JACEK GOŁASZEWSKI, GRZEGORZ CYGAN, MICHAŁ DREWNIOK

THE INFLUENCE OF COMPOSITION ON THIXOTROPIC BEHAVIOUR OF CEMENT MORTAR

Abstract
This paper presents methods of measurement and results of rheological properties including the thixotropic stiffening of designed cement mortars. The aim of the study is to determine the effect of the type of cement, w/c ratio, and chemical admixtures on the selected rheological properties of mortars. It will allow a better understanding of the nature of previously observed decreasing formwork pressure when using self-compacting concrete.

Keywords: SCC, thixotropic stiffening, rheological properties of mortars, static yield stress

Streszczenie
W artykule przedstawiono metody badawcze oraz wyniki właściwości reologicznych, w tym tiksotropowego sztywnienia zapraw cementowych. Celem badań było określenie wpływu rodzaju cementu, stosunku w/c, rodzaju superplastyfikatorów na wybrane właściwości reologiczne zaprojektowanych zapraw cementowych. Wyniki badań pozwolą lepiej rozumieć i opisać charakter zaobserwowanego już wcześniej charakteru zmniejszania się parcia mieszanki samozagęszczalnej na deskowanie.

Słowa kluczowe: BSZ, tiksotropowe sztywnienie zapraw, właściwości reologiczne zapraw, sta-tyczna granica płynięcia

1. Introduction

Concrete or mortar mixtures of cement paste and aggregate, are one of the most commonly used materials in modern construction. One of the main factors determining the properties of concrete, is the quality of the ingredients used in its production. The necessity for determining the properties of concrete, based on the properties of mortar for concrete is essential for time saving. The author [4] also demonstrates predicted directional and size changes in the rheological properties of concrete, in other words, the durability of concrete determined, inter alia, ongoing changes in the mixture under load during mixing, transporting, placing, casting and finishing (Fig. 11a). These rheological changes are described in detail in [1, 3, 4, 8–10, 12–14].

To better understand the phenomena occurring in the concrete, a thorough understanding of the rheological properties of concrete in conjunction with the changes in the structure are necessary. They include the little known thixotropic stiffening effect that intensifies at rest and persists after re-mixing.

Cement paste is often characterized by thixotropic behaviour – “fluid memory”, which is assumed to be a reversible process. It represents the body (liquid) which, being a long time at rest has “solidified” and increased its viscosity. Applying shear (supplying energy), over time, the apparent viscosity begins to decrease due to progressive destruction of the structure through shear thinning. The rate of disintegration of the bonds depends on the amount that can be destructed. After shearing, depending on the number of bonds, the structure begins to rebuild [9]. The result is, unlike a Newtonian fluid, which for increasing and decreasing,
shear rate can form hysteresis loop (Fig. 1). Different types of thixotropic liquid along with their detailed characterization provide, inter alia [7, 11, 13].

2. Research significance

Determination of the exact moment of structural damage to the mixture (depending on the composition and other technological factors) due to the effect of shear thinning, by being aware of changes in the flow curves (Fig. 1b), with the determination of static yield stress (Fig. 1c) will allow for a better understanding of the nature of previously observed decreasing formwork pressure using self compacting concrete [14].

The aim of the study is to determine the effect of types of cement, w/c ratio, and chemical admixtures on the rheological properties of selected mortars.

The results are essential for the design of self-compacting concrete, and will be used to develop a dependence which will allow for the calculation of self compacting formwork pressure.

3. Experimental investigation

The rheological parameters of mortars were determined by using a rheometer Viskomat NT described in detail in [13]. Studies [1, 4, 8, 13] show that the rheological behavior of fresh mortar can be sufficiently described by the Bingham model according to equation:

\[ \tau = \tau_o + \eta \cdot \dot{\gamma} \]  

where:

\( \tau \) (Pa) – the shear stress at shear rate,  
\( \dot{\gamma} \) (1/s), \( \tau_o \) (Pa), \( \eta \) (Pas) – the yield stress and plastic viscosity, respectively.

In rheometry the Bingham model equation (1) is used in its conventional form:

\[ M = g + N \cdot h \]  

where:

\( g \) (Nmm), \( h \) (Nmms) – parameters corresponding to Bingham’s yield stress and plastic viscosity.

The rotational speed for Viskomat NT and the measure of time is shown in Fig. 3. In parallel to the rheometric tests, technical mortar tests, are also performed according to PN-EN 1015-3. The tests were determined after 40 minutes of mixing and a further 40 minutes of resting. Then in the 80th minute, re-mixing.

The proposed procedure allow the following properties to be measured: the static yield stress after resting (1st and 2nd), the static yield stress at a constant speed of 1 rpm, the nature of the hysteresis loop, the initial yield stress and plastic viscosity with increasing shear rate in a range of speed from 1 to 30 rpm and the dynamic yield stress and plastic viscosity decreasing shear rate.
4. Material properties and composition of mixtures

For investigation was used three types of cement CEM I 42.5 R, CEM III/A 42.5N-HSR/NA oraz CEM V/A (S-V) 32.5 R-LH, two kinds of superplasticizers based on polycarboxylate ether and sand from Niedomice.

The composition of mortars are shown in Table 1.

<table>
<thead>
<tr>
<th>Symbol/Mixture</th>
<th>Component/Składnik</th>
<th>[unit]</th>
<th>Z1</th>
<th>Z2</th>
<th>Z3</th>
<th>Z4</th>
<th>Z5</th>
<th>Z6</th>
<th>Z7</th>
<th>Z8</th>
<th>Z9</th>
<th>Z10</th>
<th>Z11</th>
<th>Z12</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 42.5 R</td>
<td>[kg]</td>
<td>775</td>
<td>688</td>
<td>785</td>
<td>691</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>CEM III/A 42.5N-HSR/NA</td>
<td>[kg]</td>
<td></td>
<td></td>
<td></td>
<td>776</td>
<td>680</td>
<td>784</td>
<td>682</td>
<td></td>
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<td></td>
<td></td>
<td></td>
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<tr>
<td>CEM V/A (S-V) 32.5 R-LH</td>
<td>[kg]</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>751</td>
<td>662</td>
<td>757</td>
<td>668</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>[kg]</td>
<td>232</td>
<td>275</td>
<td>236</td>
<td>276</td>
<td>233</td>
<td>272</td>
<td>235</td>
<td>273</td>
<td>225</td>
<td>264</td>
<td>227</td>
<td>267</td>
<td></td>
</tr>
<tr>
<td>w/c ratio</td>
<td>–</td>
<td>0.30</td>
<td>0.40</td>
<td>0.30</td>
<td>0.40</td>
<td>0.30</td>
<td>0.40</td>
<td>0.30</td>
<td>0.40</td>
<td>0.30</td>
<td>0.40</td>
<td>0.30</td>
<td>0.40</td>
<td></td>
</tr>
<tr>
<td>SP PE2 [% m.c]</td>
<td>[kg]</td>
<td>2.00</td>
<td>0.75</td>
<td>1.00</td>
<td>0.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2.00</td>
<td>0.75</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SP PE1 [% m.c]</td>
<td>[kg]</td>
<td>3.00</td>
<td>1.00</td>
<td>1.75</td>
<td>0.75</td>
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<td></td>
</tr>
<tr>
<td>Sand 0–2</td>
<td>[kg]</td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tbody>
</table>

Table 1

The composition of fresh mortar – components for 1 m³ mortar

Fig. 2. The rheological test procedure for Viskomat NT

The amount of PE was chosen in such a way that the mortar spreading was 25–30 cm. When this was not possible, a maximum amount of admixture, which would not cause mortar segregation was used.
5. Experimental results and discussion

5.1. Initial mortar viscosity and initial yield stress

Research showed (Fig. 5), that after resting, initial viscosity was increasing and after re-mixing decreasing. In case of PE 2 with CEM I and CEM III the viscosity was clearly increasing than in case of PE 1, unlike in case of CEM V. After re-mixing value of the viscosity in mortars with CEM I and CEM III were similar, slightly higher than after mixing. The highest initial viscosity increase occurs in case CEM V. After re-mixing its value was lower than after mixing.

![Fig. 3. Influence of type of cement and PE on initial mortar viscosity and yield stress (w/c = 0.3)](image)

After resting, initial yield stress slightly increased. In case of PE 2 with CEM I and CEM III clearly decreased than in case of PE 1, unlike in CEM V. For CEM V initial yield stress for appropriate mortars, were similar, apparently decreasing in the case of PE1. The highest yield stress increased occurs in CEM I with PE2 and after re-mixing its value was slightly greater than its value after mixing.

![Fig. 4 Influence of type of cement and PE on initial mortar viscosity and yield stress (w/c = 0.4)](image)

After resting, the initial viscosity slightly increased and slightly decreased after re-mixing (Fig. 6). Its value was significantly smaller than in the case of mortar with a ratio of w/c = 0.3. In case of PE 2 with CEM I and CEM III its value decreased compared to PE 1, unlike in case of CEM V. After re-mixing, the value of the viscosity in mortars were similar, slightly lower than after mixing. The highest viscosity increase occurred in the case of CEM I with PE2 and was lower after re-mixing.
Initial yield stress was highest in case of CEM V (Fig. 6). After resting, mortars initial yield stress increased for CEM III and CEM V and decreased after re-mixing. In case of CEM I after resting and re-mixing there was similar value of yield stress, increased in case of PE 2.

The highest increase was in case of CEM V. For PE 2 with CEM I and CEM V initial yield stress clearly increased compared to PE 1, unlike in case of CEM III and CEM V, after resting. For CEM V, the initial yield stress for appropriate mortars were similar, but apparently decreased in the case of PE 1. The highest value of yield stress increased in case of CEM I with PE 2 and after re-mixing its value was slightly greater than its value after mixing.

5.2. Influence of type of cement and PE on mortar flowability

![Graph showing influence of type of cement and PE on mortar flowability](image)

Fig. 5. Influence of type of cement and PE on mortar flowability: on the left ratio $w/c = 0.3$, on the right ratio $w/c = 0.4$

After resting, the flowability of mortars with a ratio of $w/c = 0.3$, slightly decreased and slightly increased after re-mixing, and was again higher after mixing (Fig. 5).

The exception was in case of CEM I with PE1 and CEM I with PE2. For the first, after resting, flowability increased and after re-mixing it was again higher. For the second, it slightly its flowability, even after re-mixing. In case of CEM V, reduction of flowability after resting was greatest, but after re-mixing, as in the case of PE2 flowability was at the same level than after mixing.

After resting, the flowability of mortars with a ratio of $w/c = 0.4$ slightly decreased and again slightly increased after re-mixing, slightly lower than after mixing (Fig. 5).

Exception was in case of CEM I with PE1, in which flowability after resting increased and after re-mixing it was lower than after mixing. In case of CEM V with PE1, the reduction of flowability after resting was greatest.

5.3. Influence of type of cement and PE on the change 1st static yield stress

As the nature of the 2nd static yield stress was similar to the 1st static yield stress, this paper presents only influence of type of cement and PE on the change 1st static yield stress.

After resting, 1st static yield stress of mortars with ratio of $w/c = 0.3$, increased and decreased after re-mixing (Fig. 8). The largest increase was in case of CEM I with PE2 and CEM V with PE1 and PE2. For PE1 increase was the highest. After resting, the 1st static yield
stress of mortars with a ratio of w/c = 0.4, increased and decreased after re-mixing (Fig. 6). The largest increase was in case of CEM I with PE2, CEM III with PE1 and CEM V with PE1 and PE2. For PE1 with CEM V increase was the highest. After re-mixing in case of CEM V 1st static yield stress decreased, in case of PE2 it was lowest.

Fig. 6. Influence of type of cement and PE on the change 1st static yield stress: on the left ratio w/c = 0.3, on the right ratio w/c = 0.4

6. Conclusions

The study indicates that the thixotropic stiffening of mortars are a very complex phenomenon that requires in-depth knowledge.

Therefore, research in this direction is necessary and should be dealt with using the multi-measurement system. The measurement method of thixotropic stiffening appears to changes of the static yield stress at rest with a change of rheological parameters (viscosity and yield stress) determined in the first phase of increasing shear rate, before shear thinning of the mixture. Based on the tests performed, it can be concluded that the thixotropic behaviour of the mortar characterize mortars with a high content of cement with a low coefficient of w/c thereby also relatively high dose of superplasticizer.

Not without significance, mortar composition is of major significance, particularly the impact of the type of cement with a compatible admixture. With the use of multicomponent cement (e.g. CEM V), the thixotropic stiffening character of mortars is clearer, but this is not a rule.

The open issue is the adoption of a method to estimate which of the effects: thixotropic behaviour or stiffness caused by loss of workability, plays a leading role in the reduction of pressure on formwork. Verification results of designed mortars and SCC in terms of rheological properties, including thixotropic stiffening and loss of workability, will be assessed during research into the influence of the rheological properties of SCC on formwork pressure.

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