**Abstract**

This paper presents methods of measurement and results of rheological properties including thixotropic stiffening of designed cement mortars. The aim of the study is to determine the temperature rheological properties of selected mortars. It will help the industry to better understand and describe the nature of the previously observed decreasing formwork pressure using self-compacting concrete.

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

**Streszczenie**

W artykułe przedstawiono metody badawcze oraz wyniki właściwości reologicznych, w tym tiksotropowego sztywnienia zapraw cementowych. Celem badań było określenie wpływu temperatury na wybrane właściwości reologiczne zaprojektowanych zapraw cementowych. Wyniki badań pozwolą lepiej zrozumieć i opisać charakter zaobserwowanego już wcześniej charakteru zmniejszania się parcia mieszanki samozagęszczalnej na deskowanie.

*Słowa kluczowe: BSZ, tiksotropowe sztywnienie zapraw, właściwości reologiczne zapraw, statyczna granica płynięcia, wpływ temperatury*

1. Introduction; 2. Research significance

Compare with pages: 22-23.

3. Experimental investigation

The rheological parameters of mortars were determined by using a rheometer Viskomat NT described in detail in [11]. Studies [6, 7, 11] demonstrate what it is commonly accepted, that rheological behavior of fresh mortar and concrete may be sufficiently described by the Bingham model. In rheometry, the Bingham model is described in

\[ M = g + N h \] (1)

where:

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

To determine mortar parameters \( g \) and \( h \) was used modified in accordance with [2, 4, 7] described in detail in [1, 3] (Fig. 2).

![The rheological test procedure for Viskomat NT](image)

Fig. 2. The rheological test procedure for Viskomat NT

Parallel to the rheometric tests, performed technical mortar tests, according to PN-EN 1015-3 (without flow table, only flow mould). Tests were determined after mixing (0'), after 40 minutes of resting (40') and in the 80 minute, after re-mixing (80'). The temperature of the mortar during the measurements were kept at a determined level (10°C, 20°C and 30°C). The proposed procedure made it possible to measure the following properties: the static yield stress after resting (1\textsuperscript{st} and 2\textsuperscript{nd}, 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 an increasing shear rate in a range of speed from 1 to 30 rpm and the dynamic yield stress and plastic viscosity decreasing shear rate.

The amount of PE was chosen in such a way that the mortar spreading was 25–30 cm in temperature of 20°C. When it was not possible, it was used maximum amount of admixture, which did not yet caused mortars segregation.
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>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>
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<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>
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<td></td>
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<tr>
<td>CEM III/A</td>
<td>[kg]</td>
<td>776</td>
<td>680</td>
<td>784</td>
<td>682</td>
<td></td>
<td></td>
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<tr>
<td>42.5N-HSR/NA</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>776</td>
<td>680</td>
<td>784</td>
<td>682</td>
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<td></td>
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<tr>
<td>CEM V/A (S-V)</td>
<td>[kg]</td>
<td>751</td>
<td>662</td>
<td>757</td>
<td>668</td>
<td></td>
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<tr>
<td>32.5R-LH</td>
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<td></td>
<td></td>
<td>751</td>
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<tr>
<td>Water</td>
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<td>236</td>
<td>276</td>
<td>233</td>
<td>272</td>
<td>235</td>
<td>273</td>
<td>225</td>
<td>264</td>
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<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>
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</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>
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<td></td>
<td></td>
<td></td>
<td>2.00</td>
<td>0.75</td>
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<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>
<td>2.50</td>
<td>1.50</td>
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<tr>
<td>Sand 0–2</td>
<td>[kg]</td>
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<td>1315</td>
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</table>

5. Experimental results and discussion

5.1. Influence of temperature on initial mortar viscosity and initial yield stress

Research showed (Fig. 3), that after resting, the initial viscosity was increasing and after re-mixing decreasing. After mixing, the initial yield stress decreased in case of CEM I with PE1. For PE2 which had the same cement, viscosity in 10°C and 30°C which increased. In case of CEM III and CEM V with different admixtures, there wasn’t a significant effect of temperature at 10 and 20, 30°C. The initial viscosity decreased in the case of PE2. After resting, for CEM I with PE1 viscosity was decreasing at 10 and 30°C, greater at 30°C. Quite the opposite occurred for PE2. In case of CEM III with increasing temperature, for PE1 viscosity was decreasing and increasing for PE2. In case of CEM V and PE1 viscosity decreased at 10 and 30°C, greater at 30°C. Quite the opposite occurred for PE2. After re-mixing, for CEM I, with increasing temperature, viscosity increased. The increase was small for CEM I with PE1 in 10 and 20°C and larger at 30°C. For CEM III and different admixtures,
there wasn’t a significant effect of temperature at 10 and 20. For 30°C viscosity increased, greater in case of SP2. For CEM V with increasing temperature viscosity was decreasing. For PE1 viscosity at 10 and 20°C viscosity was similar.

Fig. 4 shows that after mixing for CEM I with PE1 there wasn’t any influence of temperature on the initial yield stress at 20 and 30°C. There was a significant increase at 10°C. For PE2 there wasn’t any influence of temperature on initial yield stress at 10 and 30°C, which was higher than at 20°C. For CEM III with PE1 initial yield stress at 10 and 30°C was higher than at 20°C. In that case at 30°C yield, the stress was higher. Quite the opposite occurred for PE2. For CEM V with increasing temperature, initial yield stress slightly increased. After resting, the initial yield stress for CEM I with PE1 was increased, higher at 30°C than at 10°C. For PE2, the initial yield stress was higher, at the same level, at 10 and 30°C than at 20°C. For CEM III with PE1 with the temperature increasing, the yield stress value slightly decreased. In the case of PE2, the initial yield stress increased at
10°C and decreased at 30°C in comparison with 20°C. Quite the opposite occurred for CEM V and PE1. For CEM V with PE2 there was a visible influence of temperature on the initial yield stress. With increasing temperature, yield stress increased. After re-mixing, the nature of the change in initial yield stress was similar when compared with mortars after mixing for CEM I, CEM III with PE2 and CEM V with PE1. For CEM III with PE1 with an increasing temperature, the initial yield stress increased and for CEM V, as with PE2, the value of yield stress at 10 and 30°C was lower than at 20°C.

For ratio \( w/c = 0.4 \), after resting, viscosity showed a significant decreased at 10°C (Fig. 5).

There was a significant effect of temperature in the case of CEM I and CEM III as with PE2. With an increasing temperature, viscosity decreased. For CEM I and PE1 at 30°C it slightly increased and for CEM III as with PE2 at 30°C it was at the same level as it was at 20°C. In case of CEM III with PE1 viscosity was lower at 10 and 30°C than at 20°C, lowest at 30°C. For CEM V at 20°C viscosity was at the same level, increasing at 10°C and decreasing at 30°C. For CEM V there was an influence of PE and temperature. For PE1 at 10°C viscosity was higher than at 30°C and vice versa. After resting with increasing temperature, viscosity increased for CEM I with PE1 and decreased for CEM III with PE1. In other cases, the respective mortars, at 10°C viscosity increased by more than at 30°C in comparison with 20°C. After re-mixing with an increasing temperature, viscosity increased for CEM I, slightly in case of PE1, slightly increased for CEM III with PE2 and decreased for CEM III and PE1. In the case of CEM V, there was no effect of temperature 10 and 20°C for PE1 and for PE2 10 and 30°C. Viscosity decreased first at 30°C (slightly) and for the second at 20°C.

Fig. 6 shows that after mixing, CEM III with PE1 and for CEM I with PE1 there wasn’t any influence of temperature on initial yield stress. For the second only for 10 and 20°C. At 30°C initial yield stress increased. For CEM I with PE2, CEM III with PE2 and CEM V with PE1, with increasing temperature, with initial yield stress decreased, more in the case of CEM I. For CEM V with PE2, the initial yield stress increased the most at 10°C, less at 30°C. After resting for CEM III with PE2, CEM I with PE2 and CEM V with PE2 there wasn’t influence of temperature on initial yield stress. For the second only for 10 and 30°C (at 20°C yield stress was higher) and for the third only 20 and 30°C (at 10°C yield stress was higher). For CEM I with PE1 for 10 and 30°C, initial yield stress was increased, to 10°C higher.

Fig. 5. Influence of temperature on initial mortar viscosity (w/c = 0.4)
The opposite was the case of CEM V with PE1, where at 20°C the initial yield stress is at its highest. For CEM III with PE1 and with the temperature increasing, yield stress slightly increased. After re-mixing for CEM I with PE2, CEM III with PE1 and PE2, yield stress increased and decreased as in case of CEM V with PE2. The largest increase is at 30°C. For CEM I with PE1 and CEM V with PE1 with the yield stress increasing at 10 and 30°C. For the first highest was at 30°C, for the second at 10°C.

5.2. Influence of temperature on mortar flowability

Fig. 7 shows that after mixing there was an influence of temperature on mortar flowability. With increasing temperature, the spreading of mortar increased. After resting, with increasing temperature, the spreading of mortar decreased for CEM I and CEM III with PE2 and increased for CEM V with PE1 and PE2. For CEM I with PE1 at 20°C the spreading of mortar was higher than it was at 10 and 30°C. For CEM III with PE1 spreading slightly increased at 20°C and at 30°C rapidly decreased. After re-mixing there was an influence of temperature on mortar flowability, but not directly. For CEM I with PE1, CEM III and CEM V with PE1 and PE2 spreading increased at 20°C and decreased at 30°C. For the last at 20°C and 30°C there was no difference between spreading. For CEM I with PE1 with increasing temperature, spreading decreased.

Fig. 8 shows that after mixing there was an influence of temperature on mortar flowability. With increasing temperature, the spreading of mortar increased. The highest was in the case of CEM I. After resting at 20°C spreading increased, largest for CEM I with PE1 and CEM V. For CEM V with PE2 there wasn’t any influence of temperature on its flowability. After re-mixing with an increasing temperature spreading decreased for CEM I with PE1 and CEM III. For CEM I with PE2 at 20°C spreading increased and decreased at 10 and 30°C. Quite the opposite was true in the case of CEM V with PE1. For CEM V with PE2 there wasn’t any influence of temperature on mortar flowability.
5.3. Influence of temperature on 1st static yield stress

As the nature of the 2nd static yield stress were similar, less pronounced in the 1st static yield stress test, this paper presents only the influence of temperature on the 1st static yield stress.

Fig. 9 shows for mortars with ratio \( \frac{w}{c} = 0.3 \), for an increasing temperature, the 1st static yield stress increased at 20°C and at 30°C decreased in comparison with 10°C. The increase was higher in the case of CEM I with PE1 and CEM V. At 30°C the value of 1st static yield stress in each case was lower than at 10°C. After mixing, the influence of temperature was the same as after mixing. The difference was in the case of CEM I with PE1 and PE2, which was the opposite. After re-mixing, the influence of temperature was the same as after resting. The highest intensity was in the case of CEM I with PE2, CEM III with PE1.

Fig. 10 shows that for mortars with a ratio of \( \frac{w}{c} = 0.4 \), for an increasing temperature, the 1st static yield stress decreased in case of CEM I, lower for PE2. For CEM III, at 20°C the 1st static yield stress increased, higher for PE2 than for PE1 and decreased. Its value was at the
same level at 10 and 30°C. For CEM V, at 20°C yield stress increased (higher for PE1) and decreased at 30°C (much more for PE2). At 30°C the value was higher for PE1 and lower for PE2 in comparison with 10°C. After resting the 1st static yield stress increased at 20°C and at 30°C decreased in comparison with 10°C. The highest change was in CEM I with PE2, CEM III with PE1 and CEM V. After re-mixing the nature of the 1st static yield stress was the same as it was when compared to mortars after resting.

6. Conclusions

The study indicates that the thixotropic stiffening of mortars are a very complex phenomenon which 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 in changes of the static yield stress at rest with a change of rheological parameters (viscosity and yield
stress) determined in the first phase by an increasing shear rate, before shear thinning of the mixture. Based on the tests performed, it can be concluded that the thixotropic behaviours which characterize mortars with a high content of cement and a low coefficient of w/c, a high dose of compatible admixtures and temperature: in particular 10°C and 20°C. With the use of multicomponent cement (e.g. CEM V, in particular with PE1), the thixotropic stiffening character of mortars is clearer. But this is not a rule (e.g. CEM I with PE1).

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 the thixotropic stiffening and loss of workability, will be useful during research into the influence of rheological properties of SCC on formwork pressure.

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References


