



Influence of the mixing procedure on the fresh state behaviour of recycled mortars

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ABSTRACT

The effect of two different mixing protocols on the fresh-state behaviour of self-compacting mortars with and without recycled sand was compared. For this purpose, the mortar mixes were designed considering three solid volume fractions, while maintaining the water to cement ratio and superplasticiser dosage constant. The results conclude that the inclusion of recycled sand harmfully affects the rheological behaviour of the mortars and this effect can be mitigated using mixing protocols with longer mixing times and delayed admixture addition times. Finally, a rheograph was developed to explain the expectable changes in a conventional mix when recycled sand is incorporated, the solid volume fraction is varied and changes are applied to the mixing procedure.

1. Introduction

One solution for achieving durable concrete structures, independently of construction work quality is the employment of self-compacting concrete (SCC) [1]. This innovative material can flow through and fill in the gaps in reinforcements, corners of moulds and voids in rock blocks without the need for vibration and compacting during the placing process, thereby improving the overall efficiency of concrete construction projects. The key requirement for SCC is high flowability without the segregation of aggregate during placement [2]. Based on the rule that a concrete is a suspension made out of a liquid phase (paste) and a solid phase (aggregates with fixed gravel/sand ratio), the self-compacting behaviour of concrete greatly depends on the characteristics of the paste [3].

Concrete is the most used material in the construction of buildings and infrastructure and its main raw materials, the aggregates, are found at a proportion of 60–80% in volume [4]. This represents a huge demand for materials that is satisfied mostly by natural sources. At the same time, demolition and repair works produce large amounts of construction and demolition waste. In this context, the use of construction and demolition waste debris as aggregates in concrete production is a logical step forward including both financial and environmental advantages that has been growing in popularity over the last two decades [5].

Although there are many works that deal with the incorporation of

recycled coarse aggregate in self compacting concretes, the use of the recycled fine fractions, and their influence on the rheological properties of this kind of concrete is not as commonly studied. Some authors have studied the fresh properties of self-compacting concrete containing fine and coarse recycled aggregates [6–9]. Their results have shown that it is feasible to produce SCC with both fine and coarse recycled aggregates up to a 50% replacement level. It was shown that the slump flow and blocking ratio increased as the recycled fine aggregate content increased. Carro-López et al. [10] studied the effect of incorporating fine recycled aggregates on the rheology of self-compacting concrete over time (at 15, 45 and 90 min). The fine fraction of the natural aggregates was replaced with recycled sand at 0%, 20%, 50% and 100%. They observed a significant increase in yield stress and plastic viscosity over time for all replacement ratios.

Güneyisi et al. [11] used coarse and fine recycled aggregates. Natural coarse aggregate was replaced with recycled coarse aggregate at 0%, 50% and 100% and natural fine aggregate was replaced with recycled fine aggregate at 0%, 25%, 50%, 75% and 100%. These authors concluded that the modified Bingham and Herschel-Bulkley models provide well defined rheological representations.

Mixing is one of the main important processes in concrete production. Usually, concrete is developed in a central-mix concrete batch plant using different mixers and varying times. After mixing, the concrete is poured into a truck and additional mixing of considerably lower energy

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but longer duration (transportation time) is then carried out. The main objective of mixing is to homogenise the mix to disperse the solids phase into the liquid phase. Mixing energy and mixing time are variables that highly influence concrete rheology and even concrete microstructure [12]. The development of new concretes, especially self-compacting concrete [13], with a high content of fines and admixtures has made the evaluation of optimum mixing procedures even more important.

There are many literature works [14–16] that study the effect of mixing time or mixing energy on the fresh state behaviour of conventional concretes. Mixing leads agglomerates of cement particles to break and better disperse. The mixing energy or extended mixing produces forces that promote the dispersion of the particles [17]. Different authors [18,19] conclude that workability improves with the increase of intensity or time, at least up to one optimum value, after which time the slump flow may decrease due to over mixing. They also conclude that not only fresh state, but also the hardened properties are affected by the mixing procedure due to changes in the hydration products, as the interactions among cement particles are also affected [20]. Extended mixing time is also desirable to better disperse the admixture. However, the optimum mixing time for cement paste can differ from that for mortar or concrete due to the ball-milling action of the aggregates [21].

On the other hand, when admixtures are used, the mixing procedure highly influences fresh concrete behaviour. The fluidity of cement pastes is affected by the addition time for organic admixtures. The results show that a delay in the addition time of the admixture reduces the values of the rheological parameters of the cement pastes [22,23]. When the quantity of admixture adsorbed by cement is low, the paste flow is high. A later addition time for the admixture reduces the amount adsorbed, as it is found in the literature, that it is smaller in the clinker compounds exposed after the dissolution of calcium ion than in the unhydrated compounds [24,25].

2. Research significance and objectives

Although extended mixing times and later admixture addition times improve workability in conventional concrete, the effect of these parameters in recycled concrete has not been studied.

Recycled concrete presents lower workability than conventional concrete and its workability loss is faster. This is due to the difference in the effective water to cement ratio, as a result of the non-compensated water absorption, and the amount of fines from the old mortar adhered to the recycled aggregate [26]. Fine particles (also named microfines) have a considerable effect on concrete rheology as they tend to agglomerate when water is introduced in the mix due to different small forces (such as capillary, electrostatic charges or van der Waals forces). In the specific case of recycled fines, they present unique characteristics that highly damage concrete rheology. These particles are rough in texture, show a high water absorption capacity and can even present hydraulic activity.

The fines particles of recycled aggregate can increase due to breakage during mixing [27,28], which means that, in these cases, increasing mixing time may not be a good option for improving recycled concrete workability. Moreno Juez et al. [29], using different mixers and mixing speeds, conclude that the degradation of the coarse aggregate during mixing is higher in recycled aggregate than in natural aggregate, and mixing parameters and initial properties of the recycled aggregates influence the breakage rate. The tests were performed considering only one concrete mix, which makes it impossible to analyse the effect of the solid volume ratio on the results. This ratio is highly important as it controls the interaction between aggregate particles. Moreover, they do not measure how this degradation changes the fresh state behaviour of the concretes.

Thereby, the main goal of this work is to observe the influence of the mixing procedure on the fresh behaviour of self-compacting mortar made with conventional and recycled sand. The objective is to clarify whether the rheology of recycled mortars, when the mixing time is

extended and admixture addition time delayed, is more influenced by the negative effects of the generation of fines due to friction or the beneficial effects of better dispersion of the fine particles and the reduction in the quantity of admixture adsorbed by the cement. To do so, we will study the rheological behaviour of mortars (conventional and recycled) using two different mixing times together with two different delayed admixture addition times.

3. Experimental programme

3.1. Materials

The following materials were used in the experimental procedure: cement, superplasticiser, water, conventional sand and recycled sand.

3.1.1. Cement and superplasticiser

CEM I 52.5 N - SR 5 Portland cement was used as binder to prepare the studied mortars. The chemical composition of this cement is given in Table 1. It showed a particle density of 3.05 t/m^3 .

A polyaryl-ether (PAE) based additive was used as superplasticiser. It offers chemical backbone units and a high density of negative charges, which enhances its affinity with the cement surface.

3.1.2. Granular skeleton: Conventional and recycled sand

Regarding the granular skeleton, two types of fine aggregates were used: conventional sand (CS) and recycled sand (RS). Regarding the nature of the conventional sand, it was obtained by mixing a limestone sand, at 70%, and a granitic sand, at 30%. The recycled sand came from the recycling of materials used to build reinforced concrete structures, which were then demolished. The debris was collected and crushed into particles with a particle size distribution between 0 and 4 mm. This recycled sand mostly contains concrete and stone (i.e. it is a recycled concrete fine aggregate).

The conventional and recycled sand need to be well graded and present a size distribution that is as similar as possible (Fig. 1). For this reason it was decided to adjust the grading curve of the conventional sand by mixing the two conventional sands at percentages of 30% and 70%. The obtained conventional sand (after mixing both) presented a size distribution similar to that of the recycled sand, with a particle size between 0 and 4 mm. The fineness modulus of the recycled sand experienced small variations for the same maximum aggregate size, at 3.37 in comparison to the value of 3.11 for the conventional sand.

Using an optical microscope, some images of the conventional and recycled sand were taken (Fig. 2). They reveal that both sands are irregular in shape (as both came from a crushing process because the conventional sand used in this work is a mix of two quarry sands). Regarding texture, as was seen in other works [2,30,31] the recycled aggregate presents a rougher texture than the conventional one.

As fine particles produce important changes in the fresh state behaviour of cement mortars, the fine fraction (particles under 0.125 mm) of both conventional and recycled sand was also studied. The particle size distribution of the fine fraction was measured using a laser diffractometer. The amount of conventional sand fines (CS-F0) was 15.39% and recycled sand fines (RS-F0) was 16.26%. The grading curves of these fines (Fig. 3) shows that both conventional and recycled sand present a similar particle size distribution. Applying the nitrogen gas adsorption technique, the BET specific surface area was calculated from the adsorption branch of the isotherms in the range of 0.05 to 0.3 relative pressure. The results show a value of $1.36 \text{ m}^2/\text{g}$ in the case of CS-F0 and $8.81 \text{ m}^2/\text{g}$ for RS-F0. This high difference indicates that the recycled fines present a high open porosity which promotes the N₂ adsorption.

Both sands (CS and RS) and their fines (CS-F0, RS-F0) were also characterised using different techniques: chemical composition (XRF analysis), mineralogical composition (XRD analysis) and thermal gravimetric analysis (TGA) up to $900 \text{ }^\circ\text{C}$ at a constant rate of $5 \text{ }^\circ\text{C}/\text{min}$.

Table 1
Chemical composition of cement (XRF analysis).

Component (oxide; wt%)										
SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	SO ₃	K ₂ O	TiO ₂	P ₂ O ₅	*LOI
18.9	6.3	2.7	0.06	1.6	59.9	3.5	1.9	0.28	0.13	4.3

* LOI = Loss on Ignition at 1000 °C

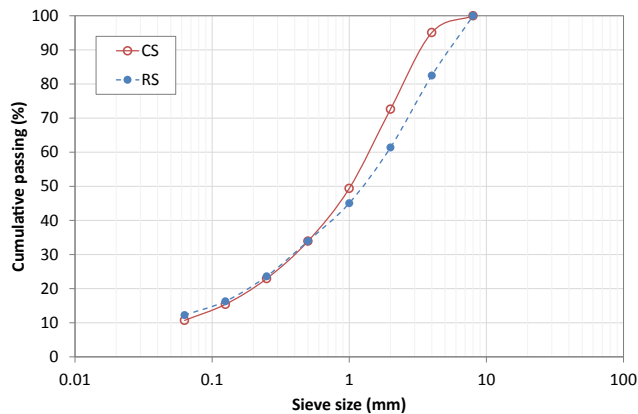


Fig. 1. Grading curves of aggregates.

Chemical composition by X-ray fluorescence (XRF) of conventional and recycled sand and their fines is shown in Table 2.

Phase identification was carried out by X-ray Diffraction (XRD) identifying the presence of different amounts of crystalline phases that correspond, in the case of the recycled sand, to the original aggregates and the adhered cement paste.

The recycled sand presented significant peaks of quartz, calcium minerals and feldspars (albite, anorthite, etc.) (Fig. 4). Anhydrous phases and calcium hydroxide were not detected as they were most likely carbonated by air and only a small peak of crystalline hydrated calcium silicate was detected. Moreover, it is difficult to distinguish the

crystalline hydrated calcium silicate phases as their peaks are usually overlapped by the peaks of calcite and feldspars.

There is also a significant peak indicating the presence of calcite, probably due to the carbonation of the aforementioned hydrated species in the adhered cement paste. The peak of the quartz and feldspars are attributed to the sand and aggregates of the original concrete.

Minor peaks of muscovite/illite/kaolinite are also detected in the RS, which can be due to the presence of bricks/ceramics, impurities that can be present after the recycling process of construction and demolition waste.

The XRF results of recycled sand confirm the diffractograms, showing that the predominant oxides are calcium oxide (CaO), silicon dioxide (SiO₂), and aluminium oxide (Al₂O₃). The first indicates the presence of adhered mortar and the last two the presence of quartz, feldspar (albite), muscovite, etc.

The peaks of quartz (SiO₂) and calcite in the diffractogram are different in the recycled fines to those in the recycled sand (Fig. 4). In the fines, the calcite peak is higher, and more calcite peaks were detected in the XRD pattern. These results indicate that the recycled fines are composed of highly carbonated adhered paste. Quartz peaks are lower in the fines than in the sand diffractogram, although their presence is significant. This indicates that a significant amount of the original aggregates is also incorporated in the recycled fines. As for the recycled sand, there is an absence of a detectable amount of anhydrous cement compounds.

The XRF results also show that recycled fines present a higher calcium oxide content and lower silicon dioxide content than recycled sand. Accordingly, LOI in recycled fines is higher than in recycled sand.

In the case of conventional sand, the crystalline phases detected in

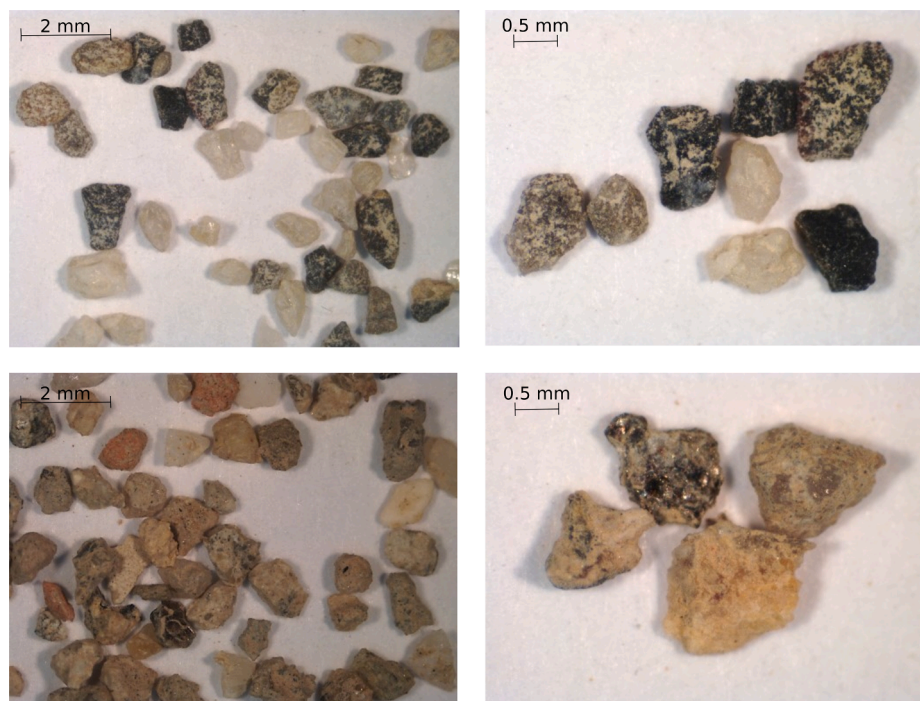


Fig. 2. Shape and texture of fine aggregate (top: conventional, bottom: recycled).

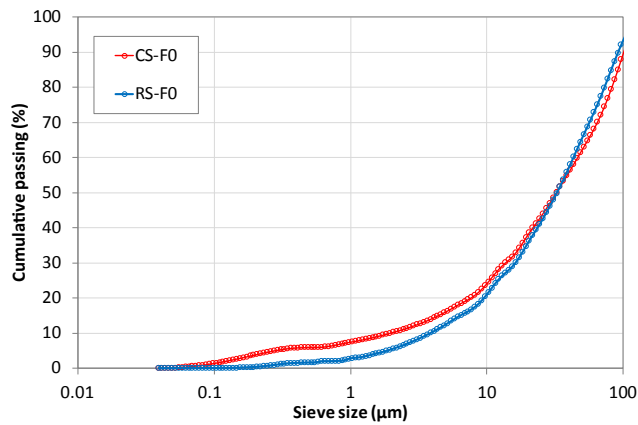


Fig. 3. Grading curves of fine particles.

Table 2
Chemical composition of conventional and recycled sand and fine particles (XRF analysis).

	CS	RS	CS-F0	RS-F0
CaO	28.9	12.2	40.0	16.9
SiO ₂	23.6	56.1	12.1	44.0
Al ₂ O ₃	4.2	11.0	3.0	13.4
Fe ₂ O ₃	1.0	3.8	1.5	5.9
K ₂ O	1.4	2.7	0.7	2.5
Na ₂ O	1.0	1.4	0.26	0.7
SO ₃	0.09	1.0	0.10	1.5
MgO	9.4	1.5	9.3	1.4
LOI	30.1	9.0	32.6	12.0

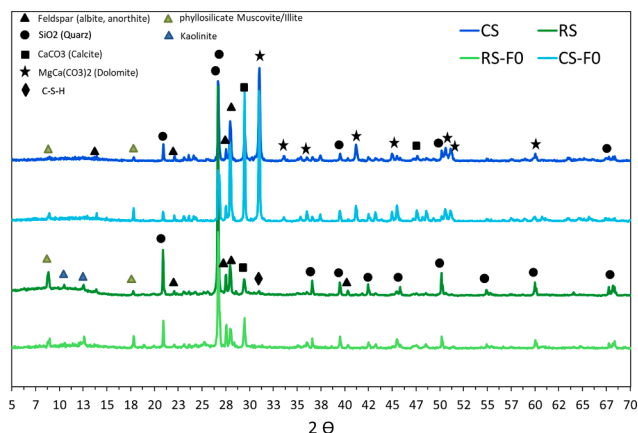


Fig. 4. Mineralogical composition of conventional and recycled sand and fine particles (XRD analysis).

the diffractograms correspond to a mix of 70% limestone sand and 30% granitic sand. The former mainly comprises calcite and dolomite and the latter mainly quartz.

As can be observed in Table 2, the XRF results show that fines of conventional sand present a higher calcium oxide and lower silicon dioxide content than conventional sand because the limestone fraction of particles passing a 125 µm sieve accounted for 80%, while the granitic fraction only 20%. It should be noted that the limestone fraction was used at 70% and the granitic at 30%. So, it is clear that a high quantity of fines in the mixed conventional sand come from the limestone fraction. The XRD diffractogram (Fig. 4) corroborates this issue, as the calcite peak is higher in the conventional fines than in the conventional sand. Accordingly, the peak of quartz is lower in the fines diffractogram than in the sand pattern. Finally, LOI in conventional fines is higher than in

conventional sand (Table 2).

The thermal gravimetric analysis (TGA) of both recycled and conventional sand is shown in Fig. 5. This is a technique where the mass of a substance is monitored as a function of temperature or time, as the sample specimen is subjected to a controlled temperature programme in a controlled atmosphere. This analysis shows weight loss as a function of temperature. This loss is typically caused by chemical reactions (decomposition and loss of water of crystallization, combustion, reduction of metal oxides) and physical transitions (vaporization, evaporation, sublimation, desorption, drying). Fig. 5 shows the weight loss of both conventional and recycled sand in percentage, from temperature 0 to 900 °C. This figure shows mass losses due to dehydration and decarbonation of different products.

According to different authors [32,33] the water that comes from hydrated compounds (mainly C-S-H) is seen up to 300 °C. Other small steps happen in this range due to the interlayer water, the adsorbed water of the gel and the water in the capillary pores. Dihydroxylation of these compounds takes place between 400 °C and 500 °C, and decarbonation occurs above 600 °C.

In this sense, as can be seen in Fig. 5, recycled sand has mass losses between 100 and 650 °C due to the dehydration of the hydration products of the adhered cement paste (tobermorite gel and hydrate of alumina and ferrite) and then a high peak around 700 °C due to the decarbonation of calcite. These results confirm the values obtained with the XRD and XRF tests.

The TGA analysis of recycled fine particles is also presented in Fig. 5. It clearly shows that the recycled fines have mass losses due to the dehydration of hydration products between 100 and 650 °C and higher than the losses experienced by the recycled sand. The decarbonation peak around 700 °C when heated up is slightly higher in the recycled fine particles than in the recycled sand.

In the case of conventional sand, it presents a one-time peak around 700 °C due to the decarbonation of the limestone fraction (calcite and dolomite). The peak of quartz was seen to be lower in the conventional fines diffractogram than in the conventional sand pattern (Fig. 4). The TGA analysis confirms these results.

All these results indicate that the adhered cement paste is ripped off during handling. Therefore, the presence of this material in the recycled fine fraction is more relevant than in the coarse fraction.

Other physical - mechanical properties of the aggregates used are listed in Table 3. It should be noted that the water absorption of recycled sand is much higher than that of conventional sand, and consequently the density is much lower. It is well known that this is due to the presence of cement mortar that remains attached to the recycled aggregate particles [34]. So, it is verified that the higher the content of attached mortar and impurities, the higher the recycled sand absorption and the lower the recycled sand density.

The maximum packing fraction (ϕ_{max}) of sand aggregates was also

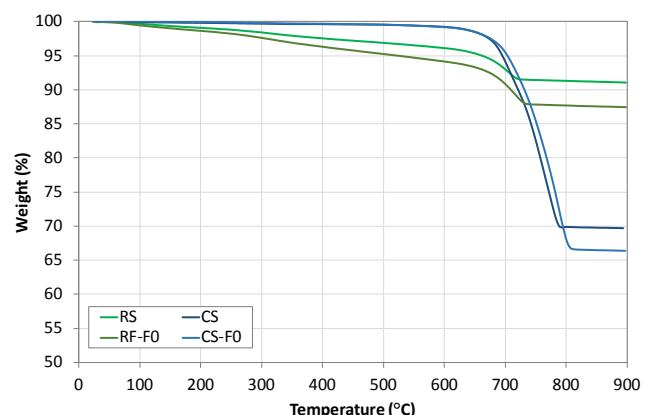


Fig. 5. The TGA of conventional and recycled sand and fine particles.

Table 3
Properties of aggregates.

Sample	Saturated-surface-dry density (t/m^3)	Water absorption (%)	Maximum packing fraction (ϕ_{max})	Sand equivalent
CS	2.71	0.72	0.81	82
RS	2.43	6.77	0.75	75

measured. In general terms the results indicate similar packing properties because shape and therefore packing are mostly imposed by processing and both sands were crushed. However, as expected, the recycled sand shows a slightly lower ϕ_{max} because it has a rougher texture than the conventional sand leading to a worse packing density.

3.2. Mixes and mixing protocols

The design of the mortar phase consisted of six self-compacting mortar mixes, three mixes with conventional sand and another three with recycled sand. In each series, three solid volume fractions (particles > 0.125 mm) were considered (ϕ): 0.20, 0.30 and 0.45. It should be noted that the target value of 0.45 had to be lowered to 0.35 in the case of the mortar mixes with recycled sand because it was not possible to carry out the tests due to the loss of flowability.

All mortar mixes were designed with a water to cement ratio of 0.4 (by weight) and superplasticiser content of 0.6% (i.e. relationship between liquid mass and cement mass). The recycled mortars were obtained by replacing the conventional sand with recycled sand (by volume) at a replacement rate of 100%.

It should be noted that all aggregates (conventional and recycled) were used in dry-state conditions and an extra quantity of water was added during mixing. This was calculated to compensate the aggregates' absorption at 24 h.

Table 4 shows the mix proportions of all mortar mixes studied.

Two mixing protocols were used in order to take into account two different mixing times (i.e. the time that materials are in the mixer from the moment of water-cement contact) and two different admixture addition delay times. Both of them were designed to provide, at the beginning, a 10 min period where the aggregates are in contact with their saturation water. The first one was thought to be as short as possible, guaranteeing periods of at least 1 min mixing of each material and a final period of 2 min to achieve a good dispersion of the superplasticizer. The second one was thought to be a long mixing procedure where a final period of 11 min is designed to improve the dispersion of the raw materials.

The first protocol, Protocol 1 (P1), establishes 4 min mixing time and 1 min admixture addition delay time, Fig. 6. In this protocol the aggregates were blended for 1 min in the mixer with the extra water to compensate for water absorption. The aggregates were then allowed to stand for 9 min to absorb this water. Then the cement was added and mixed with aggregates for 1 min. Then, the mixing water was added and after one minute of mixing, the superplasticiser was added. All materials were mixed together for 1 min. After that, the mortar mix was allowed to stand for 1 min and again mixed for one additional minute before starting the tests.

The second protocol, Protocol 2 (P2) establishes 15 min mixing time and 4 min admixture addition delay time (see Fig. 7). The same stages as

Table 4
Mix proportions of mortar mixes ($1 m^3$).

Material	CM (Conventional mortar) (kg/m^3)			RM (Recycled mortar) (kg/m^3)		
	$\phi = 0.2$	$\phi = 0.3$	$\phi = 0.45$	$\phi = 0.2$	$\phi = 0.3$	$\phi = 0.35$
Cement	1047.80	885.63	642.37	1044.43	880.58	798.65
Water	419.12	354.25	256.95	417.77	352.23	319.46
Sand (>0.125 mm)	538.00	807.00	1210.50	456.00	684.00	798.00
Sand Fines (<0.125 mm)	97.86	146.79	220.18	88.54	132.81	154.95
Superplasticiser	1.26	1.06	0.77	1.25	1.06	0.96

in P1 were followed in this P2 although the times varied. The superplasticiser was added after 4 min of mixing. The sand, cement, water and superplasticiser were then mixed for 5 min rather than 1 min. The mortar was left to rest for 2 min and finally mixed again for an additional 4 min. Then it was left to rest until its testing age.

3.3. Testing methods

In this work, mortars were firstly tested in fresh-state with the mini-slump flow test (also named the mini-cone test) and the mini-funnel test (Fig. 8). The former consists of a mould in the form of a frustum of a cone, 60 mm in height with a diameter of 70 mm at the top and 100 mm at the base. After filling the cone, it is lifted and the mortar spreads over a metal plate. Then, the average diameter of the spread is measured. The mini-funnel consists of a V-shaped recipient with an opening of 30×30 mm at the bottom. It is filled, the bottom gate is opened and the time required for the mortar to flow through the tapered outlet is determined. Secondly, mortar mixes were tested with the Viskomat XL rheometer (Fig. 8). It is a wide gap rheometer for mortar and fresh concrete up to 8 mm grain size.

The rheology was studied throughout a flow curve test. The parameters measured with this test were the yield stress (or dynamic yield stress) and the plastic viscosity. In the test, the stresses were measured in 7 steps at different speeds between 0 and 70 rpm (Fig. 9).

In both protocols, mortar tests were carried out at 21 min after the water to cement contact (32 min from the beginning). In the case of Protocol 1, before starting the tests, at the 26th minute the mortar was mixed with a spoon and let rest for 5 min. This was carried out in order to be able to compare it with the case of 15 min mixing time, which finished at the 26th minute (Fig. 10). The mortar was mixed again with a spoon and then the flow curve test was started. All measurements were taken from the self-compacting mortars with conventional and recycled sand.

4. Results

In the following sections the empirical and rheological results of both types of mortar, conventional-CM and recycled-RM are analysed. For both of them, the results are considered taking into account the two different mixing protocols, Protocol 1 (P1) and Protocol 2 (P2).

4.1. Empirical results

The mini-slump and mini-funnel values for the different mortar mixes are shown in Figs. 11 and 12. In general terms, as the solid volume fraction increases, the mini-slump diameter decreases and the mini-funnel time increases.

Mortars with conventional sand had a larger slump value and shorter

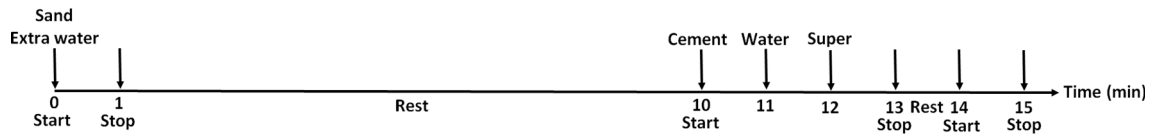


Fig. 6. Mortar mixing protocol 1 (P1). 4 min mixing time and 1 min admixture addition delay time.

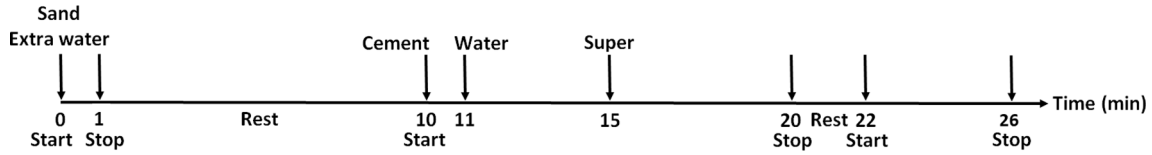


Fig. 7. Mortar mixing protocol 2 (P2). 15 min mixing time and 4 min admixture addition delay time.

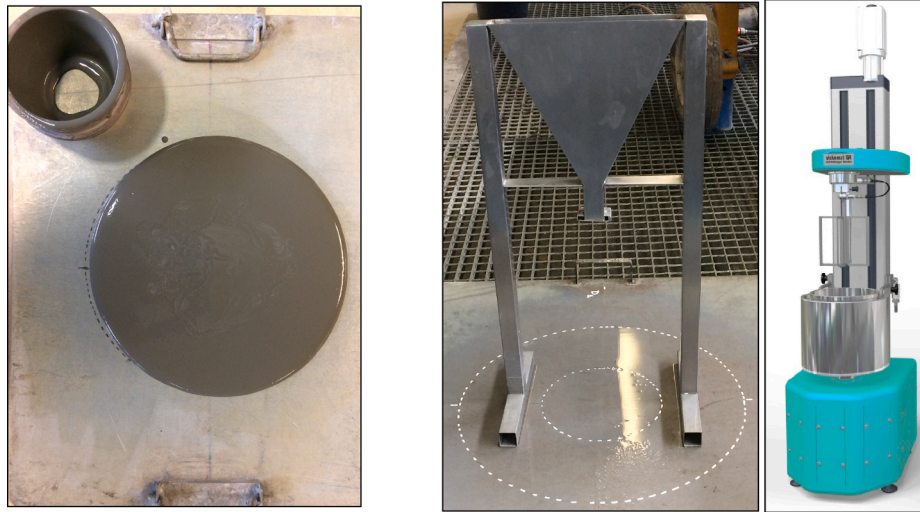


Fig. 8. Mini-slump (left). Mini-funnel (middle). Viskomat XL rheometers (right).

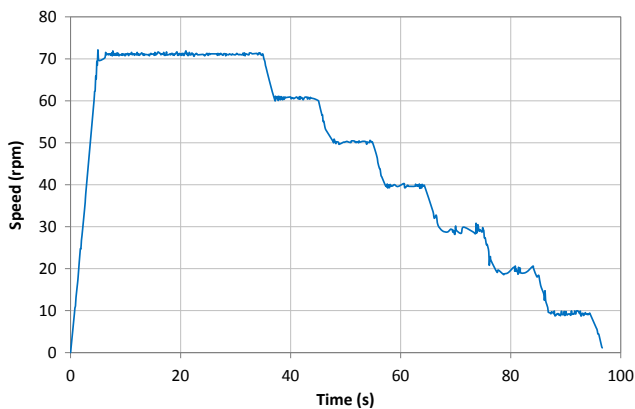


Fig. 9. Mortar rheology. Flow curve test.

funnel time than mortars with recycled sand for 0.20 and 0.30 solid volume fraction. The differences are greater as ϕ increases.

The mixing protocol was also observed to affect mini-slump and mini-funnel values. For protocol 2 (longer mixing time and longer admixture addition delay time) the flowability tended to increase in all mortars with the increase in solid volume fraction, but up to a maximum limit (0.45). In the recycled mortars, the decline was so steep that it could not be measured for 0.45 solid volume fraction. In this case, the limit was for 0.35, as aforementioned.

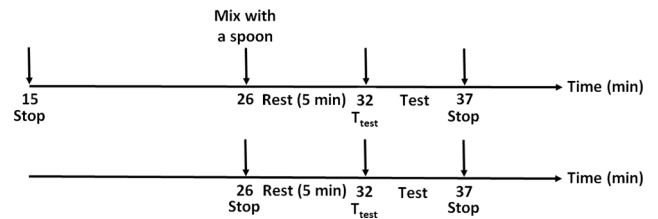


Fig. 10. Mortar testing times: Protocol 1 (up) and Protocol 2 (down).

4.2. Rheological results

A flow curve was adjusted with the results obtained in the flow curve tests, drawing the points of each step of the descending branch. The rheological model was then adjusted with a line, showing a good agreement with the experimental results which means that mixes show Bingham behaviour. In Fig. 13 and Fig. 14 an example of the adjustment can be seen.

In the flow curve test, we measured the yield stress (dynamic) and the plastic viscosity. Both were measured after the structural breakdown of the mortar mix, thereby avoiding the effects of thixotropy.

In Fig. 15 and Fig.16 the yield stress and the plastic viscosity are plotted, respectively. It can be seen that both the yield stress and plastic viscosity are higher in recycled mortars than in conventional ones.

In particular, when the solid volume fraction is low (paste is dominating the rheological behaviour) the differences between recycled and

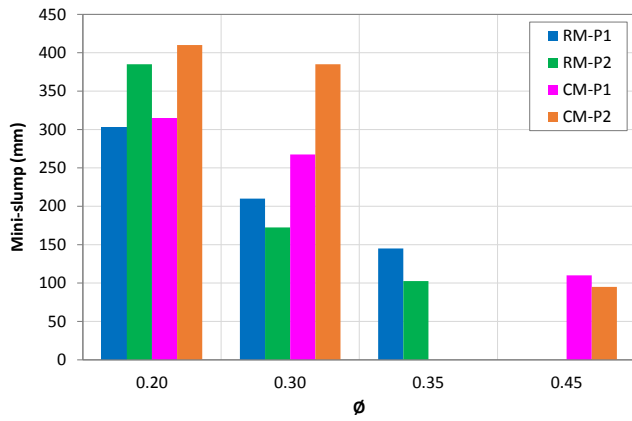


Fig. 11. Mini-slump results.

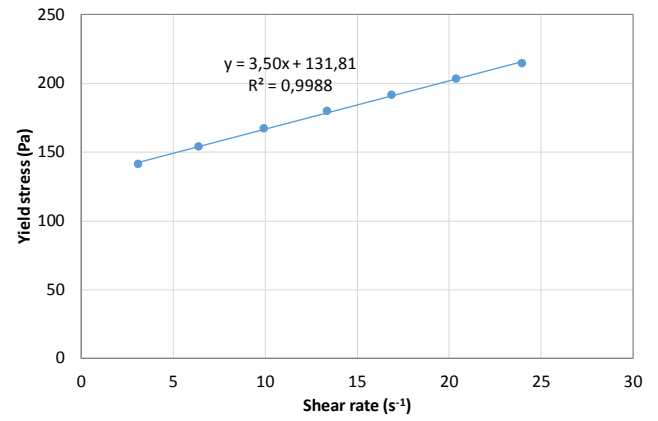


Fig. 14. Flow curve adjustment of RM-P2 (phi = 0.30).

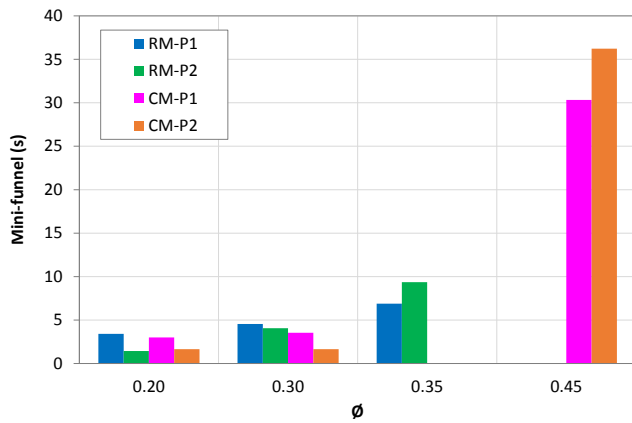


Fig. 12. Mini-funnel results.

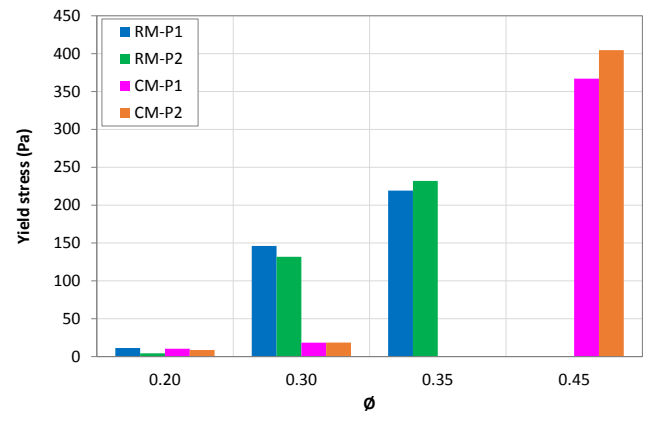


Fig. 15. Yield stress results.

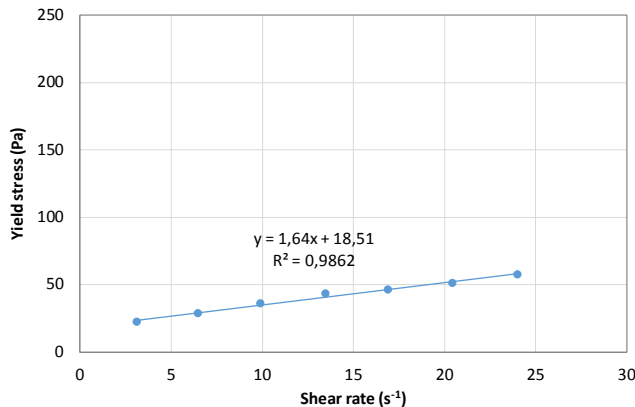


Fig. 13. Flow curve adjustment of CM-P2 (phi = 0.30).

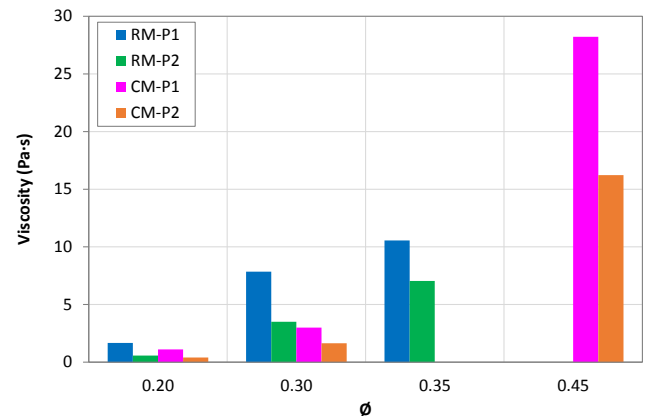


Fig. 16. Viscosity results.

conventional concrete in yield stress and viscosity are low. However, as the solid volume increases these differences also increase.

Regarding the mixing protocol, protocol 2 (longer mixing time and longer admixture addition delay time) enhances both rheological parameters when the paste volume is high (i.e. phi = 0.20 or 0.30) for both conventional and recycled mortars. However, when the paste volume is low (i.e. phi = 0.45 or 0.35) the yield stress seems to be negatively affected by mixing protocol 2, whereas the viscosity seems to be improved. This tendency can be observed for both conventional and recycled mortars.

5. Discussion

5.1. Relationship between rheological and empirical results

With the development of self-compacting concrete, many industrial tests were developed to characterize its fresh behaviour. The slump flow test is an industrial test that provides information about flowability of the mix under unconfined conditions, that is, the capacity of a mix to flow under its own weight. V-funnel was also developed to get information about filling ability or flowability although as the shape restricts flow it gives some indications of the susceptibility of the mix to blocking.

Rheological tests provide information about the concrete as a fluid, measuring yield stress (the stress needed to initiate or maintain the flow) and plastic viscosity (the resistance of the mix to flow once this has been initiated).

The industrial tests provide qualitative information about workability and they usually correlate well with quantitative rheological measurements. It is widely accepted in the literature that there are some relationships between the empirical and rheological parameters [35,36]. Yield stress shows a strong relationship with the slump flow value while viscosity displays a reasonably good relationship with mini-funnel time [37].

Roussel et al. [38] proposed a relationship that results in an accurate interpretation of the slump flow test in the case of pastes. This relationship reflects that yield stress depends on the slump flow value raised to the power of minus five. Other researcher established exponential or logarithmic trends [22,39].

Figs. 17 and 18 show the relationships between the main rheological parameters measured in this work (that is yield stress and viscosity) and the parameters measured with the industrial tests (slump flow and mini-funnel test respectively). In both these figures, other results obtained from the literature [39–41] have been included to compare the trends obtained in this work.

In Fig. 17, we can observe the relationship between the mini-slump flow diameter and yield stress. It is seen that the results show similar tendency to the one exhibited by the results obtained with conventional mortars (literature points). Therefore, this leads to state that the relationships and models analysed in the literature are suitable and can be used when recycled aggregates are employed.

In Fig. 18, the relationship between the mini-funnel time and viscosity is shown. In this case some scatter can be observed as the mini-funnel test is very sensitive to the operator, especially when flowability is high. However, again the tendency of the experimental values is similar to the one shown by values obtained from the literature. It is seen that viscosity and mini-funnel time are linearly related.

5.2. Effect of the solid volume fraction

Figs. 19 and 20 plot the yield stress and the plastic viscosity while varying the amount and type of sand respectively. Each mortar mix is named using the code “X-Y”, where “X” refers to conventional (C) or recycled (R) mortar and “Y” is the mixing time, 5 or 15 min. The curves were adjusted following the Krieger-Dougherty equation, $\mu_m = \mu_p \cdot (1 - \frac{\phi}{\phi_{max}})^{-[\eta] \cdot \phi_{max}}$, Where: μ_m is the viscosity of the mortar, μ_p is the viscosity of the paste, $[\eta]$ is the intrinsic viscosity of the solid phase, ϕ is the solid volume concentration, ϕ_{max} is the maximum packing fraction

The values of ϕ_{max} (the maximum packing fraction) were obtained from Table 3 while the values of $[\eta]$ (the intrinsic viscosity of the solid phase) were adjusted using regression analysis considering the

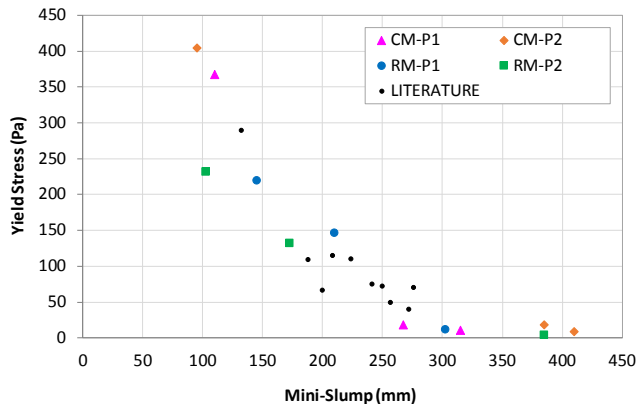


Fig. 17. Yield stress vs Mini-slump flow.

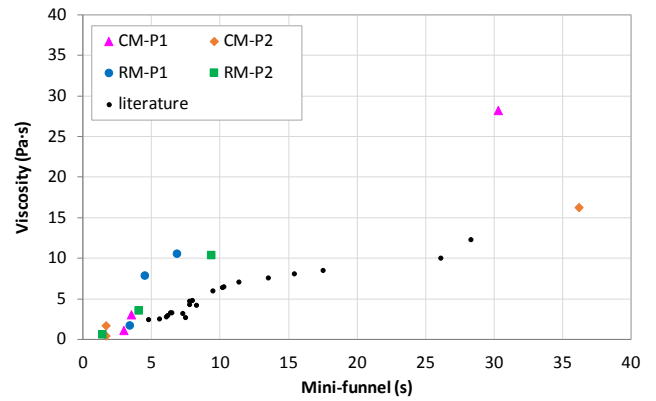


Fig. 18. Viscosity vs Mini-funnel time.

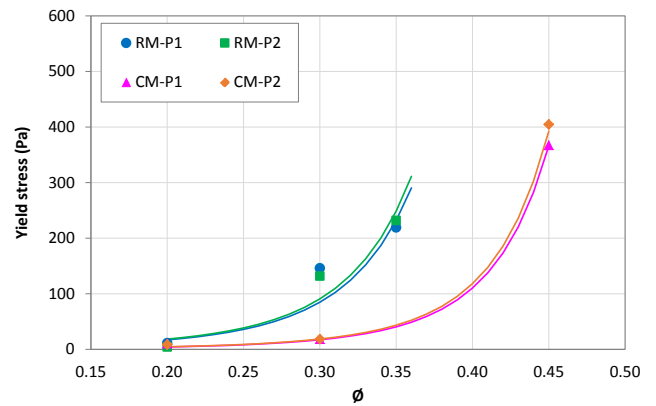


Fig. 19. Yield stress as a function of the solid volume fraction.

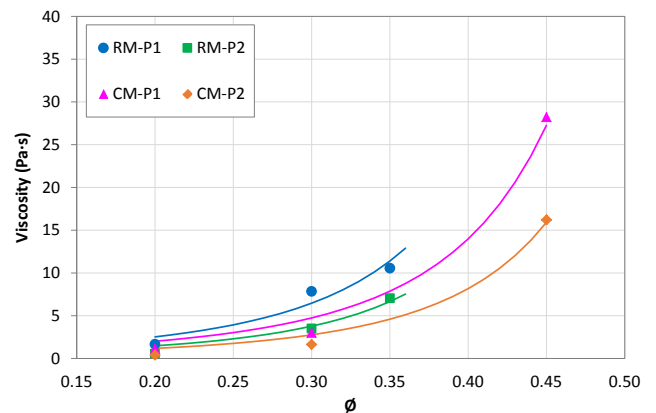


Fig. 20. Viscosity as a function of the solid volume fraction.

recycled and conventional aggregate as the solid phase of the recycled and conventional mortar respectively. The μ_p (the viscosity of the paste) could also be determined with regression analysis considering the Krieger-Dougherty equation where the solid phase is the powder material of the mix, i.e. the cement and fine particles of the aggregates.

As aforementioned, the yield stress and the plastic viscosity were measured in a flow curve test. The former is measured after the structural breakdown of the mortar mix, thereby avoiding the effects of thixotropy.

The viscosity and yield stress varying according to amount and type of sand is given in Figs. 19 and 20. Both, yield stress and plastic viscosity increase as the solid volume fraction increases. The solid volume

fraction is one of the main factors affecting rheology and it can be clearly seen that both mortar with recycled sand and mortar with conventional sand increase in yield stress and plastic viscosity as the solid volume fraction increases. Furthermore, although mix proportions are the same in recycled and conventional mortar, a considerably different rheological behaviour is presented for the same solid content.

According to Figs. 19 and 20 the shape of the rheological curves of a conventional and recycled mortar are clearly different. Recycled sand properties have more of a negative effect than those of conventional sand (even when compared to a crushed conventional sand with more angular and rougher surfaces than a natural rounded conventional aggregate). The rheological curves of recycled mortar rapidly present a very high slope. In this manner, the region of the curves in which small changes in the mix proportions hardly affect the rheological parameters (i.e. the region with a low solid content where the shape, texture or grading of the aggregate hardly affects mortar rheology) is small, leading to the conclusion that the recycled mortar is less robust than the conventional kind.

It is worth mentioning that to achieve the same yield stress in a recycled mortar and conventional mortar, the solid volume content has to be reduced by about 0.10. A similar yield stress was measured in the recycled concrete with 0.35 solid volume content as in the conventional mortar with 0.45 solid fraction. In the case of viscosity, reducing the solid volume content of recycled mortar by about 0.05 allows a similar plastic viscosity to be obtained as that in conventional mortar.

Analysing the curves, it is observed that when the solid content in the mix is low and the paste quantity high (i.e. $\phi = 0.20$) the yield stress and plastic viscosity of recycled mortar are 1.5 and 1.1 higher than those of conventional mortar. These differences, which are especially high in the case of yield stress, demonstrate the certainty that the presence of fine particles of recycled sand play an important role in this behaviour. When the solid content is low the rheological behaviour of the mortar is controlled by that of the paste, the interaction and friction forces among sand particles are low and the negative effect of introducing recycled sand is reflected in the changes that occur in the paste composition due to the fines content of the sands. The differences between the rheological behaviour of conventional and recycled mortar are greater as the solid volume fraction increases. Two characteristics of the recycled sand justify this effect. The first is the rough texture of its particles and its high water absorption capacity and the second is the greater amount of fines incorporated into the recycled sand. This great water absorption capacity is especially observed in the particles made from adhered paste which are more abundantly found in the fine particles.

This is in agreement with the material characterization results. Differences between recycled and conventional sand are more significant in the fine than in the coarse particles. These fine particles modify paste composition and thereby both rheological parameters, although the yield stress is affected to a greater extent, probably due to the water demand of these fines not being accurately corrected with the water absorption compensation value measured in the sand particles. Further research to analyse the effect of the recycled fine fraction on the rheological behaviour of the cement based materials is recommended, in order to establish mix design methods to mitigate this effect.

5.3. Effect of the mixing procedure

Increasing the mixing time and delaying the admixture addition time also play an important role in mortar rheology. Different authors [18,19] state that longer mixing times or more efficient mixing provide more homogeneous systems. Increasing the mixing time allows agglomerates to breakdown, homogenizes the mix and so improves the flow behaviour of cement mortars. However, in the case of recycled aggregate, this counteracts any possible generation of fine particles due to wearing during mixing.

In addition, the later addition of admixtures also improves flowability. The amount of admixture needed to obtain fixed fluidity is

related to the amount of admixture adsorbed by the cement used. It is also known that if a cement adsorbs a large amount of admixture, it is going to demand a high quantity of said admixture to maintain a fixed workability. Changes in the addition time of any organic admixture affects the adsorptive process. The amount of additive adsorbed by the unhydrated phases (especially C3A and C3S) is higher than the amount they adsorb after the dissolution of calcium in water. Therefore, a delay in the addition time reduces the amount of adsorbed admixture as there are less unhydrated phases present, so the admixture remains in the mixing water and improves concrete fluidity.

According to Figs. 19 and 20, it can be established that the effect of these actions (increasing mixing time and delaying the adding time of the admixture) is similar in conventional and recycled mortar. This means that the beneficial effects of improving the fine particle dispersion and reducing the quantity of admixture adsorbed by the cement due to extending the mixing time and delaying the admixture addition time, are more significant than the generation of fines due to wearing. Furthermore, it is clear that the results depend on the solid content. Mixes with a high solid content are more sensitive to any variations and therefore more sensitive to variations in the mixing process than mixes with a low solid volume fraction. Figs. 19 and 20 also show that the effect of changing the mixing process on plastic viscosity is different than the effect on yield stress. In both conventional and recycled mortar, yield stress is hardly affected by the different mixing processes (decreasing when the solid volume fraction is low and slightly increasing when it is high), while plastic viscosity notably decreases with a longer mixing process and a delayed admixture addition time. Vance et al. [42] stated that plastic viscosity is more affected by interparticle spacing and the specific surface area of powder materials, whereas yield stress is more influenced by interparticle forces. Variations in the rheological parameters according to different mixing processes are due to the changes in solid-liquid dispersion, which highly affects interparticle spacing with positive effects on plastic viscosity. Increasing the mixing time and delaying the admixture addition time contribute to breaking any agglomerates present and improving particle dispersion.

The results also show that with a longer mixing time and a delayed admixture addition time, it was possible to obtain a recycled mortar with a viscosity similar (even slightly lower) to that displayed by a conventional mortar manufactured using a short mixing process.

5.4. Rheograph

A rheograph explaining expectable rheological variations, as a function of mixing procedure (mixing time and delayed admixture addition time), recycled sand incorporation and solid volume fraction, in the self-compactability of conventional mortars has been built (Fig. 21). The real objective of the “plastic viscosity – yield stress” diagram is to systematically show the effect of different changes on the

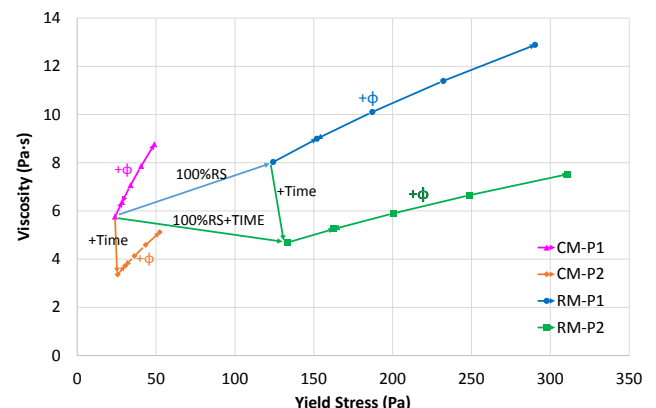


Fig. 21. Rheograph.

rheological behaviour of the cement-based mortars with conventional sand [43,44].

As can be observed in Fig. 21, introducing the recycled sand increases both yield and plastic viscosity. The effect of the mixing procedure can be seen in the reduction of the viscosity and the maintenance of the yield stress. Finally, the increase in the solid volume fraction is reflected by the increase in both rheological parameters, although higher rheological variations are shown in the recycled mortar than in the conventional.

This kind of approach is known as the vectorized-rheograph approach. It is useful to explain the effect of two or three parameters on the rheological behaviour of a concrete mix and although it shows general trends, it sure can help to indicate what is expectable when these changes occur. A rheograph can help to find out where one is, where to go and how to get there [33].

6. Conclusions

In this work a study was carried out on the influence of the mixing procedure on the fresh state behaviour of self-compacting mortar made with conventional and recycled sand. For said purpose, two different mixing times together with two different delayed admixture addition times were applied to mixes where the solid volume fraction was increased. According to the results obtained, the following conclusions can be drawn:

- Independently of the use of recycled sand and the mixing protocol, a strong relationship was found between the yield stress and the mini-slump flow value (in agreement with the those defined in the literature). Concerning viscosity, this parameter showed a reasonably good relationship with the mini-funnel time.
- Both the yield stress and plastic viscosity increase as the solid volume fraction increases. The increment is higher in recycled than in conventional mortar. This is reflected by the slope of curves “rheological parameter vs solid volume fraction”, leading to the conclusion that the robustness of recycled mortar is going to be lower than that of conventional mortar. Recycled sand particles, especially the recycled fines with a considerably high water absorption capacity justify this effect that is more noteworthy in the yield stress than the plastic viscosity.
- Changes in the mixing procedure (longer mixing time and longer admixture addition time delay) similarly affect the rheological parameters of conventional and recycled mortars. The increased mixing time contributes to breaking any agglomerates present and improving particle dispersion, and the delayed admixture addition time reduces the amount of admixture adsorbed by the cement. Applying these changes to the mixing procedure, the yield stress is hardly affected while the plastic viscosity notably decreases. The results lead to the conclusion that with a longer mixing time and a delayed admixture addition time, it was possible to obtain a recycled mortar with a viscosity similar (even slightly lower) to that shown by a conventional mortar manufactured with a short mixing process.

Finally, in this work, using rheographs it was possible to explain what rheological changes are expectable in a conventional mix when recycled sand is incorporated, the solid volume fraction is increased and changes are applied to the mixing procedure (increasing both the mixing time and the admixture addition delay time).

CRedit authorship contribution statement

Belén González-Fontebó: Conceptualization, Formal analysis, Investigation, Writing – original draft, Writing - review & editing, Supervision, Visualization. **Iris González-Taboada:** Formal analysis, Investigation, Writing - review & editing. **Diego Carro-López:** Formal analysis, Investigation, Writing - review & editing. **Fernando Martínez-**

Abella: Formal analysis, Investigation, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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