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Rheology of selfcompacting mortars with sustainable binders

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ABSTRACT

According to the data related to cement consumption in Europe during the period 2000-2013, the construction sector is demanding more and more the cement with additions, and currently the norm for the composition of cement is under revision to incorporate new cement types. The use of ternary cements, cement with large volume of two supplementary materials, as for example blast-furnace slag and fly ash, is one of the strategies investigated to improve sustainability in the construction. In this line, previous work focused on the study of the physico-mechanical properties from the very first age for reference mortar (R1) and mortar with blended cement (SF2) containing a 26% of slag and 10% of fly ash. Additions have an effect in the hydration rate of ternary cements; curing temperature has proved to be a key aspect in the accuracy of the measurement of early ages properties as rheological evolution and shrinkage, since directly influences the hydration process. Conclusions from the previous work showed that there is very high instability during the five initial hours, when a transformation from a viscous suspension into a porous rigid solid is happening; this period has a high influence on the interpretation of the experimental results. In this line, a better understanding in how the binder-admixture affects rheology of mortars when there is a difference in temperature is needed.

Keywords: cements, rheological characterization, temperature effect, experimental techniques

1. INTRODUCTION

1.1. State of the art

The use of ternary cements, cement with large volume of two supplementary materials, as for example blast-furnace slag and fly ash, is one of the strategies investigated to improve sustainability in the construction. Additions have an effect in the hydration rate of ternary cements; curing and environmental temperature has proved to be a key aspect in the accuracy of the measurement of early ages properties as rheological evolution and shrinkage, since directly influences the hydration process.

It is already well know that there are many factors affecting rheology: water-solid ratio; chemical composition of cement; chemical reactivity of supplementary materials; particle size distribution, specific gravity, surface texture and geometrical shape of powders (cement and filler); properties of chemical admixtures; temperature and humidity; initial mixing conditions; testing procedure. According to references, out of them, w/b ratio and specific surface are among the most relevant, while chemical composition has a lower effect. However the chemical composition of the cement in combination with the type of admixture have also a relevant influence since the effect of the SP depends on the cement chemistry [1-5].

In addition to the differences in PCE, it must be studied the rheology of the cement systems containing mineral additions. With aluminious materials such fly ash, yield stress and plastic viscosity are generally noted to decrease with increasing fly ash content. Reduction in yield stress can be attributed to the spherical shape of fly ash particles and their consequent ability to impart fluidity to the paste. If in addition the particle size is coarser than OPC, then the physical separation can be increased. In ternary combinations the influence of additions in yield stress and plastic viscosity are complex [5-6].

There is also a high dependency with the equipment used and methodology being followed. Nendi et al. used four different rheometers setups with cement pastes of 100% OPC, binary blends with slag and fly ash, and also one with superplasticier stabilizer [7-8]. Six rheological models were used to calculate the rheological parameters from the experimental data. Generally, yield stress increases with a reduction of w/b ratio and plastic viscosity also increases with a reduction of w/b ratio. Consistency values increased with a reduction of the w/b for all cement pastes, irrespective of the test geometry used and rheological model considered. Rate index decreased with a reduction of w/b. RMA are also used in 3 dosages and it shows that increases both yield stress and viscosity. In the conclusions of that work, Modified Bingham models estimates lower yield stress values and gives the higher values of plastic viscosity. This model reflects better the influence of low shear strain region. Herschel-Bulkley model shows higher yield stress values [9-13].

For pastes and mortars, a variation of suspension density may occur due to several effects [15-18]:

- Particle sedimentation to the bottom of the container. It is related to liquid viscosity and density, particle diameter, shape and density and fractional volume concentration of the dispersed particles. This leads to a decrease torque with time that can give misleading results. (In our studies when it was a very high particle sedimentation it was marked as wrong results in the program).
- Wall effect, either slippage or particle sedimentation. In some studies the use of rough surface wall limited this effect.

In our study, in order to reduce those effect the following measurements were considered:

a) to use a combination of the paddle and scrapper for helping to move particles to the center and better vertical particle size distribution;
b) to keep the w/b ratio below 0,4 as recommended for coaxial cylinder test.

1.2. Objectives of the work

Two different objectives are followed with this work: first, to study the changes with time in rheological performance for different cement systems; second, to look after the effect of temperature in the stability a flowability of the mixes. In both cases two different w/b ratios were tested.

2. MATERIALS AND EXPERIMENTAL PROGRAM

2.1. Materials used

Experiments were carried out using two different types of cement and two water-to-cement ratios (w/c) of 0.30, 0.40. The two cement types were a Portland cement used as a reference (R1) and one blended cement, SF2, that was prepared in the laboratory with the following proportion: 64% of R1, 26% of slag and 10% of fly ash. The characteristic of the materials are presented in Table 1, and the mineralogical characterization of the blended cement are presented in Table 2. Majority phase of all is the alita or C3S; the determination of content on amorphous phase is in relationship with the content of supplementary component.

Table 1. Chemical characterization of materials used.

		Na_2O	K_2O	Al_2O_3	CaO	Fe_2O_3	SiO_2	MgO	SO_3
F	*Fly ash*	*1.53*	*2.50*	*27.03*	*2.75*	*9.07*	*53.02*	*1.76*	*0.52*
S	*Slag*	*0.15*	*0.66*	*11.87*	*41.29*	*0.63*	*36.42*	*6.87*	*0.29*
R1	CEM I 42,5R/SR3	0.18	0.34	4.68	60.3	5.08	17.4	1.78	3.17

Table 2. Mineralogical characterization of cements used.

	R1	*SF2*
C_3S	69.15	36.87
C_2S	6.37	2.62
C_3A-c	0.61	0.45
C_3A-o	2.13	0.74
C_4AF	12.34	7.39
CaO	0.4	0.00
Amorph	--	45.14

The particle size distribution representing the 10%, 50% and 90% of sample smaller than the size indicated d10, d50 and d90 respectively, and average particle size (dm) are listed in Table 3.

One polycarboxylate ether (PCE) HRWR was used. According to the manufacturer the main characteristics of superplasticizers are listed in Table 4. Sand used for the preparation of the mortars was normalized sand according to EN Standard.

Table 3. Information about the particle size.

	d_{10} *(μm)*	d_{50} *(μm)*	d_{90} *(μm)*	d_m *(μm)*	*Specific surface area* (m^2/g)	*Density* (g/cm^3)
R1	2.499	14.323	40.126	19.33	1.12	3.19
SF2	2.377	13.218	39.280	18.42	1.14	3.02

Table 4. Chemical characteristics and configuration of superplasticizers.

	φ (g/cm^3)	*RI*	*pH*	*Cl (%)*	*Alkali (%)*
SP2	1.04 ± 0.02	20 ± 1.0	5.5 ±1.0	0.1	1.0

Mixing profile for mortars was modified to better guaranty the particles dispersion and the water-admixture system. All powder were dry blended prior to wet mixing. Superplasticizer and water were added together. After 30 seconds of mixing the cement with the water the sand was added in the following 30 seconds. For 2 minutes the mixer was at higher speed. After 1 1/2 minutes of resting, again a higher speed was runt for other 2 minutes. That means that the initial measurements, what in this work is call time 0, it is at 7 minutes after contact of the water with the cement.

Mortars have a cement to sand ratio of 1:2. The optimum dosage of admixture to ensure proper dispersion, beyond which no further significant improvement in fluidity can be achieved, was determined (slump and flow time, using the mini-cone and Marsh cone). Samples were produced to get a similar slump of 300 mm. In the amount of superplasticizer only the solid content was considered.

2.2. Equipment and testing program

The measurement of rheological properties were carried out using a rotation viscometer Viskomat NT (see Figure 1). This device is a modified coaxial cylinder viscometer whose inner cylinder was replaced by a concentric measuring paddle. This entails the advantage that sedimentation cannot occur. Furthermore, mortars with a grain size of 2 mm can be investigated because a wall-slipping effect is minimized. The Viskomat NT is equipped with a tempering device (a cooling bath enabled constant temperature conditions during the test), so that steady state conditions are ensured during the investigation time of e. g. 90 min. For mortars a profile was set that have a step increment o decrement of rotational speed, and a constant rotational speed at 20 rpm and 120 rpm. This relatively high "shear stress" was chosen in order to quickly dissolve a possible initial flocculation and to be able to compare the results with other investigations.

The temperatures studied were 5ºC, 10ºC, 20ºC and 37ºC. The test performed are listed in Table 5. Table 6 present the basic parameter for selfcompactability characterization.

Table 5. Test performed in mortars with the rheometer.

%SP	*w/b*	*Temperature*				*SERIES*
		5 ºC	*10 ºC*	*20 ºC*	*37 ºC*	
0.75	0.40	R1	R1	R1	R1/R1*	R1_04_07_2
		SF2	SF2	SF2	SF2*	SF2_04_07_2
3.00	0.32	R1*	R1*	R1*	R1*	R1_03_3_2
		SF2*	SF2*	SF2*	SF2*	SF2_03_3_2

*Ramp profile

Table 6. Results from slum flow and Marsh funnel.

	D_f (cm)				*t_{funnel} (s)*			
	t_0	t_{30}	t_{60}	t_{90}	t_0	t_{30}	t_{60}	t_{90}
R1_04_07	28	29	27	21	3.16	3.44	5.16	7.1
SF2_04_07	30	31	29	28	3.18	3.18	2.78	4.4
R1_03_3	30	--	--	--	4.23	--	--	--
SF2_03_3	31	--	--	--	3.60	--	--	--

With this rheometer steps profile was set in most cases, from 20 to 120 rpm. The sample is exposed to a higher increment of energy with the step profile than the ramp profile, since it has to reach the rotational speed of 120 rpm in ½ minute instead 2 minutes. In Figure 1 different phases can be appreciated: a liquifying effect during the first cycle, steady state periods in the following cycles, and stiffening period in some cases. However it was not possible to use safely this profile with all the test, so a ramp profile had to be alternatively programmed. Both profiles are correlated for the same mortar sample (see Figure 1).

A constant shear profile (shear rate of 120 rpm) was chosen to visualize the stiffening effect. When the material is tested at a constant speed of 120 rpm the difference between maximum and minimum torque values are larger; this difference is defined as stiffness of the cement system and it give us information about the homogeneity of the system and building up of a changing structure.

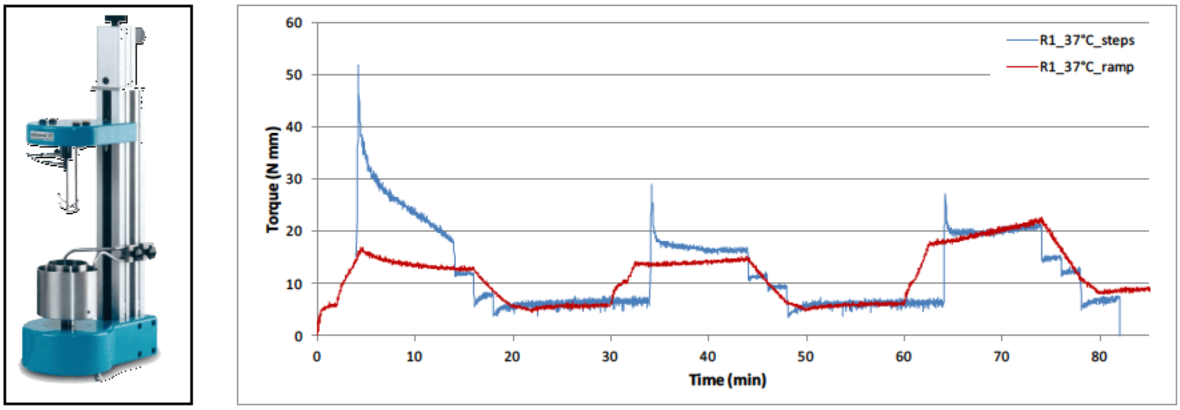


Figure 1. Results for reference mortar using step and ramp profile with Viskomat NT.

3. RESULTS OF TESTING

3.1. Results for different binders

Figure 2 plots the register of torque versus time. First it can be observed is the different response with energy: at 20 rpm registers are very similar; however, at 120 rpm interaction between particles, free water, different superplasticizer adsorption, have an important role in the answer. It needs one cycle to reach equilibrium. After the first cycle, the response is quite stable.

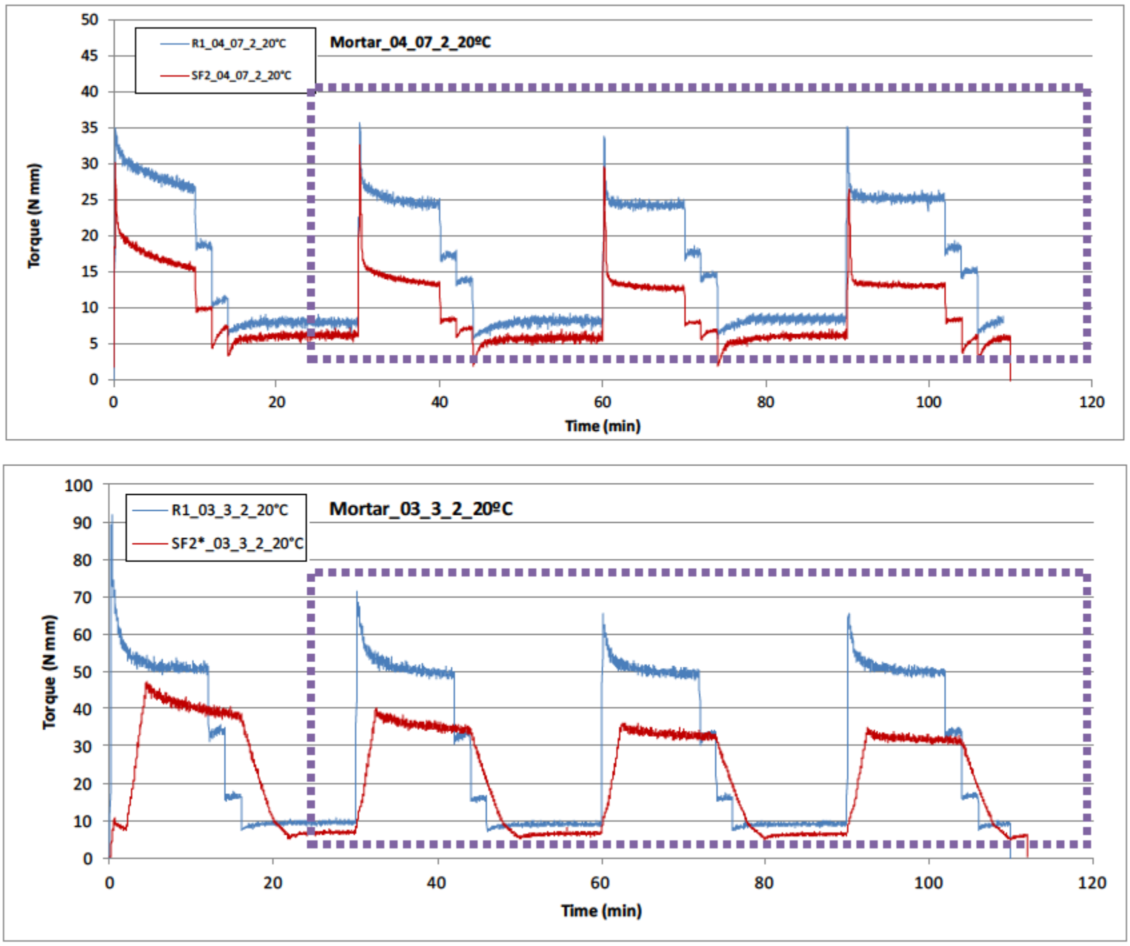


Figure 2. Influence of the time on rheological behavior for different binder.

Regarding the response for different cement systems, reference mortar has a higher resistance to flow and it is a more stable cement system as can be observed specially in the first cycle.

3.2. Results for different temperatures

There is a strong temperature dependency in the rheological behavior. There is an inverse relationship between temperature and resistance to flow; the lower the temperature the higher the torque value measured. However more reactive cements may observe a negative effect with temperature and a strong loss of workability with time (see Figure 3).

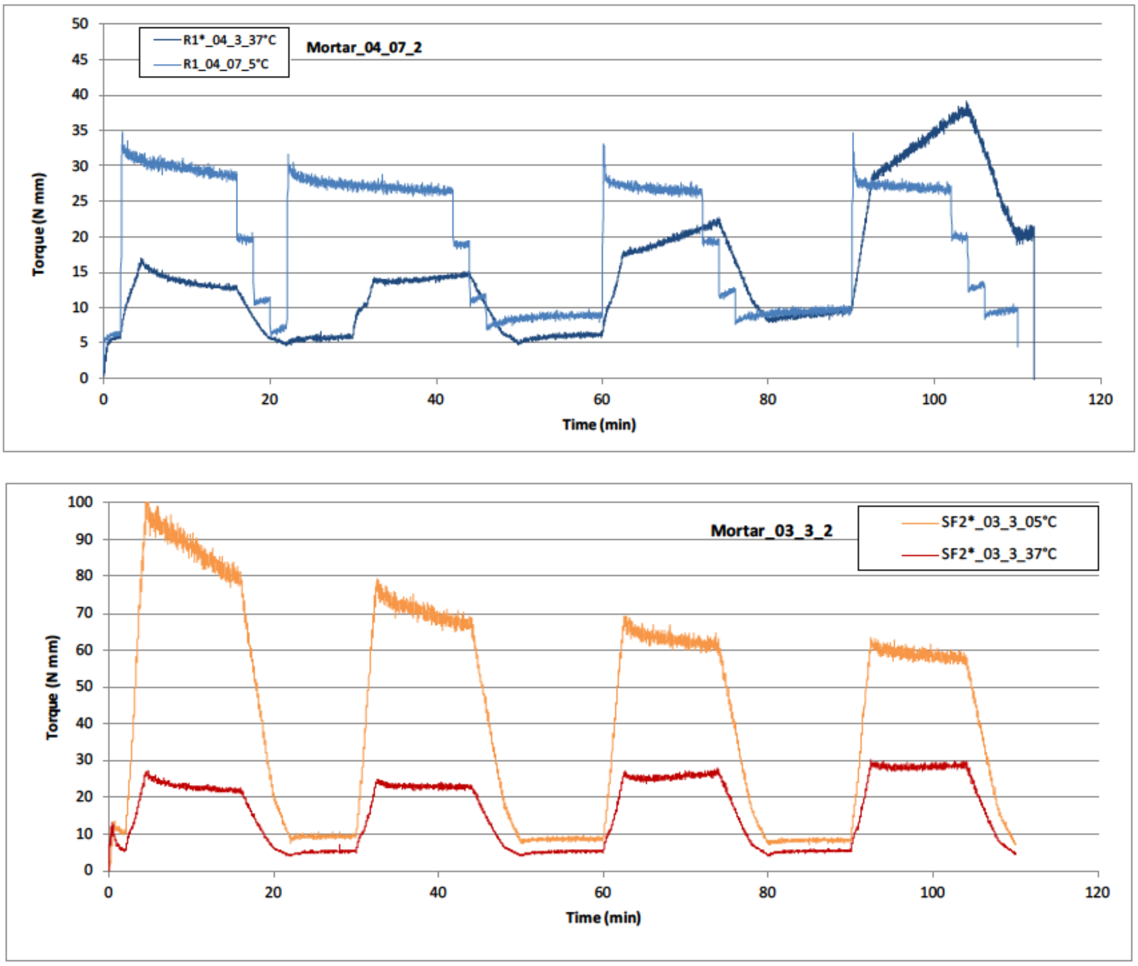


Figure 3. Influence of time on rheological behavior for different temperatures.

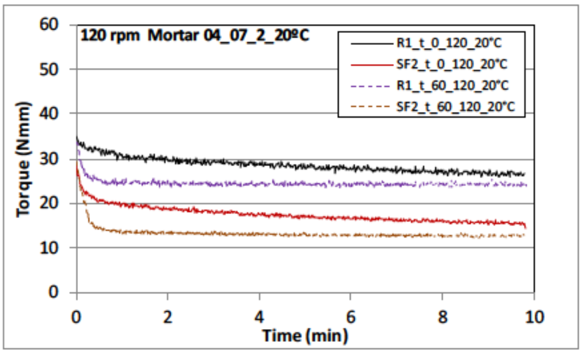
4. DISCUSSION OF THE RESULTS

Two different results are discussed in this paper: stability of the mortar with time at the higher rotational speeds, and modification of stiffening with time and temperature.

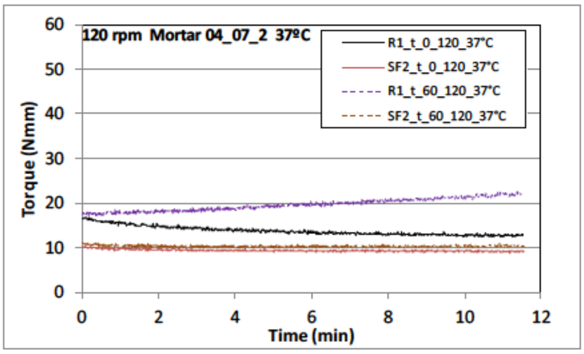
4.1. Influence of temperature on stability

In the first cycle, during the first minutes an initial liquefying can be appreciated, followed by a more homogeneous state. Blended systems need more time to reach the steady state region as after 10 minutes of continuous rotational speed, have not reached a clear steady state. After 60 minutes of continuous stirring, the torque value is reduced, and the state more steady (see Figure 4a). With temperature torque values are reduced in all cases, mortars reach the stead state at earlier times, and some accelerated stiffening may be observed (see Figure 4b). This phenomena is more accused for reference mortars due to the earlier hydration processes.

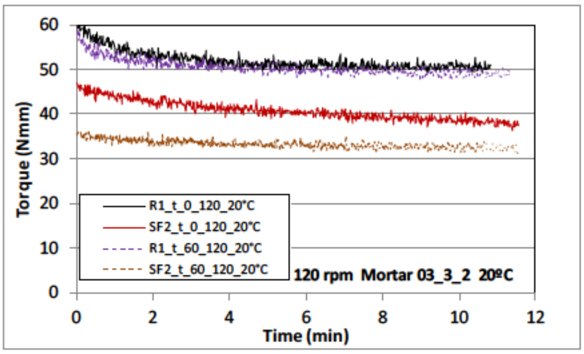
Tendency is similar for mortars with w/b ratio of 0.3 (see Figures 4c and 4d). This is in agreement with some results from mini-slump test. It is needed 90 minutes (or higher temperature) for getting a strong stiffening in the cement systems.



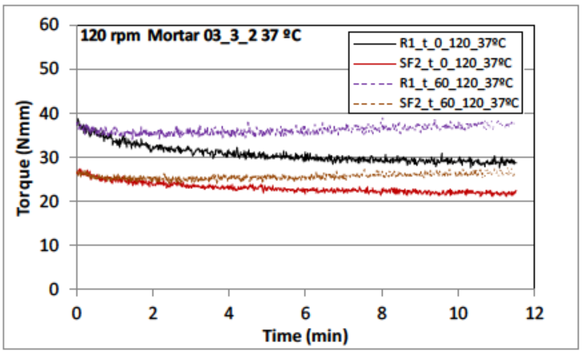
a) w/b ratio of 0,4, at t=0 and t=60 min, 20°C



b) w/b ratio of 0,4, at t=0 and t=60 min, 37°C



c) w/b ratio of 0,3, at t=0 and t=60 min, 20°C



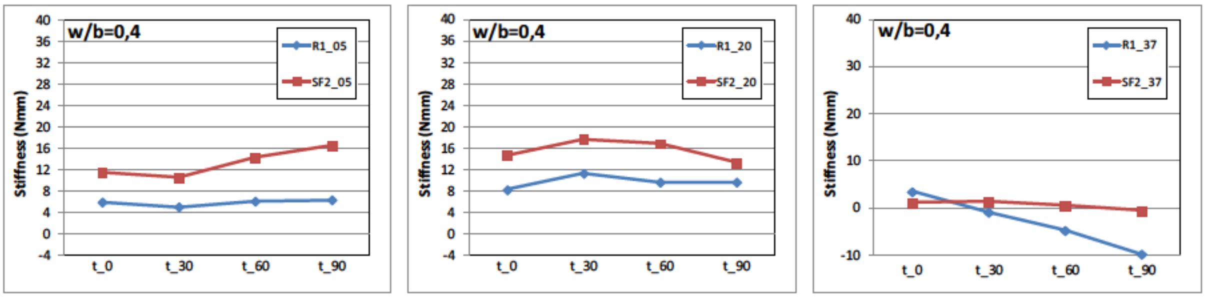
d) w/b ratio of 0,3, at t=0 and t=60 min, 37°C

Figure 4. Structural breakdown with time of the cement systems, for different w/b ratios.

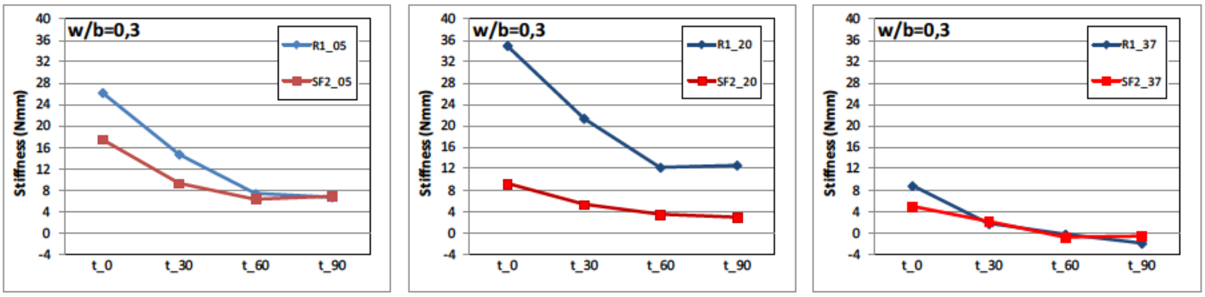
4.2. Influence of temperature on stiffness

Figure 5 plot the temperature influence on stiffness. As it was mentioned in paragraph 2.2 stiffness may give us information about the homogeneity of the system and building up of a changing structure. First it can be observed, is that the mortars with w/b ratios of 0.4 are more homogeneous than mortars with w/b ratio of 0.3 (lower stiffness values). They are also quite stable with time (except reference systems at 37 ºC).

Still, mortars with blended cements are more unstable. Increment in temperature is positive for the structure building-up for blended cements, while for the reference mortar there is a strong loss of workability and increment in resistance to flow (see Figure 5a). Figure 5b present the stiffness variation for mortars with a higher viscosity (w/b ratio of 0.3). The higher values of stiffness report for mortars with poor homogeneity. The strong change of stiffness reports for mortars that build up a structure with time. Again, increment in temperature is positive for the structure building-up for mortars with standard and blended cement.



a) Influence of temperature on mortars with w/b ratio of 0.4.



b) Influence of temperature on mortars with w/b ratio of 0.4.

Figura 5. Stiffness variation with time and temperature.

5. CONCLUSIONS

The test program was prepare to determine the relationship between binder systems and to find out the influence of the temperature on the rheological properties of mortars. Blended systems need more time to reach the steady state region as after 10 minutes of continuous rotational speed, have not reached a clear steady state. After 60 minutes of continuous stirring, the torque value is reduced, and the state more steady.

With temperature, torque values are reduced in all cases, mortars reach the stead state at earlier times, and some accelerated stiffening may be observed. Increment in temperature is positive for the structure building-up for blended cements, while for the reference mortar there is a strong loss of workability and increment in resistance to flow.

Continuous work is being done related to find the model, lineal or non lineal flow curves, with better accuracy of fitting experimental results (is important to check if the model predictions are physically reasonable and correspond to the type of behavior it is trying to predict). In some cases, supplemented experimental test should be performed to check for physical consistency of the results.

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