars	reference	M10	M20	M30	C10	C20	C30
Plastic viscosity (Pa.s)	7.9	-	6.7	14.2	3.9	15.7	27.7
Solid concentration ϕ	0.694	0.718	0.726	0.732	0.694	0.703	0.700

The results show that a high replacement rate of cement would render the fresh mortar more viscous, compared to the reference. Although M10 did not yield reliable result, at lower replacement rates (up to 20% for MSF and 10% for CSF) viscosity seems to decrease compared to the reference. A certain level of viscosity is required for the mortars to be easily compacted, but in other cases, a higher viscosity serves as resistance to segregation in highly flowable mortars. Although both test materials alter viscosity, MSF seems to produce mortars of lower viscosity compared to CSF. This can be attributed to the increased fineness of MSF compared to CSF. Indeed, the fineness of MSF contributes also to a higher packing, as expressed by the increase in solid concentration compared to the reference mortar, which increases proportionally to the rate of MSF use. CSF on the other hand, does not alter solid concentration significantly, owing to the similar particle size distribution to cement, as shown already in Figure 1.

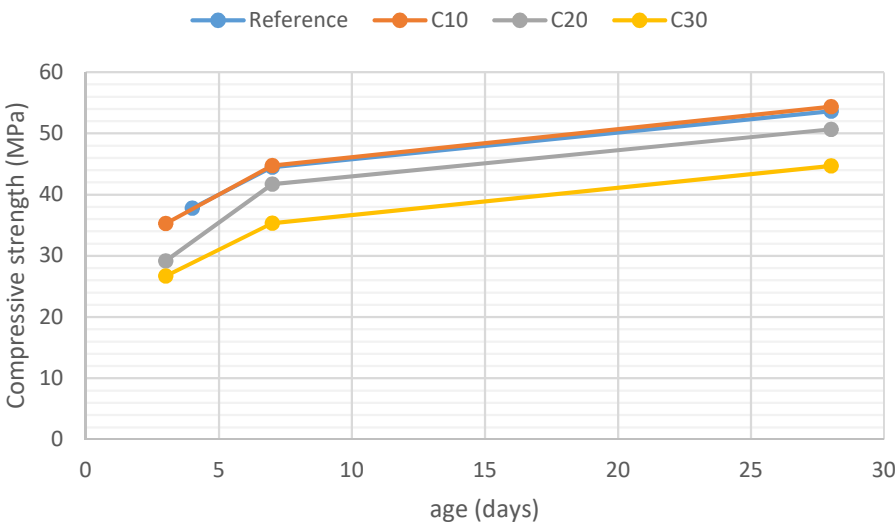
3.2.2 Hardened mortar properties

245 Regarding mortar strength development, increasing MSF use results in reducing compressive strength
 246 (Figure 6), however at 10% cement replacement rate, mortars with MSF exhibit 98.0% and 91.7% of the
 247 reference compressive strength at 7 and 28 days, respectively. The early age strength development of mortars
 248 with MSF may be attributed to the filler effect.
 249



250
 251 Figure 6. Compressive strength development of cement mortars with MSF.
 252

253 When CSF was used as 10% cement replacement, no reduction in strength development was observed
 254 (Figure 7). By increasing the rate of cement replacement, compressive strength decreased, but at 20% CSF use
 255 the mortars reached 93.8% and 94.5% of the reference compressive strength at 7 and 28 days, respectively.
 256 Although the filler effect also explains early strength development, CSF seems to perform better compared to
 257 MSF regarding cement hydration. This occurrence could be attributed to the considerably higher alkalinity of
 258 CSF ($\text{pH} = 12.1$), compared to MSF ($\text{pH} = 9.7$), which facilitates cement hydration. When a filler is added in
 259 a certain percentage, it influences the pH value of the binders in the pore solution. If the pH is reduced, this
 260 affects the hydration and as a result the compressive strength [44].
 261



262

Figure 7. Compressive strength development of cement mortars with CSF.

Flexural strength on the other hand, reduced in all cases, and the reduction was proportional to cement replacement (Figure 8). The flexural strength ranged from 88.3% of the reference for 10% MSF use to 67.6% of the reference for 30% CSF use. This indicates that the interfacial transition zone between the cement matrix and the aggregate, deteriorated when the industrial by-products were used and the deterioration can be attributed to the surface characteristics and water absorption of the MSF and CSF particles. Indeed, CSF, which showed increased water retentivity at the mortar production stage, showed higher decrease in flexural strength development.

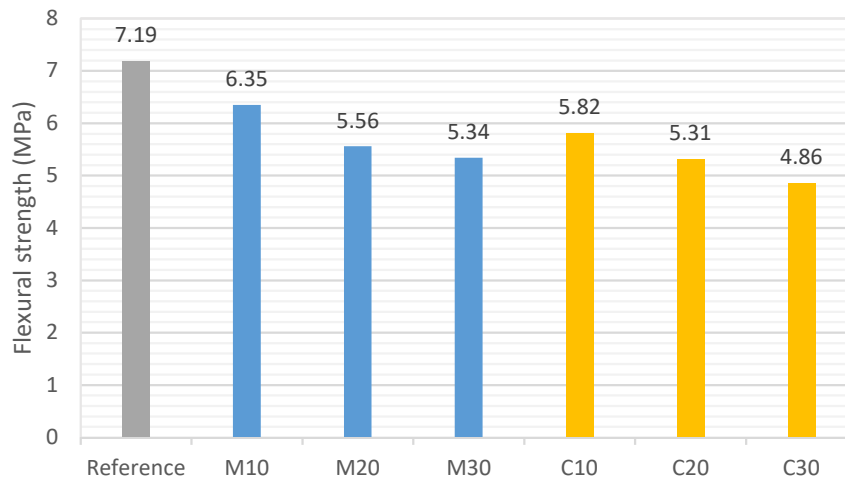


Figure 8. 28-day flexural strength of cement mortars with MSF or CSF.

The capillary absorption and porosity results shown in Table 5 validate the above statement, since mortars with CSF show increased absorption, porosity and capillary coefficient, compared to mortars with MSF. The reference mortar shows the lowest values in these three tests, while increasing cement replacement increases absorption and porosity values proportionally. In addition, the apparent specific density of the mortars reduced with increased cement replacement, as expected, but CSF use reduced density more than MSF, despite having itself higher density compared to MSF. This also implies that CSF use resulted in more voids in the cement matrix and can be attributed to the coarser granulometry and higher water retentivity of CSF compared to MSF.

Table 5. Physical properties of mortars with MSF and CSF.

Constituents	Reference	M10	M20	M30	C10	C20	C30
Apparent specific density [kg/m ³]	2152	2068	2074	2065	2056	2062	2030
Absorption [%]	1.6	2.8	2.7	3.8	3.2	5.0	4.4
Porosity [%]	3.5	5.9	5.5	7.8	6.6	10.3	9.0
Capillary coefficient c [g/cm ² *min ^{1/2}]	0.043	0.057	0.066	0.103	0.088	0.112	0.122

4. Conclusions

The results show that the two by-products (MSF and CSF) share considerable similarities; they have

287 similar chemical composition, consisting mostly of calcium carbonate; they are both largely inert; and their
288 fineness renders their most probable use as fillers. In addition, they are both produced in wet condition and
289 require similar processing and handling prior to use (drying, crushing, sieving). MSF and CSF, when
290 investigated as supplementary cementitious materials by replacing cement in mortars, also showed some
291 differences that must be taken into account. MSF was finer, had lower water retention than CSF, and therefore
292 provided better packing ability, resulting in lower absorption and porosity of the hardened mortar; CSF on the
293 other hand showed higher compressive strength values compared to MSF, when used as 10%, 20% or 30% wt.
294 cement replacement, probably owing to its higher alkalinity.

295 Based on the results, it seems that both MSF and CSF can be used as filler for cement replacement in
296 mortars, since their intrinsic properties, including fineness, chemical composition and reactivity seem adequate
297 for such a use, while the strength reduction obtained is reasonable. Further research is required, of course,
298 regarding utilization in wet condition and quality control of the materials.

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