EFFECT OF ENTRAPPED AND ENTRAINED AIR ON THE WORKABILITY AND RHEOLOGY OF CEMENTITIOUS MATERIALS

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ABSTRACT

The influence of air on the fresh properties of cementitious materials is still a point of discussion in concrete research. On the one hand, fluctuations of entrapped air can change the predicted properties in an undesirable way. On the other hand, it is know that the addition of air entrainment changes the rheology significantly. A profound research on the impact of different methods to change the air content, is necessary. We investigated the influence of vacuum mixing and air entrainment on the workability of paste and mortar. For self-compacting mortar also the influence on the rheology was examined.

For vibrated paste and self-compacting mortar a clear decrease of the workability is determined when air is added. This was also found for the rheology of self-compacting mortar. In case of vibrated mortar an overall increase of the workability occurred, when the air content increased. In conclusion, vacuum mixing can control the air content and changes the workability and rheology in an acceptable way. A more important change of the slump flow and, if determined, the rheology, is obtained by air entrainment. Further research should be performed on the influence of air on the workability and the rheology of ultra-high performance concrete.

Keywords: vacuum mixing, entrained air, workability, rheology

INTRODUCTION

At this moment, various techniques exist to introduce air in concrete. Probably one of the most common is the introduction by air entraining agents (AEA). These active anions lower the surface tension and stabilize the air bubbles. In case of vibrated paste with only AEA, Struble and Jiang, report an increase in yield stress and a decrease in plastic viscosity with a higher amount of air. In contrary, a combination of superplasticizer and AEA gives an increasing yield stress and plastic viscosity with a higher amount of air [1]. They also performed experiments on concrete with only AEA. An increased slump was registered. Again the yield stress tended to increase, while the plastic viscosity decreased [1].

A second type of air, that can be introduced in the mixture is entrapped air. This type is less predictable and often researchers try to control it. Zain et al. investigated the influence of it on the workability of high performance concrete. In conclusion, the slump flow and the V-funnel flow decreased with an increased air content [2]. This type can also be introduced by pumping vibrated or self-compacting concrete (SCC). During the pump tests of Feys et al. [3] the air content was progressively increased each time the concrete passes the pump. From these tests, it was found that the yield stress increased with an increasing amount of entrapped air. In contrary, the apparent viscosity was reduced at higher air contents.

Literature also mention the introduction of air bubbles by the addition of superplasticizers in concrete. Lazniewska-Piekarccyk, reports an increase in air content for some new generation superplasticizers, especially when an anti-foaming admixture is missing. As a side effect, they behave as an air entraining agent[4].

Finally, entrapped air can be controlled by a vacuum mixer. During the mixing process, a lowered air pressure is established in the mixing pan. According to Mazanec et al. a large amount of entrapped air can be removed by this technique [5]. For ultra-high performance mortar (UHPM) an increase of the slump flow was found, when the pressure was reduced from 1013 mbar to 100 mbar.

The previous summary, often correlates a change in air content with a change in rheology and/or workability. This is also done by De Larrard, were the fresh air content is a function of the slump, the fraction of coarse and fine aggregate and the amount of superplasticizer. An improvement of the slump or decrease of the yield stress of a mixture with constant proportioning, leads to a decrease of the fresh air content [6].

In this paper the effect of entrapped and entrained air on the workability of cementitious materials is examined. In case of self-compacting mortar the influence on the rheology was also investigated. A vacuum mixer served to determine the effect of entrapped air. The influence of air entraining agent is examined by a proper dosage of Micro air. First, the effet of AEA on the workability of a simple vibrated paste (VP) is investigated. Next the impact of vacuum mixing or AEA is determined on vibrated mortar (VM). At the end, a self-compacting mortar (SCM) is also been tested on its workability and rheology for both techniques.

EXPERIMENTAL WORK

Materials and mix proportions

In this research, two vibrated pastes (VP), five vibrated mortars (VM), and one self-compacting mortar (SCM) is investigated. The composition of the mixtures is given in Tab. 1.

Table 1. Mix proportioning [kg/m^3]

Materials	VP0.5	VP0.4	VM0.55	VM0.5	VM0.45	VM0.4	VM0.3	SCM0.4
CEM I 52.5 N	1216	1384	499	550	525	620	692	519
water	608	554	275	275	237	248	208	207
Sand 0/4	/	/	/	1450	/	1459	1489	1284
CEN Sand[1]	/	/	1496	/	1574	/	/	/
Limestone filler	/	/	/	/	/	/	/	346
Superplasticizer[%][2]	/	/	/	0.04	/	0.11	0.41	0.76
W/C	0.50	0.40	0.55	0.50	0.45	0.40	0.30	0.40
W/P	0.50	0.40	0.55	0.50	0.45	0.40	0.30	0.24

[1] CEN standard sand to comply with NBN EN 196-1; [2] percentage solid particles of the cement content

Table 2. Chemical composition [%]

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3
CEM I 52.5 N	18.50	5.94	4.15	62.91	0.97	0.46	0.69	3.28

The cement, CEM I 52.5 N, had a d_{50} of 14.3 µm and a Blaine fineness of 4322 cm^2/g The chemical composition of the cement is given in Tab. 2. The limestone filler had a d_{50} of 10 µm. The superplasticizer used in this program was a polycarboxylate ether. It had a molecular weight of 40000 g/mol and a solid content by weight of 35%. Micro air was used as an AEA. For vibrated paste and mortar three different dosages were tested, namely 0.22%, 0.44% and 0.66% of the cement content (by weight). In case of self-compacting mortar more dosages were tested.

Procedure and apparatus

The paste is mixed in a Braun PowerBlend, this caused enough agitation to disperse the AEA properly. A Thermo Haake RS 150 viscometer served as a rheometer, which complies to the DIN 53018. For vibrated and self-compacting mortars an intensive vacuum mixer with inclined mixing drum was used, Fig. 1. An Anton-Paar MCR 52 rheometer examined the effect of air bubbles on the yield stress and plastic viscosity of self-compacting mortar. For paste, the water was first added in the blender and then the cement. Next, a slow mixing phase of 25 s was followed by an intensive phase of 25 s. Between the two phases, the sides of the blender were scrapped. In case of mortar, the dry materials were mixed for 15 s, before the water and superplasticizer were added. The mixing time was determined based on a power curve [5]. For vibrated mortar an intensive phase of 45 s at a rotor speed of 3.3 m/s gave the best workability. For self-compacting mortar an intensive phase of 110 s at 3.3 m/s was applied. In case of vacuum mixing, a reduction from 1013 mbar to 500 mbar or 50 mbar was established the last 45 s or 70 s, respectively for vibrated and self-compacting mortar. If an air entraining agent is used, it is premixed with the water.

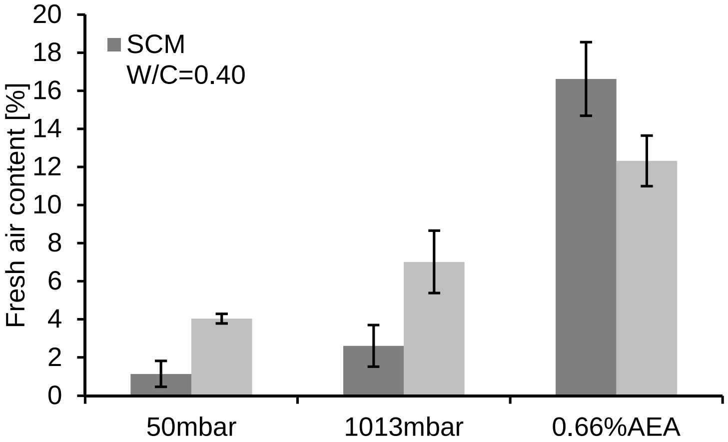
Figure 1. Intensive vacuum mixer with inclined mixing drum.

Next, the slump flow (NBN EN 12350-8) or flow (NBN EN 1015-3) are taken 3 minutes after stopping the mixer. For mortars the fresh air content according to the water column method (NBN EN 12350-7) is also performed. For self-compacting mortar the rheology was tested at the end of the program. A preshear period of 40 s was followed by a decreasing rotational velocity profile. The transformation formulas of Nehdi et al. were used to go from torque-speed T(N) to shear stress-shear rate τ(γ’) [8]. The modified Bingham model was used to fit the τ(γ’) data from which the yield value and the plastic viscosity followed [9].

RESULTS AND DISCUSSION

In this paper, two mechanisms are used to cover a wide range of air contents and examine their influence on the workability and rheology of concrete. A vacuum mixer is used to establish very low air contents, AEA is used to cover higher amounts of air bubbles, Fig. 2. Although more air entraining agent is used for vibrated mortar compared to self-compacting mortar (Cf. cement content Tab.1), the latter reaches a higher fresh air content. This can be attributed to the larger amount of superplasticizer used in SCM [4]. Starting from 1013 mbar, the vacuum technology establishes the largest reduction for vibrated mortar. The minimum fresh air content, obtained with this technique, is registered for self-compacting mortar. The latter can be explained by an increase in workability, leading to a better air release [6]. Furthermore, this technique reduces the standard deviation of the water column method, leading to more reliable measurements.

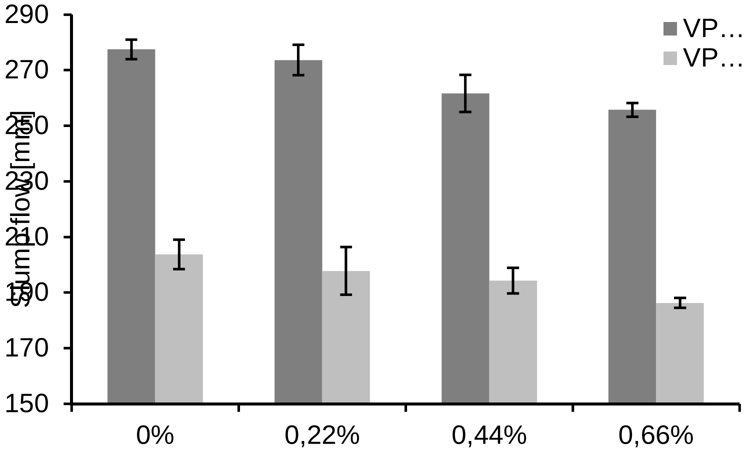
Figure 2. Variation of the fresh air content by vacuum mixing and addition of AEA on self-compacting and vibrated mortar.



At this point, researchers have a good knowledge about the effect of air on the mechanical properties of concrete [10]. In contrary, the influence on the workability and rheology is not that clear. This impact will be addressed in the rest of the paper.

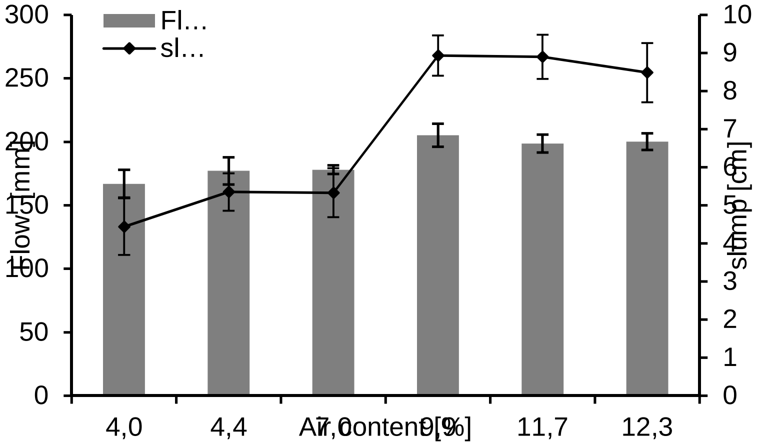
First the effect of AEA on the workability of simple paste mixtures is tested. In conclusion, Fig. 3 gives a decreasing mini-slump flow with an increasing amount of air entraining agent. The yield stress evolved from 7.5 Pa to 9.4 Pa for VP0.5 and from 33.3 Pa to 31.5 Pa for VP0.4. The plastic viscosity varied from 0.16 Pa.s to 0.23 Pa.s for VP0.5 and from 0.96 Pa.s to 1.02 Pa.s. In general, both parameters increased with an increasing dosage of air entraining agent. A plausible explanation for this phenomenon is that of the air bubble bridges. Namely, as cement hydrates the particles become positively charged. The anionic surfactants will adhere to the cement particles, which are on average smaller than the air bubbles. A coating of the bubbles by cement particles is formed, increasing the cohesiveness. These bridges lead to an increase of the flow resistance [1].

Figure 3. Influence of AEA on the workability of vibrated paste.



Next, the influence of vacuum mixing and air entraining agent is examined on vibrated mortar. For VM0.5 three different pressures and three different dosages of AEA were tested. The effect on the flow and the slump were investigated (Cf. Fig. 4).

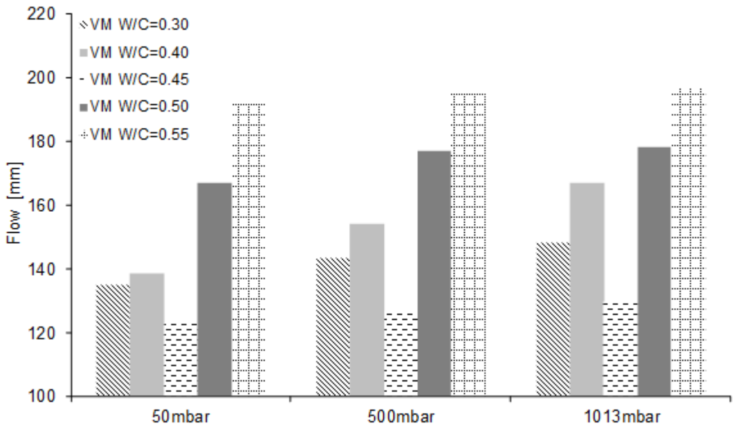
Figure 4. Influence of a reduced air pressure or AEA on the workability of vibrated mortar.



Reducing entrapped air by lowering the air pressure in the mixing pan from 7% to 4%, decreases the workability of vibrated mortar. This can be explained by the capillary number. The shear rate during the workability tests, is probably large enough to overcome the surface tension between the air bubbles and the surrounding mortar. A large part of the air bubbles will deform under this shear rate. Thus, by removing these bubbles by vacuum mixing a higher flow resistance will be obtained [11].

In case of air entraining agent a jump in air content is seen from 7% to 12.3%. As a consequence the workability has the tendency to increase first. At higher levels a slight decrease is noticed. However, this drop is less pronounced compared to the paste level. Probably the sand serves as a grinding agent, destroying some of the air bubble bridges during the test. Consequently, more of the bubbles will act as a fluid reducing the flow resistance of the mortar during the flow and slump measurement [2].

Figure 5. Effect of vacuum mixing on vibrated mortar in function of the water-to-cement ratio.



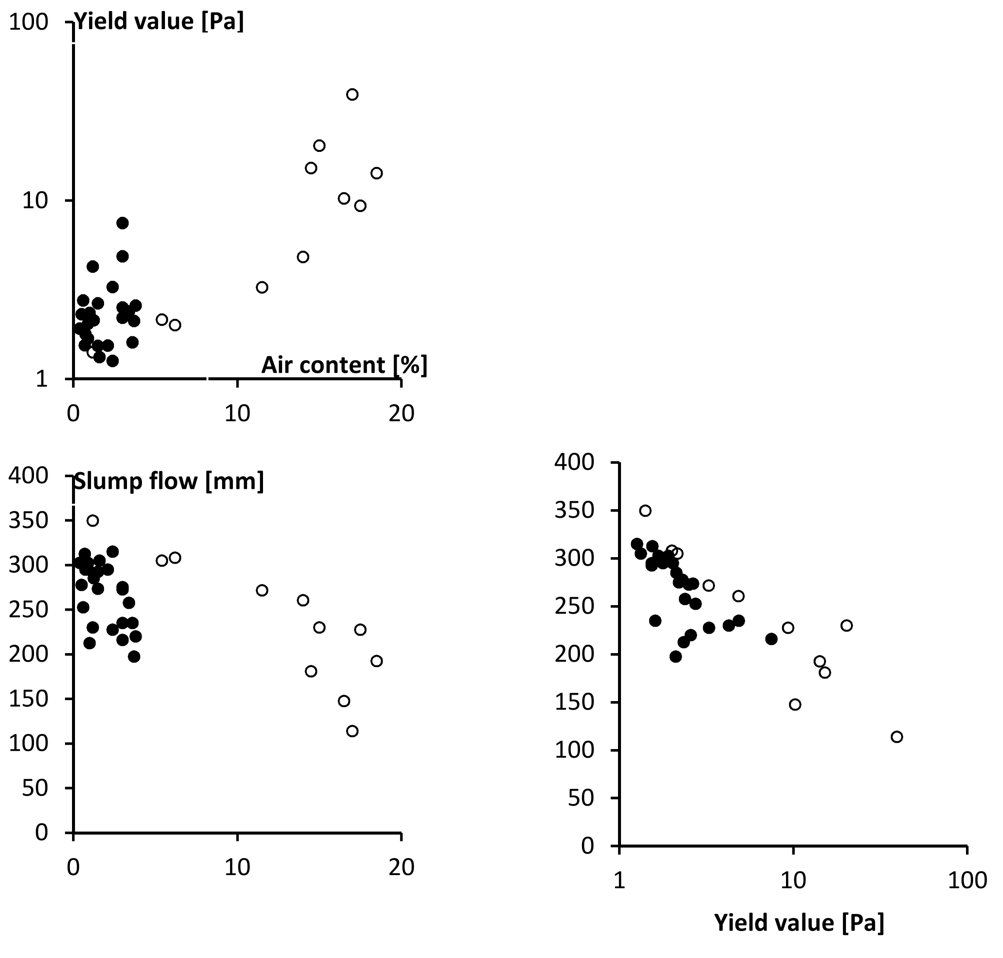
Furthermore, five mixtures with a different water-to-cement ratio were examined, (Cf. Fig. 5 and Tab. 3). For all mortars a decrease of the flow is registered when the pressure drops from 1013 mbar to 50 mbar.The highest decrease was found for VM0.4 and VM0.3. Apparently the amount of entrapped air has a more critical impact on the workability of mixtures with a lower water-to-cement ratio. The low value of the flow for VM0.45 is due to the lack of superplasticizer in this mixture (Tab.1).

Table 3.Fresh air content of the mixtures in Fig. 4 in function of the air pressure

Air pressure [mbar]	1013	500	50
W/C	Fresh air content [%]		
0.30	2.5	3.2	0.4
0.40	3.2	2.5	1.2
0.45	2.2	1.7	0.7
0.50	3.5	1.9	0.8
0.55	/	/	/

Finally the effect of air entraining agent and vacuum mixing is examined for self-compacting mortar. Again the higher air contents were reached by the AEA (designated with empty circles) and the lower values by an air content reduction (designated with full circles) (Cf. Fig. 6 and Fig. 7).

Figure 6. Effect of the fresh air content on the slump flow and yield value of SCM.



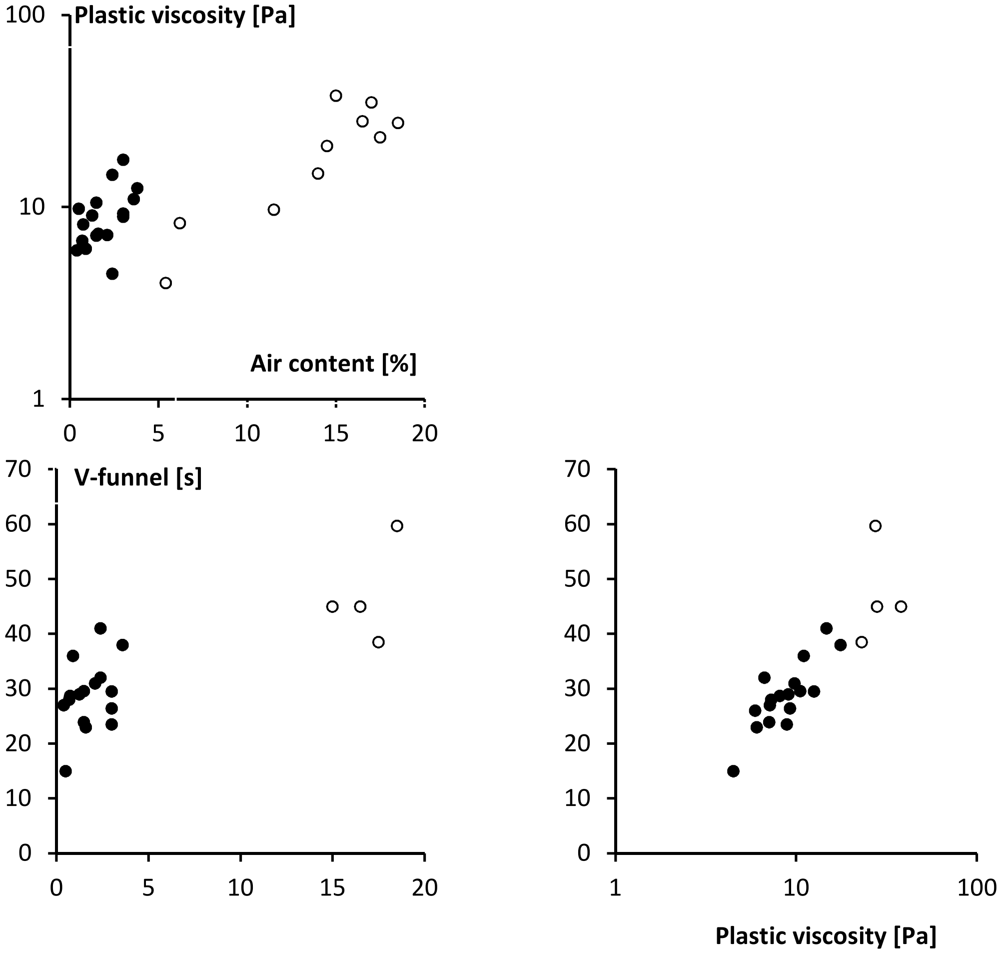
In conclusion, Fig. 6 gives an increase in yield value when the fresh air content is varied from almost 0% to 18.5% by vacuum mixing and air entraining agent. It seems that the AEA has a more significant influence on the yield value than the changes obtained by the lowered air pressure. The increase, by addition of AEA, was also found by Struble and Jiang for vibrated concrete [1]. For the slump flow, a decrease is plotted in Fig. 6. This is a reasonable result, as the parameter is directly correlated to the yield value [12]. Namely, an increase in yield value, leads to a lower slump flow. Apperently for vacuum mixing, a steaper decrease of the slump flow is determined in function of the air content compared to the variation as a consequence of air entraining agent.

The decrease in workability, in case of AEA is due to the air bubble bridges, as explained on paste level. In case of vacuum mixing the workability reaches a maximum at low values of entrapped air. This effect can be explained by the capillary number. Different from vibrated mortar, the viscosity of self-compacting mortar is much lower. Therefor, the shear force during the slump flow will be too small to overcome the surface tension between the air bubbles and the surrounding mortar. As a consequence, the bubbles will keep there form and act as an obstacle during the slump flow test. Thus, removing them will lead to a better workability [11]. In order to verify this hypothese, new techniques should be used. One possibility could be small angle light scattering (SALS).

Finally the correlating between the yield value and the slump flow is confirmed in this work. An increase in yield value reduces the slump flow. The autors also want to stress the high scatter of the yield value obtained in this work. A similar scatter was found by Struble and Jiang in case of AEA [1]. It was attributed to the difference in sizes and spatial distribution of the air voids.

Fig.7 gives the results of the plastic viscosity and the V-funnel. Increasing the air content leads to a higher value of the viscosity. Different as for the yield value, a significant increase of the viscosity is also obtained when vacuum mixing is applied. The increase is again steaper compared with the increase due to AEA. Similar results are found for the V-funnel. An increase of the workability is determined when the air content increases. At the end, a good correlation was obtained between the plastic viscosity and the V-funnel. Increasing the viscosity by a higher air content, leads to a higher V-funnel time. A similar conclusion was found in literature [13]. Again these evolutions can be explained in a similar way as for Fig. 6. The increase of the viscosity and V-funnel time in case of air entraining agent can be attributed to the air bubble bridges. In case of vacuum mixing, removing undeformable air bubbles leads to a lower flow resistance and thus a lower viscosity and workability. Fina

Figure 7. Effect of the fresh air content on the V-funnel and plastic viscosity of SCM.



CONCLUSIONS

In this paper the effect of entrapped and entrained air on the fresh properties of vibrated mortar and paste, and self-compacting mortar is investigated. The following conclusions can be made:

- Vacuum mixing not only reduces the fresh air content of vibrated and self-compacting mortar, it also decreases the standard deviations of the water column method.
- The workability of vibrated paste decreases when air entraining agent is added. In case of mortar the workability first increases. At higher levels of AEA it started to decrease. However, this was less pronounced than for paste. When vacuum mixing was applied a decrease in workability was determined for vibrated mortar. A possible reason is the removal of deformable air bubbles by the lowered pressure inside the mixing pan.
- For self-compacting mortar, both the workability as the rheology decrease at higher amounts of air bubbles. In case of AEA this was attributed to the formation of air bubble bridges. The vacuum technique removed undeformable air bubbles,

as a consequence the workability and rheology increased. A possible way to investigated these hypotheses could be small angle light scattering (SALS).

- The relationships between the rheological parameters and the workability, found in the literature are confirmed in this project. The yield value is strongly correlated to the slump flow and the plastic viscosity to the V-funnel.

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