ГИАБ. Горный информационно-аналитический бюллетень /
MIAB. Mining Informational and Analytical Bulletin, 2022;(10-1):194–204
ОРИГИНАЛЬНАЯ СТАТЬЯ / ORIGINAL PAPER

УДК 622 DOI: 10.25018/0236_1493_2022_101_0_194

ОЦЕНКА ТЕКУЧЕСТИ САМОУПЛОТНЯЮЩИХСЯ СТРОИТЕЛЬНЫХ РАСТВОРОВ С ИСПОЛЬЗОВАНИЕМ НОВОГО ОПТИЧЕСКОГО МЕТОДА

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Аннотация: Свойства вязкости строительных растворов, содержащих различные количества суперпластификатора и модификатора вязкости, исследованы с использованием нового оптического метода, основанного на методе построения таблицы расхода/растекания. Поток раствора отслеживается с помощью высокоскоростной камеры, а кривые потока генерируются с помощью программного обеспечения для постобработки. Наши результаты показывают, что оптический метод обладает очень высокой точностью оценки вязкости цементирующих материалов, а также привносит новые возможности в традиционный метод. Вязкость строительных растворов, содержащих различные количества суперпластификатора и модификатора вязкости, исследованная оптическим методом, подтвердила результаты предыдущих исследований. Добавление суперпластификатора улучшило обрабатываемость строительных растворов, но оказало неблагоприятное воздействие в виде растекания и расслоения. Добавление модификатора вязкости, помогает предотвратить эти явления, однако, существует необходимость в соблюдении осторожности с используемым количеством модификатора, так как он резко повлиял на вязкость исследуемых строительных растворов.

Ключевые слова: самоуплотняющийся бетон, обрабатываемость, предел текучести, пластическая вязкость, таблица расхода, суперпластификатор, модификатор вязкости.

Для цитирования: *Металлари А., Вахаб М., Бир Т.А.* Оценка текучести самоуплотняющихся строительных растворов с использованием нового оптического метода // Горный информационно-аналитический бюллетень. – 2022. – № 10-1. – С. 194–204. DOI: 10.25018/0236_1493_2022_101_0_194.

Evaluating the flow properties of self-compacting mortars using a new optical method

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Abstract: The flow properties of mortars containing different amounts of superplasticizer and viscosity modifying agent are investigated using a new optical method based on the flow/spread table method. The flow of the mortars is tracked down using a high-speed camera and the flow curves are generated using post-processing software. Our findings show that the optical method has a very high accuracy in estimating the flow behavior of cementitious materials at each point in time, while bringing also new features to the traditional method. The flow behavior of the mortars containing different amounts of superplasticizer (SP) and viscosity modifying agent (VMA) investigated with the optical method confirmed the findings of previous studies. The addition of superplasticizer improved the workability of the mortars but had the adverse effect of bleeding and segregation. The addition of the viscosity modifying agent helped against these phenomena, but care must be taken with the amount used, as the viscosity modifying agent drastically affected the flow behavior of the investigated mortars.

Key words: Self-compacting concrete, workability, yield stress, plastic viscosity, flow table, superplasticizer, viscosity modifying agent.

For citation: Metallari A., Wahab M., Bier T. A. Evaluating the flow properties of self-compacting mortars using a new optical method. *MIAB. Mining Inf. Anal. Bull.* 2022;(10-1):194—204. [In Russ]. DOI: 10.25018/0236_1493_2022_101_0_194.

1. Introduction

In the last decades, the development of viscometers and rheometers has been subject to great improvements [1]. They are widely used amongst many workers to quantify the rheological parameters such as yield stress and plastic viscosity of cementitious materials. Their use however comes also with drawbacks. One of them is the possible discrepancy in quantifying the rheological parameters. Different rheometers with different geometries of container or propeller will provide different results for yield stress and viscosity [2]. This is more related to the fact that concrete in its fresh state is a non-Newtonian fluid with rather complex behavior and it has been very difficult to extract consistent results from different rheological setups. The complexity of the rheological behavior of mortar and concrete, comprising of particles of very different shapes and sizes, complicates the matter even further [2]. For this reason and also for their flexibility and practical usefulness, empirical test methods are still amongst the favorite tools to get an indication of the quality of pastes, mortars, or concrete in terms of rheological behavior.

One of the most used empirical test methods is the famous slump test which is also the oldest test used for rheological indications of concrete. It was developed in the USA more than a century ago by Duff A. Abrams [3], [4]. In this test, a cone of given dimensions is filled with concrete, the cone is lifted and then one single value, the slump value, is measured.

A few decades after the use of the slump test another empirical method, the flow/spread table test was developed in Germany by Graf [5]. Unlike the slump test where the slump (the difference between the height of the cone and the height of the concrete after the cone has been lifted) is measured, in the flow/spread table test the final diameter of the material after it has stopped flowing is measured. This is because the flow table test is intended for materials that flow well (e.g. self-compacting concrete) and not for materials that do now flow (e.g. traditional concrete). There are also other empirical test methods such as the L-box test, V-funnel test, and J-ring test, used for the highly flowable concrete, but these tests are beyond the scope of this paper. More information on these tests however can be found elsewhere [6]–[8] and also in some DIN EN norms [9], [10].

All the empirical test methods mentioned above are almost always operator-sensitive, as already stated by

Wallevik [11]. This infers that the human-induced error during the execution of the test is large enough to give a different result every time the test is repeated. In this paper, we discuss a new measurement method developed in our laboratory which addresses human-induced errors and brings some advantages compared to the traditional empirical test methods. Our current optical method is based on the empirical method of the flow/spread table test. After a series of adjustments and improvements, the repeatability of the method was verified and will be discussed here. Using this new method, flow measurements on mortars containing different amounts of superplasticizer (SP) and viscosity modifying agent (VMA) were conducted. The results confirmed the findings of other studies on the effect of these admixtures on the flow properties of cementitious materials [12]–[16].

2. Experimental

2.1. Raw materials

Portland cement (PC) of class CEM I 42.5 N according to DIN 197-1 with a density of 3.4 g/cm^3 was used as a binder. The aggregate used was natural sand with a maximum grain size of 2 mm. The particle size distributions of both, aggregate and cement are given in Fig. 1.

The chemical composition and physical properties of the Portland cement used for this study are given in Table 1. The chemical composition was characterized using X-ray fluorescence spectroscopy while the fineness of the cement was determined using the Blaine air-permeability apparatus which measures the fineness of the cement in terms of the specific surface area expressed as total surface area in square centimeters per gram.

Two admixtures were additionally used. A polycarboxylic ether-based superplasticizer (SP) of type Melflux® 2651 F is expected to improve the workability of the mortars and a viscosity modifying agent (VMA) of type Starvis® 3003 F should reduce segregation whenever present. Both admixtures are commercial products supplied by BASF.

2.2. Mix design

For the evaluation of flow properties using the optical set-up which is detailed in Subsection 2.4, mortars with a paste content of 35% were used. Two series of mortars with various amounts of SP and VMA were selected for this study: the M35 series, with a water-to-cement ratio (w/c) of 0.35, and the M45 series with a water-to-cement ratio of 0.45. For each series, the amount of superplasticizer varied from 0.25 to 1% by mass of cement, while the amount of the viscosity modifying agent varied from 0.1 to 0.5% by mass of cement. The detailed mix design is given in Table 2.

2.3. Mixing procedure

The mortars were prepared in a Hobart mixer with a capacity of 5 l with two grading speeds, as per DIN 196-1: the lower speed at 62 ±5 rpm and high speed at 125 ±10 rpm. The water used for the mixture was room-temperature tap water with pH of 6.9 – 7.1. Before water was added to the mixture, the dry components were premixed for 60 seconds to obtain a homogenous dry mass. After the water was added, the mixture was mixed for 60 seconds at the lowest speed, and then stopped for 30 seconds to scrape of the walls and the bottom of the bowl. After that, the mixing was resumed at high speed for 90 seconds. Immediately after mixing, the mortars were subjected to flow measurements.

2.4. Experimental set-up

2.4.1. General description

The set-up used to conduct the flow measurements consist of four main parts: the cone, the flow table, the lifting mechanism, and the fast camera as shown in Fig. 2. The flow measurements of the

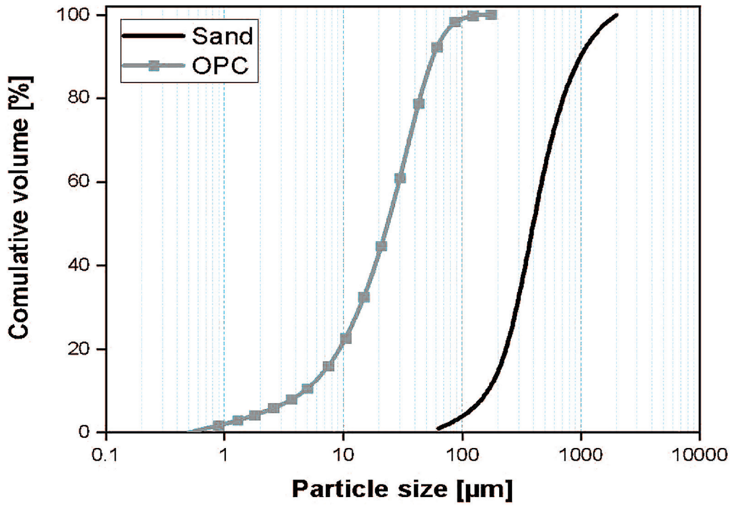

Fig. 1. Particle size distribution of the aggregate and Portland cement

Table 1

Chemical composition and physical properties of Portland cement (CEM I 42.5 N)

Chemical composition								Physical properties	
CaO (%)	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	MgO (%)	K_2O (%)	Na_2O (%)	SO_3 (%)	Blaine Value (cm^2/g)	Density (g/cm^3)
62.2	**20**	**4.8**	3.8	1.4	1	0.1	2.7	3632	3.4

Table 2

Mix design used for the evaluation of flow.

Sample ID	PC (%)	Aggregate (%)	SP (%)	VMA (%)	w/c
M35_SP0.25	35	65	0.25	–	0.35
M35_SP0.5			0.5	–	0.35
M35_SP0.75			0.75	–	0.35
M35_SP1			1	–	0.35
M45_SP0.25			0.25	–	0.45
M45_SP0.5			0.5	–	0.45
M45_SP0.75			0.75	–	0.45
M45_SP1			1	–	0.45
M35_SP1_VMA0.1			1	0.1	0.35
M35_SP1_VMA0.25			1	0.25	0.35
M35_SP1_VMA0.5			1	0.5	0.35
M45_SP1_VMA0.1			1	0.1	0.45
M45_SP1_VMA_0.25			1	0.25	0.45
M45_SP1_VMA0.5			1	0.5	0.45

mortar compositions were realized using the Hägermann cone according to DIN 1015-3.

The cone (1) has a height of 60 mm, with a bottom base diameter of 100 mm, and a top base diameter of 70 mm. The cone is placed in the center of the table (2).

The flow table consists of PMMA (Polymethyl-methacrylate) supported by four adjustable legs for leveling the table. The dimensions of the table are 490x490 mm with rulers drawn along both axes. In one corner of the table, a line of 100 mm in length has been drawn. This line is used as a reference for the conversion of pixel distances into centimeter distances.

In order to reduce human-induced error while performing the flow measurements, the cone is not lifted by a human operator as in the traditional method, but instead, a semi-automatic pneumatic lifting mechanism has been constructed for this purpose.

The Hägermann cone is supported by an arm (5) that is connected to a sliding plate (6) that moves along the y-axis between two end positions: the upper position and lower position (with no stop in between). The sliding plate is put into motion through a pneumatic piston (7) with 4 bar air pressure running through it. The movement of the sliding plate, which will move the cone eventually, is controlled manually by a lever (8) on the back side of the construction body. Once the cone has been filled with material, the lever is pushed allowing the pressurized air to run through the piston and lifting the cone smoothly and allowing for the material to flow out on the table.

The flow of the material is recorded using a high-speed camera (3) mounted above the cone. The camera captures images at 243 frames per second (fps) with a resolution of 1.57 Megapixel (MP). The camera is connected to a computer where the captured video frames are saved for further processing.

2.4.2. Technical background

The principle of the optical flow measurement is based on the recording of the flow process through a high-speed camera in connection with the semi-automatic cone-lifting device described in 2.4.1. This procedure not only allows

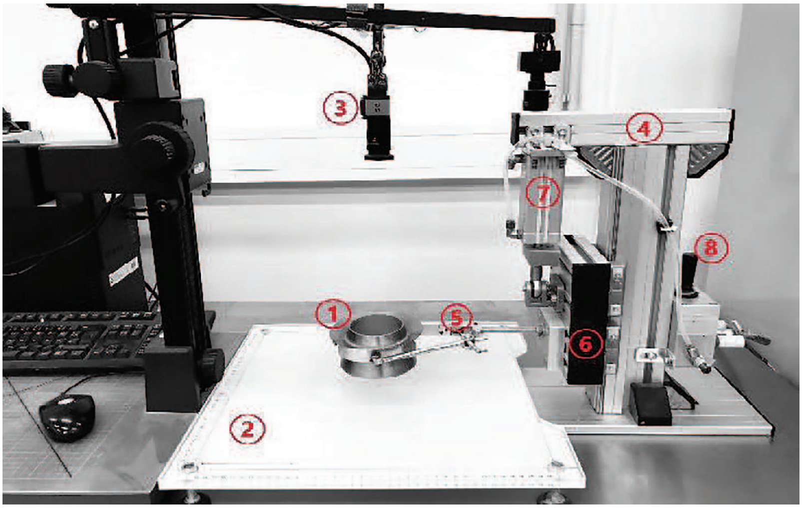

Fig. 2. The current set-up used for the flow measurements, where: 1 — Hägermann cone; *2* — Flow table; *3* — High-speed camera; *4* — Supporting mechanism; *5* — Connecting arm; *6* — Sliding plate; *7* — Piston; *8* — Lever

the determination of the final spread diameter but also the tracking of the change in the flow velocity during the flow which has significant advantages over the conventional determination of the flow diameter. Once the method has been established, reproducible flow measurements of fresh cement paste and mortars (especially for systems with low yield point and high flowability) can be obtained, stored and post-processed. This allows for revealing correlations of flow times (e.g. T25 or T30) and other rheological parameters such as yield point and viscosity. The recorded and archived flow videos can be used for subsequent analysis with improved methods and examined through other experimental techniques. For the accurate evaluation of rheological behavior, it is desirable to observe a flowing material under different shear rates. In order to obtain the flow parameters, the flow curve has to be extracted accurately by post-processing. The steps required for flow curve-extraction can be divided into:

a) removal of image noise from the frames of the video, and

b) detecting the progression of the front of the flowing material across all video frames.

The requirement for the image enhancement step (a) depends on the quality of the camera sensor. For the available recordings in this investigation, it proved to be sufficient to run a 3x3 Gaussian filter across all video frames. Before starting the geometrical evaluation of the flow (b), the center of the Hägermann's cone in the video must be detected. This is done manually for the first frame by clicking on a pixel belonging to the cone. From the color value, the whole cone and its center can be identified. The actual dimension (in pixels) of a reference marker on the flow table is also determined. This relation comprises a factor F_L between pixel count and linear dimension on the flow table which can be used for the following evaluation steps. In addition, all pixel values of the first frame (reference) of the video are subtracted from all subsequent frames in order to trace the image differences along the time axis. For numerical evaluation of the flow, the subsequent frames of the video are monitored along four or five differently oriented lines centered in the middle of the cone area and projected out to the borders of the flow table. At the beginning of the analysis, these lines are initialized with zero values. As the flow progresses along the frames, the brightness values of the pixels corresponding to the tracked lines change. Any change indicates the progress of the front of the flowing material. During the analysis, the measured time and distance values for the lines are stored in a file for subsequent processing. Based on these values, the shape of the flow area and its mean diameter value along the time axis can be calculated and visualized. The software used for the determination of the flow curves is a combination of C++ and Python programs developed in our group (using the freely available graphics library OpenCV).

3. Results and discussions

3.1. Effect of superplasticizer (SP)

The flow behavior of the M35 and M45 series with w/c ratios of 0.35 and 0.45, respectively, containing different amounts of superplasticizer are given in Fig. 3. The control samples of both series (the samples not containing any amount of SP or VMA) do not flow and are not included in the graphs.

For the M35 series (Fig. 3a), when an amount of 0.25% SP was added the mortar does not flow (horizontal line). The water content in this series was too low for low amounts of SP to initiate the flow of the mortar. The addition of 0.25% of SP did

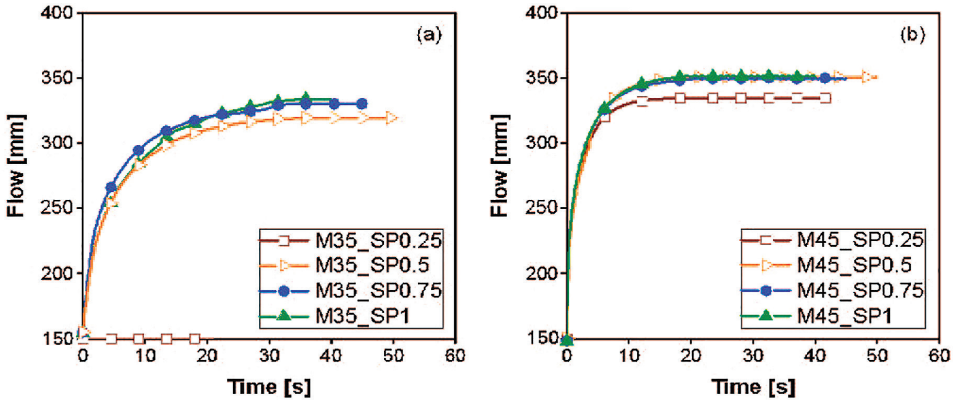

Fig. 3. The flow curves of M35 series (a), and M45 series (b), using software-generated data from the recorded video frames

however change the consistency of the sample slightly. An improvement was noticed when filling the cone to conduct the flow measurement and this suggests a decrease in viscosity and most probably in yield stress. This is in agreement with other studies that have investigated the influence of SP on cementitious materials and concluded that the addition of SP even in small amounts will influence the rheological properties of the material [17]–[19]. When the amount of SP was increased to 0.5%, a drastic improvement in the workability of the mortar was noticed. The final flow of the material reached a value of about 32 cm. After further increase of the SP amount, the flow behavior of the mortars changed only slightly. When an amount of 0.75% of SP was used for the series, the final flow reached a value of 33.5 cm, almost identical to the sample containing 1% of SP as can be seen in the corresponding graph.

In contrast to the M35 series, all the samples in M45 containing SP (Fig. 3b) exhibited a pronounced flow. Even the mortar containing the lowest amount of SP (0.25%) of this series reached a slightly higher flow value than the mortar containing the highest SP amount in the M35 series. This is obviously related to the higher amount of water used in this case. The further increase of the amount of SP to 0.5% resulted in higher flow values as expected, however, when further increasing the amount of SP, the flow behavior of the mortars was practically identical. This means, that above the 0.5% value, the SP does not further increase the flow value of the mortar. The 0.5% value of SP can thus be regarded as the saturation dosage above which no influence on the rheological behavior of the material is to be expected, as already stated by Perrot et al. [20].

For all the samples of the M45 series, at first a high flow speed of the mortars can be seen immediately after cone lifting, then the curves reach the final flow values (stop flowing) quite fast. This is not the case for the samples of the M35 series, where the time required to reach the final flow value is considerably higher. This can be explained by the differences in the plastic viscosity of the two series. Since the flow time is a direct indication of the plastic viscosity, we can say with certainty that the mortars of the M35 series have a higher plastic viscosity than the mortars of the M45 series, thus the different appearance of the flow curves.

3.2. Effect of viscosity modifying agent (VMA)

In Fig. 4, the flow behavior of the two series (M35 and M45) with samples containing 0.1%, 0.25%, and 0.5% of VMA is summarized. For all the samples in both series, the amount of SP was kept constant at 1%.

The reason why the amount of SP was kept constant at 1% when VMA is incorporated into the mix design is related to the presence of bleeding and segregation in both series. The addition of VMA into the mix design enhances the resistance of the material to bleeding and segregation. This is especially the case for mixtures containing a high amount of water and water-reducing admixtures such as SP, as already reported by other investigators [21]–[23].

For the M35 series (Fig. 4a), where only mild segregation and bleeding was found, the incorporation of VMA had a drastic effect on the flow behavior of the mortars. The final flow value of the sample containing the lowest amount of VMA was reduced for more than 25% compared to the initial value (the sample with 1% SP and no VMA). The final flow value of the mortars decreased even more when the amount of VMA was increased further. The sample containing the highest amount of the viscosity modifying agent experienced a decrease in the final flow value of more than 40% compared to the initial value. This effect is related to the increased plastic viscosity of the material, since there is a direct relation of the increased viscosity with the dosage of viscosity modifying agent [24]. After the incorporation of the VMA for the M35 series, bleeding and segregation were completely absent. This means that the dosages of 0.25% and 0.5% of VMA for this series are too high and unnecessary. The highest amount of VMA could even have an adverse effect on the mortar by potentially inflicting stagnation phenomena.

In contrast to the M35 series, bleeding and segregation were quite prominent in the M45 series (Fig. 4b). The addition of 0.1% and 0.25% VMA didn't appear to have much influence on the final flow of the mortars. There was however a slight increase in the flow time to reach the final flow value, but bleeding and segregation were still detectable in the mortars, although not prominent anymore. For this series, a considerable change in the flow behavior was noticed for the highest dosage of VMA. Also, in this case, there was a notable decrease in the final flow values and bleeding and segregation were not present.

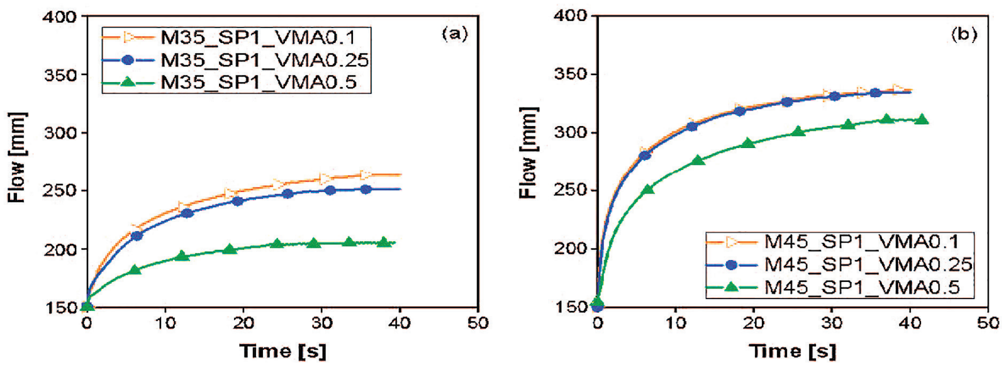

Fig. 4. The flow curves of the M35 series (a) and M45 series (b) containing different amounts of VMA and 1% of SP.

3.3. Repeatability of the optical method

As already discussed earlier, the major problem with the empirical methods is that they are operator sensitive [11]. Our optical set-up was designed to reduce or even eliminate human-induced errors. An important part in testing the performance of the optical method was the repeatability of the results. Examples for flow-curve reproducibility using our optical method are shown in Fig. 5 for two different compositions.

The first sample (Sample 1), contained a higher amount of water (w/c =0.42) and a small amount of SP (0.1%), while the second sample (Sample 2), contained a lower amount of water, but a higher amount of SP. For each sample, three consecutive measurements were conducted. All measurements were conducted using the same conditions. In a recent study, Raja et al. [25] reported a standard deviation for their final flow values around or below 5.5 mm. In our study, however, the standard deviation appears to be improved, with the values of the flow remaining under 2 mm at each point in time. The measured standard deviation in both studies is caused predominantly by variations in the raw materials and their mixtures and is most probably even less related to the method itself, proving thus the high potential of the optical method and its effectiveness in reducing human-induced errors.

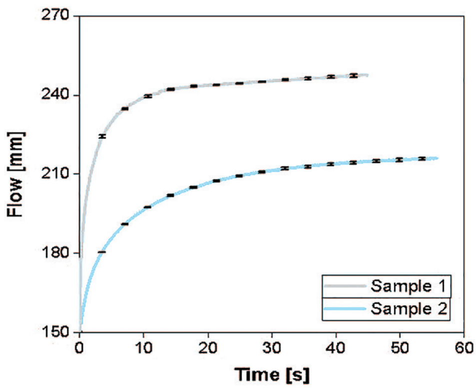

Fig. 5. The standard deviation for two different samples containing different amounts of water and superplasticizer

4. Conclusions

A new optical method in combination with a semi-automatic pneumatic lifting device was introduced to characterize the flow behavior of mortars containing different amounts of superplasticizer and viscosity modifying admixtures. From the work carried out, the following conclusions for the method can be drawn:

– The human-induced error is greatly reduced using our set-up due to the automatization of the cone-lifting.

– The flow curves of the evaluated mortars, generated after processing the raw video frames, allow us to have a clear indication of the flow behavior at each point from the very first second until the material stops flowing.

– By storing the raw data (the videos) for the flow, we were able to create a data stock that can be used for future work.

The software developed for flow evaluation allows easy and accurate processing of the raw data. As for the influence of the admixtures on the mortars used for this study, we can conclude that our findings are in good agreement with previous investigations on the subject. The incorporation of the superplasticizer considerably improved the workability of the mortars in both series. Above a specific amount, however, its effect is obsolete due to the saturation of the material with SP. In both series, the presence of SP caused bleeding and segregation, with the phenomena being more prominent in the case where a higher amount of water was used. The incorporation of the viscosity modifying agent into the mix design solved the issue in both series, but care must be taken to properly assign the correct amount as the VMA has a drastic effect on the final flow when higher amounts were used.

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Получена редакцией 20.03.2022; получена после рецензии 27.06.2022; принята к печати 10.09.2022.
Received by the editors 20.03.2022; received after the review 27.06.2022; accepted for printing 10.09.2022.