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ORIGINAL ARTICLE

Evaluation of flowability of cementitious self-leveling pastes by varying the superplasticizer and viscosity modifying admixture

Avaliação da trabalhabilidade de pastas cimentícias autonivelantes através da variação do teor dos aditivos superplastificante e modificador de viscosidade

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(i)

Abstract: To meet the self-leveling pastes' performance requirements, its flowability is linked to the absence of segregation or exudation, which makes a high cohesion between the components mandatory. Therefore, the ideal dosage for these materials is an essential topic to provide such specific properties. To evaluate the composition effect on fresh state, a method was proposed to quantify self-leveling pastes' uniformity through the analysis of the spread pastes' diameter and borders. Thus, different dosages were tested varying some input contents. The uniformity was quantified with the development of indexes such as Perimeter Ratio (PR) and Input Relation (IR). It was concluded that lower spread diameters incurred in higher uniformity index. Furthermore, it was observed that to achieve an ideal dosage for pastes, there is a tendency of direct correlation between the uniformity index and the water/powder ratio (w/p) and superplasticizer (SP) content and an inverse one between it and viscosity modifying admixture (VMA).

Keywords: self-leveling pastes, cementitious paste, dosage, pastes' uniformity index.

Resumo: Para cumprir os requisitos de desempenho das pastas autonivelantes, a sua fluidez está ligada à ausência de segregação ou exsudação, o que torna obrigatória uma elevada coesão entre os componentes. Assim, a dosagem ideal destes materiais é um tópico essencial para proporcionar estas propriedades específicas. Para avaliar o efeito da composição no estado fresco, foi proposto um método para quantificar a uniformidade das pastas autonivelantes através da análise do diâmetro e das bordas das pastas espalhadas. Assim, foram testadas diferentes dosagens variando alguns teores de insumos. A uniformidade foi quantificada com o desenvolvimento de índices como a Razão de Perímetro (RP) e a Relação de Insumos (RI). Concluiu-se que menores diâmetros de espalhamento implicam em maior índice de uniformidade. Além disso, observou-se que, para se obter uma dosagem ideal para as pastas, há uma tendência de correlação direta entre o índice de uniformidade e a relação água/pó (a/p) e o teor de superplastificante (SP) e uma correlação inversa entre este e o aditivo modificador de viscosidade (VMA).

Palavras-chave: pastas autonivelantes, pasta cimentícia, dosagem, índice de uniformidade das pastas.

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1 INTRODUCTION

The technology of self-leveling cement materials is studied as an alternative to the traditional constructive methodology. This technique achieves better results regarding the economical expenses, execution speed and imperfection surfaces when compared to the traditional solutions [1]–[4]. Additionally, it allows achieving feasible application of specific composites, which present low workability, such as high-performance concretes and materials with fibers addition [5]–[10]. To be classified as a self-leveling material, the pastes must present specific properties on its fresh state, most importantly high flowability and cohesion [7], [11], [12].

In order to guarantee the properties and performance required for this material, the use of additives, such as superplasticizer (SP) [13]–[17] and viscosity modifying admixture (VMA) [11], [18], [19], is mandatory. SP acts as a chemical dispersant promoting electrostatic repulsion between particles, being influenced by the composition and pH of the components as well as by the additive's composition and structure chain [20]. VMA is used to balance the high workability of the material, keeping its ability to flow without segregation. This additive guarantees a higher cohesion to the pastes by modifying the chemical structure of the hydration products [21], [22].

Besides admixtures, silica fume is one of many additions that can be used on self-leveling pastes [23]–[25]. The silica fume particles are smaller than cement particles, therefore its addition to the mixture improves its particle packing and cohesion [24], [26]. Moreover, as a mineral addition, the silica fume combines with calcium hydroxide, which densifies the paste's microstructure, leading to the reduction of its porosity [26], [27]. This improvement in the microstructure of the paste allows the reduction of cement content in the mixture, which leads to a minor shrinkage [28], [29].

Martins [30] and Wu et al. [31] demonstrated that in the absence of a correct and precise dosage, the balance of the effects caused by the chemical interaction of both additives may not be achieved. This could lead to the occurrence of segregation, by separating the liquid and solid phases, and exudation, marked by migration of water through the cement paste [14], [32].

By studying the ideal dosage for self-leveling pastes, Martins [30] has tested mixtures by varying additive contents and used visual analysis to evaluate its consequences on pastes' fresh state behavior. In this work, the geometry of pastes spread through an adapted Ford Viscosity Cup16 (AFVC) was analyzed. The visual analysis methodology proposed by Martins [30] consisted of evaluating the appearance of the border in terms of uniformity. For this, as shown in Figure 1, the spreads were qualitatively classified as uniform and irregular.



Figure 1. Visual analysis of the pastes' borders. Legend: a) irregular border; b) uniform border. Reference: Martins [30].

It is important to mention that the qualitative visual analysis tends to generate subjective results because it is influenced by each observer's sight. This study aims to contribute to filling the lack of an evaluation method for self-leveling pastes flow behavior by quantifying the uniformity of the pastes' borders.

2 MATERIALS AND EXPERIMENTAL PROGRAM

To achieve the study aim, eight compositions of cement pastes were dosed and submitted to a spreading test for further analysis of borders' uniformity using the methodology proposed in the current research.

2.1 Pastes' composition

Based on the visual analysis methodology of borders' uniformity, Martins [30] has established criteria to select the pastes that presented satisfactory behavior. After testing several compositions, the author achieved four ideal pastes' mixtures, as presented in Table 1. The admixture dosages were adopted concerning the cement mass.

Mixture	Cement (g)	SF (g)	w/p	SP (%)	VMA (%)	Diameter (mm)
Martins 1	460	40	0.70	0.50	0.70	250
Martins 2	465	35	0.75	0.50	1.10	250
Martins 3	470	30	0.70	0.50	0.70	268
Martins 4	475	25	0.80	0.50	0.70	288

Table 1. Description of pastes composition proposed by Martins [30].

Legend: SF - silica fume; w/p - water/powder ratio; SP - superplasticizer; VMA - viscosity modifying admixture.

For the present study, the analysis of 8 mixtures was proposed based on Martins' dosages. The highest cement (475 g) and the lowest silica fume (25 g) content were adopted to vary the portions of VMA and water (Table 2) in order to evaluate their influence on the behavior of pastes regarding its consistency index and uniformity. Knowing that higher amounts of silica fume can affect the admixtures' performance, a smaller silica fume content was adopted in this research. The mixture dosages studied in this campaign are presented in Table 2.

Commercial Portland cement CPV-ARI-RS (with clinker content similar to OPC), polyethylene glycol superplasticizer and sulphur-based viscosity modifying admixture were used for the mixtures' dosages. The SP content was set at 0.4 (B mixtures), and the water/powder ratio (w/p) was varied in three ranges, which were: 0.4; 0.5; and 0.6, with VMA adjustment required. To define the content of each input, it was aimed to reach a diameter range between 250 and 300 mm. In the sequence, the amount of SP was exceeded at 0.9 of the cement mass (C mixtures) to verify the interaction of the three variables and evaluation of the dosage limits.

Mixtures	Cement (g)	SF (g)	w/p	SP (%)	VMA (%)
B1	475.00	25.00	0.40	0.40	0.60
B2			0.40	0.40	1.00
B3			0.50	0.40	0.80
B4			0.50	0.40	1.00
B5			0.60	0.40	0.60
C1			0.40	0.90	0.60
C2			0.50	0.90	0.80
C3			0.60	0.90	1.00

Table 2. Mixtures dosages.

Legend: SF - silica fume; w/p - water/powder ratio; SP - superplasticizer; VMA - viscosity modifying admixture.

2.2 Pastes' mixing procedure

All pastes were obtained by using the same mixing procedure, maintaining the input sequence, mixing duration and speed, as proposed in Martins [30]. The samples were mixed at low speed using a laboratory vertical mixer, adding the water amount in two equal values and mixing the additives as final inputs.

Firstly, all cement and silica fume content is mixed with 50% of the water amount, throughout 1 minute. After that, the rest of the water content is added and mixed for the same duration followed by the addition of the SP for 40 seconds. Finally, VMA is added and mixed with the other materials for more than 40 seconds.

2.3 Flow analysis method

The Ford Viscosity Cup (FVC) (Figure 2a) is an equipment used to evaluate the viscosity of Newtonian or near-Newtonian paints, varnishes, and lacquers [33]. In the current paper, the cup's geometry was adapted in order to characterize self-leveling pastes. Thus, it was used a cylinder of 60 mm diameter and 90 mm height with a 5 mm diameter orifice on its bottom (Figure 2b). The main differences between this equipment and the FVC are the cups' volume and geometry, as the cylinder has a flat bottom instead of a funnel-shaped one, as in the FVC. For the flow analysis, this cylinder was suspended by a rod at a height of 150 mm over a container (Figure 3a), where the pastes were spread (Figure 3b).



Figure 2. Traditional and adapted Ford viscosity cups' geometry. Legend: a) Traditional Ford viscosity cups' geometry; b) adapted Ford viscosity cups' geometry. Reference: Martins [30].



Figure 3. Adapted Ford viscosity cup test. Legend: a) Adapted Ford viscosity cups' geometry suspended by a rod; b) Test's resulting paste spread. Reference: Martins [30].

The experiment consists of filling the cylinder with the paste (254,340 mm³) and draining it through the opening at the recipient's bottom. Once the cylinder is empty, the paste's average diameter is obtained by two orthogonal measures on the surface. The larger the diameter achieved, the higher the paste's flowability.

In order to obtain the pastes' perimeter ratio, images of spreading were analyzed on the AutoCAD program. For this, the pictures taken after the Spanish Cylinder test were plotted on the software and transformed into draws by outlining its perimeter with polylines. Using the same image for each sample, an elliptical shape was designed around the material, which was considered the ideal spread representing a uniform spread border. Then, the perimeter's ratio (PR) was obtained by calculating the ideal/real values as shown in Equation 1:

$$PR (\%) = \frac{Ideal \ perimeter}{Real \ perimeter} \times 100$$

(1)

Figure 4 presents an example of the resulting image obtained by comparing the area from both real and theoretical perimeters, where the grey area represents overrun paste and the red one, the gap between both borders. The ideal pastes should not present any overrun or gap, whereas the pastes with more grey or red areas are considered unsuitable.



Figure 4. Comparison between ideal and real spreading borders

3 RESULTS AND DISCUSSION

The results obtained in the experimental tests previously described are presented in this section. Concerning the diameter, which represents the flowability of the pastes, the water/powder ratio and the superplasticizer contents increased flowability as expected. On the other hand, the increase of viscosity modifying admixture reduced it (Table 3). These results confirm the findings shown by previous studies [30], [31]. By comparing B and C mixture diameters, it was possible to observe that the increase of SP content leads to higher values.

Analyzing VMA influence at pastes flow, it was noticed a decrease in spreading diameters as the additives' content was increased. The main cause of these lower values is the effect of increasing the viscosity of the mixture promoted by this additive, as shown by other authors [21], [22]. It is noteworthy that the viscosity property establishes the fluids' flow resistance, influencing the resulting diameter of the mixtures. This behavior was observed in both mixtures B and C, except for mixtures B1 and B2. It was inferred that the reason why VMA couldn't act with the same performance as it did on the other pastes was because these pastes were prepared with the same w/p ratio and SP content.

Regarding flowability, B mixtures achieve flows between 200 to 320 mm, and all of the C mixtures overrated 300 mm. Martins [30] determined the ideal diameter to be comprehend between 250 to 300 mm. Therefore, only the mixture B3. However, the diameter must always be analyzed together with other parameters such as borders' uniformity index and exudation, for example, as highlighted by that same author.

The uniformity index was correlated with the perimeter ratio (PR) results. Table 3 shows that pastes with higher flowability have lower PR values. This phenomenon was evident in all mixtures which achieved diameters higher than 300 mm. It can be explained by the exudation promoted by the high content of w/p and SP, as noted in the pastes' images. These results confirm the importance of considering not only the diameter, but more parameters correlated to flowability.

Based on the results shown in Table 3, the influence of each component was evaluated separately. However, the correlation between the inputs and the PR resulted in low correlation coefficient (R^2) values, situated in a range between 0,11 and 0,49, which indicates that the individual action of each variable does not fully explain the uniformity index. Therefore, it was noticed that is the interaction between w/p, SP and VMA content that influences the PR values. According to the previous explanation, water and SP contribute to flowability while VMA acts in opposite directions. In order to analyze the actuation mechanism of those three inputs, it was proposed the relation shown by Equation 2.

Inputs relation (IR) =
$$\frac{(w/p \times SP)}{VMA}$$
 (2)

Once the IR was calculated for the mixtures, it was obtained the correlation between those results and PR by the analysis indicated in Figure 5. Through a second-degree polynomial equation trend, the value of R^2 was obtained, which translates to a correlation of approximately 81% between the variables IR and PR. The R^2 value shows a tendency of action directly proportional to the PR by the components' w/p and SP, and an inversely one regarding the VMA content. Moreover, the chosen trend curve (parabolic) represents the additives' performance accurately because there is a saturation point presented by such materials.

Table 3. PR and diameter spreading of the mixtures.

Mixtures	PR (%)	D (mm)	Non-scale images
B1	97	200.0	
B2	99	205.0	
B3	100	252.5	
B4	99	240.0	
B5	94	320.0	
C1	91	330.0	
C2	89	337.0	
C3	92	340.0	

Legend: Gap Overrun

PR - perimeter ratio; D - diameter.

It is shown in Figure 5 an increase of PR values whereas IR suffers a reduction. Therefore, it can be assumed that to obtain a higher uniformity index for the pastes, represented by PR, it is required to minimize water and superplasticizer and/or to raise viscosity modifying admixture contents.

As previously presented, Martins [30] suggested an ideal diameter range of 250 to 300 mm for further application. Following this finding, the results indicated that diameters higher than 300 mm promote lower uniformity index. However, all the pastes with diameters lower than 300 mm achieved satisfactory PR values. In Figure 5 the results were organized in different color patterns based on diameter ranges, as shown in the legend. Hence, it was noticed that the diameter analysis was insufficient to reflect the pastes' quality. For example, there is a tendency that diameters lower than 250 mm to have higher PR values. On the contrary, for the 260 to 300 mm range, like Martins' ideal range, some pastes had unsuitable PR values.



Figure 5. Correlation between PR and IR results

A classification was developed regarding the PR results by comparing them with the draws plotted in the images of Table 3, produced from pictures of the scattered pastes. It was proposed three classes according to the borders' uniformity analysis provided by visual evaluation methodology, developed by Martins [30]. The Ideal class comprehends PR values higher than 98.9%, the Admissible one between 95.0 and 99.0%, and finally the Unsuitable is for values lower than 94.9%, as shown in Figure 5.

After defining the classification, it was observed that ideal IR values are contained in the range of 0.15 to 0.35, limited in the Figure 5 by dashed lines. Besides that, as known that IR values are influenced by mixtures components, it was assumed that this range is suitable for the pastes analyzed in the current paper.

4 CONCLUSIONS

The importance of dosage combined with the uniformity of self-leveling cement pastes' borders was studied in this paper. It was possible to conclude the interaction between the superplasticizer (SP), viscosity modifying admixture (VMA) content together with the water to powder ratio (w/p) in pastes' performance.

The analyses of the pastes' fresh state have shown that is the interaction of the additives that modifies the material properties, not simply the sum of each input's content separately. Therefore, it was proposed a perimeter ratio (PR) equation that enables the evaluation of the consequential properties of pastes' dosage, indicating higher values to more uniform spreading borders. It was noticed that the combination of additives' contents influences the mixture flow and uniformity, with a trend of increasing the flowability by adding SP whereas the opposite effect is caused by VMA addition, which contributes to the material's cohesion.

The additives' mechanism could be evaluated by the proposed inputs relation (IR), which also correlates with PR values presenting a tendency of a directly proportional action by the components' w/p and SP, and inversely one by VMA. All the pastes with diameters lower than 300 mm achieved satisfactory PR values while the ones that overrated the mark of 300 mm presented a lower uniformity index. Furthermore, a classification was developed regarding the PR results: Ideal, with PR higher than 98.9%; Admissible between 95 and 99%; and Unsuitable for values lower than

94.9%. Consequently, the results indicated that, for the materials used in this study, ideal IR values are contained in the range of 0.15 to 0.35. It was concluded that the Ideal PR represents the best conditions for uniformity which is marked by borders with cohesion and without exudation outcomes. The Admissible PR tolerates lower level of exudation, and the Unsuitable one shows an unsatisfactory level of uniformity.

This paper aimed to propose a new methodology to quantify self-leveling pastes' uniformity through the analysis of the spread pastes' diameter and borders. It was achieved by this new method, which enables classifying the pastes borders and determineing the best pastes dosages.

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