

## Rheological properties of geopolymer binders and new method of measurements

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### Abstract

The aim of this work is to study rheological changes of geopolymers in the initial phases of solidification of stabilization binders using two standard and one new method. Measurement of dynamic viscosity with a rotary viscometer and determination of the initial setting time of solidification with a Vicat instrument were used as standard. In order to better characterize the solidification process of geopolymer materials, a method for measuring the flow rate through the needle under defined conditions was designed and confronted. These methods were focused on the influence of the water content in initial stages of solidification. The results show that the rotary viscosimeter is not suitable for such measurements which is related to the thixotropy of the material. The Vicat's device gives relevant times of solidification, but it measures the already technologically unprocessable form of binders. The optimal method for practical use and for the estimation of the workability times of geopolymers is the designed measurement of the flow velocity through a needle under controlled and well defined conditions.

**Keywords:** geopolymer, binder, rheology, initial setting time, viscosity.

### Introduction

A large amount of waste and its high variability are the problems of present waste processing and they exceed the recycling options and capacities now available. Therefore, some problematic waste has to be treated before disposal to prevent environmental contamination by dangerous compounds. In many cases, stabilization of the compounds can solve the problem and one of the most used stabilization processes is solidification.

For solidification and immobilization of dangerous or unsuitable wastes, that have either organic or anorganic origin, the solidifying binders on various bases are used. These can be cement binders, lime-based binders, ash, sulfur or asphalt based binders<sup>1-5</sup>. Recently, alkali activated aluminosilicate based binders are used which are known as geopolymers. Such binders can be prepared from various precursors by many different ways. Resulting geopolymer then may have a broad range of properties (mechanical, structural or chemical etc.). The advantage over other materials is the possibility of preparing a stabilizing binder with properties optimized for a specific waste. Geopolymers are materials most often prepared by mixing aluminosilicate powder with an alkali metal hydroxide solution (usually NaOH and KOH) or with an aqueous alkali metal silicate solution (water glass)<sup>6</sup>. After mixing the raw materials and their homogenization, the alkaline hydrolysis process begins, where the aluminosilicate powder is partially dissolved. This is followed by a polycondensation process associated with the growth of a three-dimensional polymer network. The resulting geopolymer can have mechanical properties comparable (or even better) with concrete and it has very good ability to bind metal cations at the same time. Geopolymers can be used to stabilize e.g. waste sludge from alumina production, waste containing heavy metals, or radioactive waste<sup>7-11</sup>.

Geopolymers can also be prepared from power plant fly ash<sup>12-14</sup>. The potential of such geopolymers is mainly in the construction industry. According to The Association for Utilization of Coal Combustion

Products, the Czech Republic produces 11 to 14 million tons of energy by-products as waste from coal power plants and heating plants annually. With price of tens of crowns (CZK) per ton, it is a very interesting raw material for the production of geopolymers in large volumes<sup>15</sup>. However, the disadvantage is that it is chemically very heterogeneous raw material, which often contains hazardous elements, because the fly ash chemical composition is strongly dependent on the input material and the combustion technology.

To ensure the stability of the geopolymer properties as binders for the solidification of waste, it is necessary to produce them from stable raw materials. A suitable raw material is aluminosilicate with high content of metakaolinite, which is produced on an industrial scale by calcination of kaolins or kaolinitic claystones<sup>16,17</sup>.

For industrial applications of geopolymer binders, including solidification of waste, an important property is their viscosity and its changes over time, i.e. during the solidification of geopolymer binders. It is suitable to use aluminosilicate from kaolinitic claystone in case of low viscosity geopolymer binder, as reported in the literature<sup>16,17</sup>.

However, a fundamental problem of geopolymers is the determination of rheological properties and their workability time. Standard methods, both the dynamic method using a rotary viscometer and the static method using a Vicat instrument, do not meet the real requirements for the workability time determination of geopolymers. Therefore, a new method better corresponding to the real needs was proposed and confronted in this work.

## Materials

Industrially produced precursors were used for the preparation of the geopolymer binder. Aluminosilicate with a high content of metakaolinite with the trade name Mefisto L<sub>05</sub> produced by the calcination of kaolinitic claystone in a rotary kiln at a temperature of 750 °C (České lupkové závody, a.s., Czech Republic), potassium water glass KVS 3,2-3,4 (Vodní sklo, a.s., Czech Republic) and solid potassium hydroxide (G.R. grade, 88.2 wt% KOH, Lach-Ner, s.r.o., Czech Republic) were used. The chemical composition of the base materials is given in Table 1. The phase composition of Mefisto L<sub>05</sub> is shown in Figure 1. It is clear from the particle size analysis that 50 % of all Mefisto L<sub>05</sub> particles is smaller than 3.7 µm and 90 % of all particles is smaller than 10.6 µm. The particle distribution is shown in Figure 2.

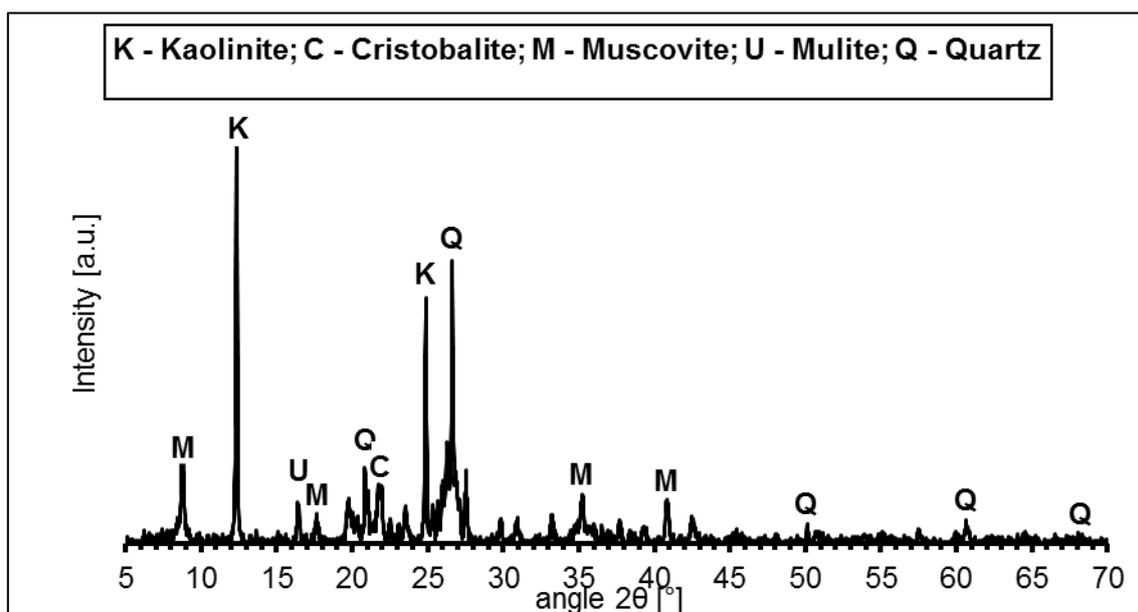
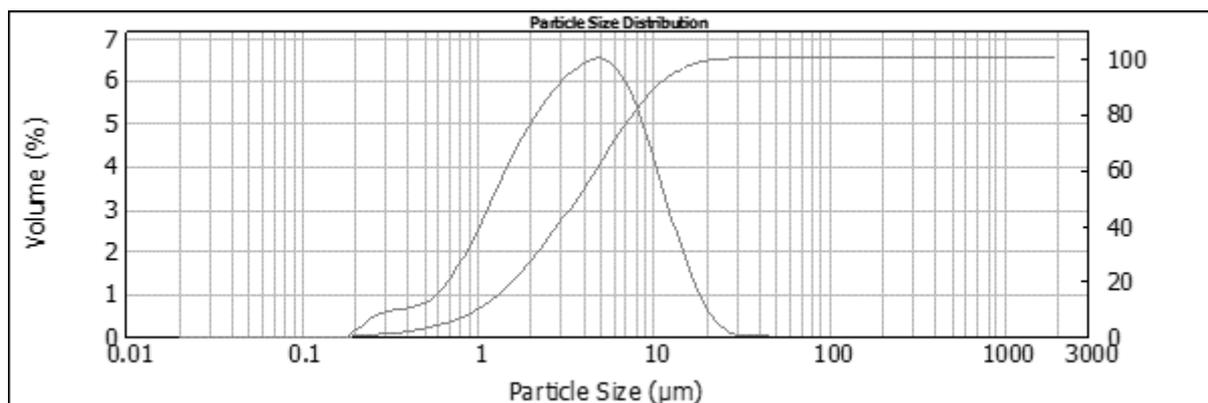


Figure 1: XRD patterns of aluminosilicate Mefisto L<sub>05</sub>

**Table 1: Chemical composition (wt%) of raw materials**

	Mefisto L <sub>05</sub>	potassium silicate
LOI <sub>a</sub>	1.53	-
H <sub>2</sub> O	-	60.44
SiO <sub>2</sub>	51.9	27.00
Al <sub>2</sub> O <sub>3</sub>	42.5	0.05
TiO <sub>2</sub>	1.66	-
Fe <sub>2</sub> O <sub>3</sub>	0.88	-
K <sub>2</sub> O	0.83	12.12
CaO	0.16	-
MgO	0.16	-
SO <sub>3</sub>	0.08	-
P <sub>2</sub> O <sub>5</sub>	0.07	-
V <sub>2</sub> O <sub>5</sub>	0.05	-
MoO <sub>3</sub>	0.04	-
Na <sub>2</sub> O	0.04	0.38
Cr <sub>2</sub> O <sub>3</sub>	0.03	-
ZrO <sub>2</sub>	0.02	-
ZnO	0.01	-
SrO	0.01	-
CuO	0.01	-
NiO	0.01	-
Ga <sub>2</sub> O <sub>3</sub>	0.01	-
As <sub>2</sub> O <sub>3</sub>	0.01	-

<sup>a</sup> LOI = Loss on ignition.



**Figure 2: Particle size distribution of aluminosilicate Mefisto L<sub>05</sub>**

## Experimental

### *Analytical and testing methods*

The chemical composition of the aluminosilicate was determined by X-ray fluorescence (BRUKER S8 Tiger).

The phase composition of Mefisto L<sub>05</sub> was determined by X-ray diffraction (BRUKER D8 Advanced equipped with a BRUKER SSD 160 detector). The XRD was measured using Cu-K $\alpha$  radiation at 40 kV and 25 mA. A scan with steps of  $2\theta = 0.02^\circ/\text{sec}$  for the angles ranging from  $5^\circ$  to  $70^\circ$  was used.

The chemical composition of the water glass was determined using an OPTIMA 8000 inductively coupled plasma optical emission spectrometer (Perkin Elmer). The total content of alkali metals and SiO<sub>2</sub> in the water glass was determined by conventional acid-base titration method.

The Mefisto L<sub>05</sub> metakaolinite particle size distribution was measured using a Mastersizer 2000 laser diffraction analyzer (MALVERN Instruments). Prior to the actual measurement, the agglomerates were disrupted by sonication.

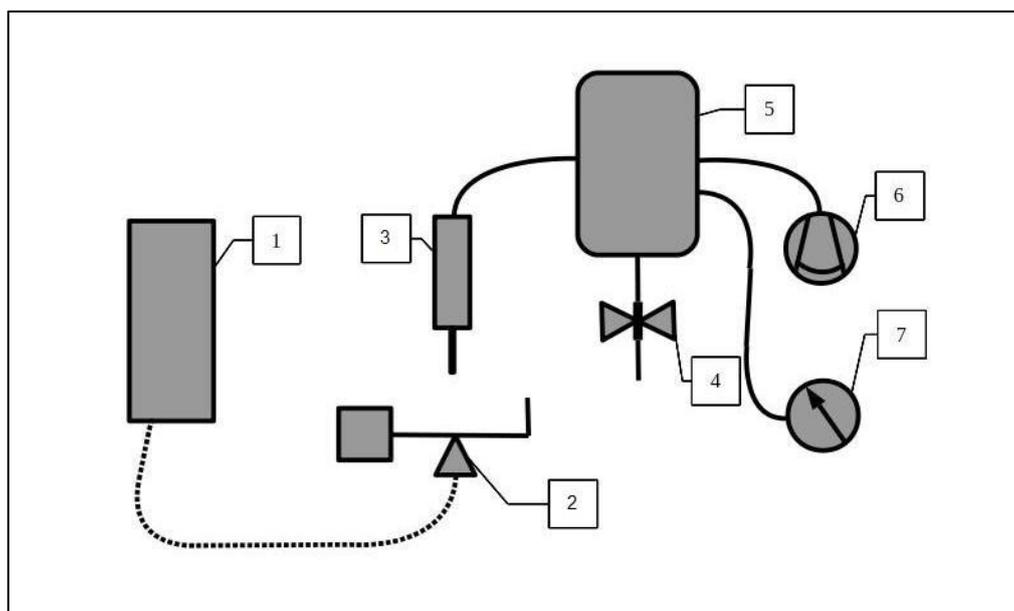
The dynamic viscosity of the geopolymer binder was measured using a Rheotest RN 4.1 rotary viscosimeter (Rheotest Medingen) with an arrangement of concentric cylinders with a 1 mm gap. The outer diameter of the rotating cylinder was 36 mm and the inner diameter of the static cylinder was 38 mm. The static cylinder was tempered at  $25^\circ\text{C}$  by a thermostat. The binder was transferred to the measuring cylinder immediately after mixing and the dynamic viscosity measurement was started twenty minutes after the binder mixing began. The shear rate was  $75\text{ s}^{-1}$ . The time curves of the dynamic viscosity had a characteristic shape consisting of three distinct parts. The initial part of each curve was almost linear until a significant convex part followed by another almost linear viscosity increase. Initial setting time was calculated from the intersection of the two linear parts of each curve. The first regression line based on the initial part of the measurement was considered to be a constant, i.e. a line parallel to the x-axis (the same values of viscosity were obtained for an extended period of time with sufficient accuracy). The second regression line corresponds to the linear part with a bigger slope. Furthermore, the slopes of the second part of the curves characterize the solidification rates of the binders. The larger the slope, the faster the binder is solidified. The initial setting time, measured with the rotary viscosimeter are discussed later.

Determination of the initial setting time of the geopolymer binders was performed using a Vicat's instrument VICATRONIC (MATEST). The measurement was performed according to the adapted standard ČSN EN 196-3:2017<sup>18</sup>. The Vicat apparatus was placed in an air-conditioned chamber at  $25^\circ\text{C}$  and 90 % relative humidity (r.h.) throughout the measurement, to ensure reproducibility of solidification conditions. The high humidity in the air conditioned chamber reduced the water evaporation from the geopolymer surface. The initial setting time was then determined by the time when the needle of the Vicat apparatus for the first time did not fall to the bottom of the test vessel, but remained above its bottom.

The determination of the geopolymer temperature during the initial setting time were recorded using the GMH 3250 electronic logger (GREISINGER electronic) with a standard K-type thermocouple. The temperature values were recorded every 5 minutes.

Determination of initial setting times of geopolymer binders with different water contents using a pressure needle apparatus was performed on an apparatus designed on the basis of previous experience with the test of geopolymer binders applicability using injection needles, described by Koutník et al.<sup>19</sup>. The test was based on measuring the flow of binder extruded through a needle with an outer diameter of 2.0 mm with constant air pressure of 13.3 kPa (100 Torr) generated by a pump. The air pressure in the apparatus was regulated by a needle valve and measured with an accurate digital pressure gauge. The binder flow rate was evaluated by measuring the weight of the binder passing through the needle in real time. Digital weighing scales with an accuracy of one mg connected to a PC with the weight being logged with interval of 1 s were used. The whole apparatus was placed in an air-conditioned chamber, which maintained constant conditions ( $25^\circ\text{C}$  and 50 % r.h.). The measurement took place immediately after mixing the binder or with the start delayed by up to 240 minutes. For the

delayed start measurement, the binder sample was transferred to a syringe placed in the apparatus after mixing and the compressed air was not introduced into the syringe immediately, but with appropriate delay. The initial setting time of binder was defined as the delay time at which the binder flow in the first 10 s of the measurement reached  $30\pm 5\%$  of the flow of freshly prepared binder. This limit was based on previous experience and a number of observations. Longer delay time of the binder before processing, which results in flow rates below 30 % of the initial value, usually does not allow its application with achieving comparable properties after hardening. A simplified diagram of the apparatus is shown in Figure 3.



**Figure 3: Schematical drawing of the needle pressure apparatus: 1 – control and data collection PC; 2 – digital weighing scales with digital output; 3 – 50 ml PP syringe with needle; 4 – air needle valve; 5 – expansion tank; 6 – diaphragm air pump; 7 – pressure gauge.**

### **Preparation of geopolymer binders**

The alkaline activator was prepared by dissolving potassium hydroxide in potassium water glass. To remove water absorbed during grinding and storage, Mefisto L<sub>05</sub> was dried in an oven at 110 °C. The preparation of the geopolymer binder itself consisted in mixing the alkaline activator with the dried aluminosilicate raw material in a planetary mixer at room temperature for 10 minutes. Then, distilled water was added to adjust the water content of the geopolymer binder, followed by further stirring for 5 minutes. Five binders with total water content of 30, 35, 40, 45 and 50 % were prepared. The total water content of the geopolymer binder is the sum of the water added during homogenization in the planetary mixer and the water contained in the raw materials. The Me/Al molar ratio was kept at 1.0 in all binders and the Si/Al molar ratio was always 1.5.

These values were chosen on the basis of the research of Koutník et al.<sup>16</sup> due to the low viscosity of the binder after mixing and high strength of the binder after solidification at the same time.

The sample labeling is given in Table 2.

**Table 2: Sample labeling and parameters. Ratios Me/Al, Si/Al and water content in weight %**

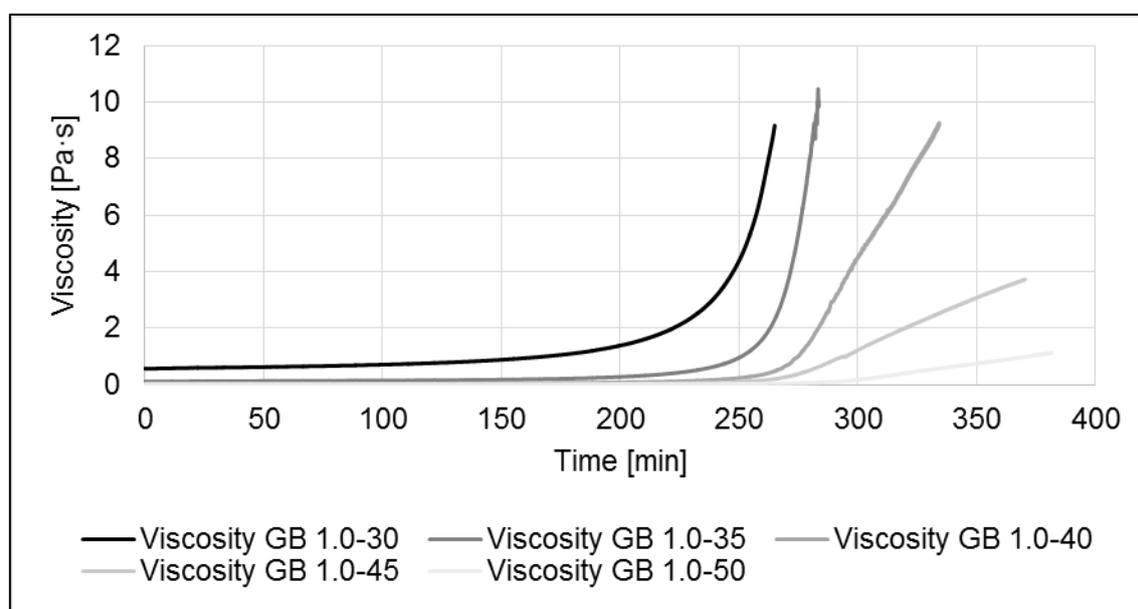
Marking of geopolymer binders	GP 1.0-30	GP 1.0-35	GP 1.0-40	GP 1.0-45	GP 1.0-50
Molar ratio Me <sup>a</sup> /Al	1	1	1	1	1
Molar ratio Si/Al	1.5	1.5	1.5	1.5	1.5
Water content [%]	30	35	40	45	50

<sup>a</sup>Me = Na + K

## Results and discussion

### Determination of dynamic viscosity with a rotary viscosimeter

The dynamic viscosity estimated by measurement with the rotary viscosimeter is shown in Figure 4 for binders with different water contents. Clear differences in time dependence of viscosities can be seen. All measured curves show an almost linear dependence of viscosity on time until a steep increase is detected as already mentioned (see Figure 4). Initial setting time was calculated from the two linear parts intersections as described in the Experimental section. Similarly, the results of viscosity measurements were evaluated e.g. in the works of Arnoult<sup>20</sup> and Archez<sup>21</sup>. For the curves corresponding to binders with 30, 35 and 40 % of water, the steep part of the curve shows a sharp increase in viscosity corresponding to the solidification process. The curves of 45 and 50 % binders obviously show the same change, however, the slope of the steeper part of their curves is significantly lower compared to 30-40 % binders. An example of the solidification time evaluation is shown in Figure 5 (binder with 30 % water in this case). Formally, the 45 and 50 % curves can be evaluated in the same manner, but the solidification does not take place in this range of the water content. Moreover, the initial setting time calculated by the method of two linear parts intersection for 45 % binder is shorter than in case of 35 % binder, which is rather unexpected and improbable. Figure 6 shows the initial setting times of geopolymer binders with different water contents. From above mentioned reasons, the use of the rotary viscosimeter for initial setting time calculation has some limitations and should be carefully checked. The most probable reason for such behaviour is the thixotropy of the geopolymer binders as described by Bong<sup>22</sup>.



**Figure 4: Time changes of dynamic viscosity measured by a rotary viscosimeter**

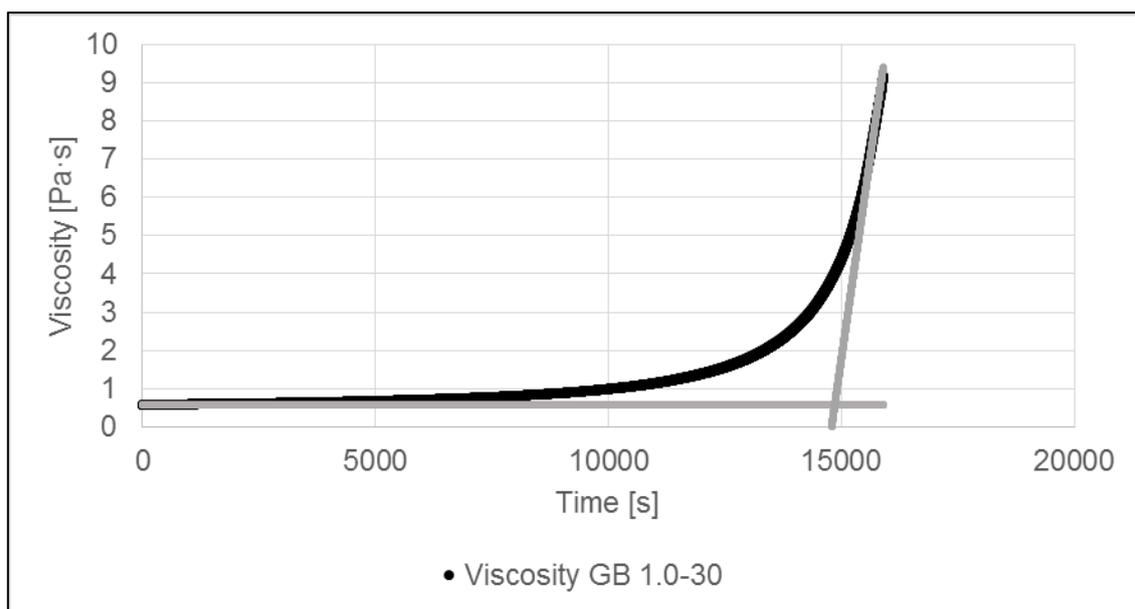


Figure 5: Example of initial setting time calculation from rotary viscosimeter measurements

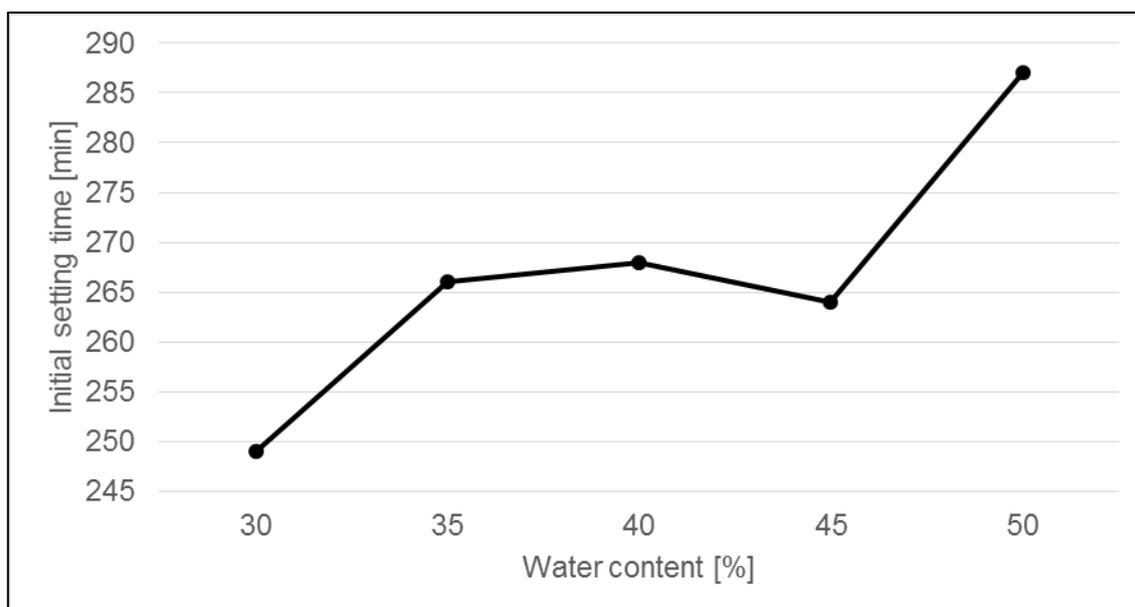
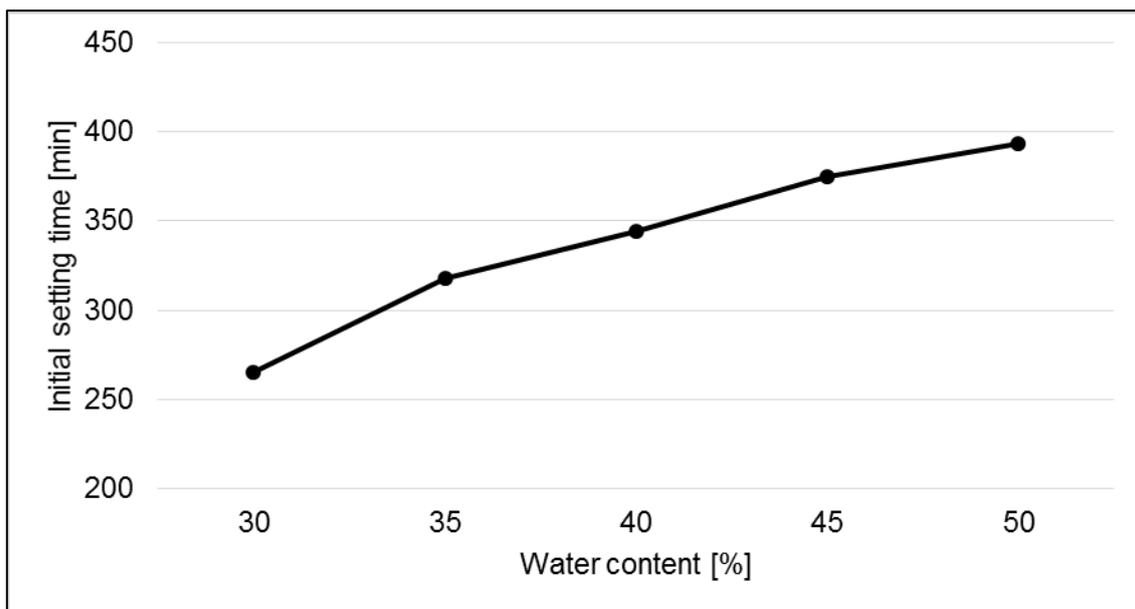


Figure 6: Initial setting times calculated from rotary viscosimeter measurements at 25 °C

### Determination of initial setting time by a Vicat device

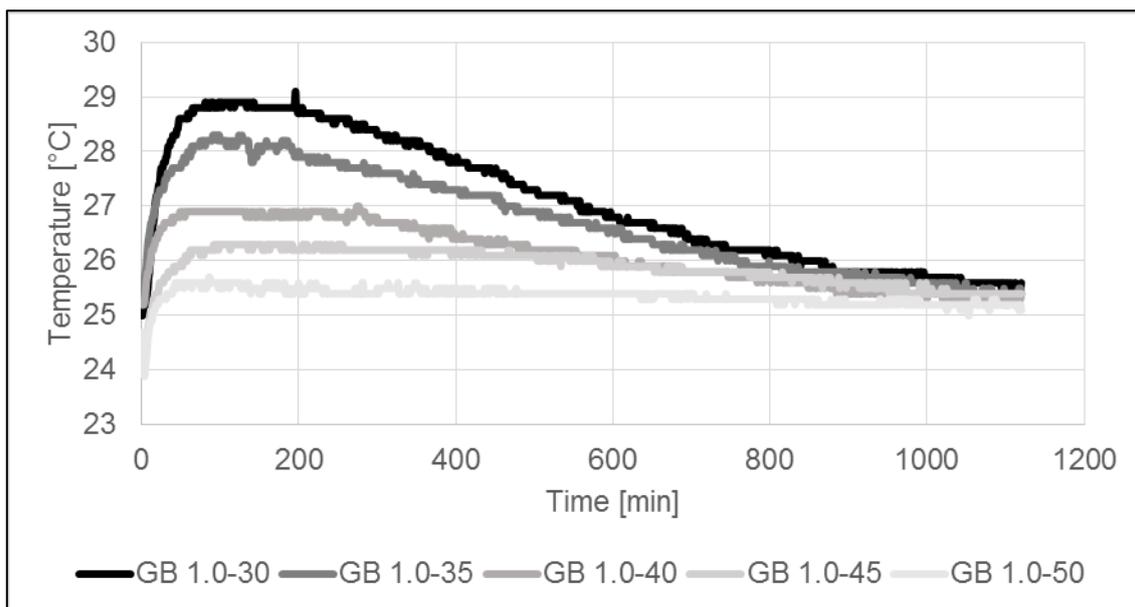
The results for geopolymer binders with different water contents GB 1.0 - 30 to GB 1.0 - 50 are shown in Figure 7. For all samples, control vessel (shape-identical to the vessel with the Vicat device) was used for the simultaneous temperature measurement. The temperature was always measured in the middle of the control vessel. The results of the temperature measurements are shown in Figure 8.

GB 1.0-30 binder started to solidify the fastest and the initial setting time of geopolymer binder increased with an increasing amount of water from 265 minutes (K2G80n GB 1.0 - 30) to 393 minutes for GB 1.0 – 50 binder. The Vicat test corresponded to the real behaviour of the geopolymer solidification time at rest, i.e. without any agitation, depending on the water content. However, the real workability time of the binder is shorter than the initial setting time, when the geopolymer is no longer in a liquid state. Gao<sup>23</sup> and Kaja<sup>24</sup> also used the Vicat apparatus and the standard EN 196-3<sup>18</sup> to determine the initial setting time. However, their geopolymer mixtures were with the addition of limestone, so the initial setting times are incomparable.



**Figure 7: The initial setting times of geopolymer binders with 30 to 50 % of water at 25 °C determined by a Vicat apparatus**

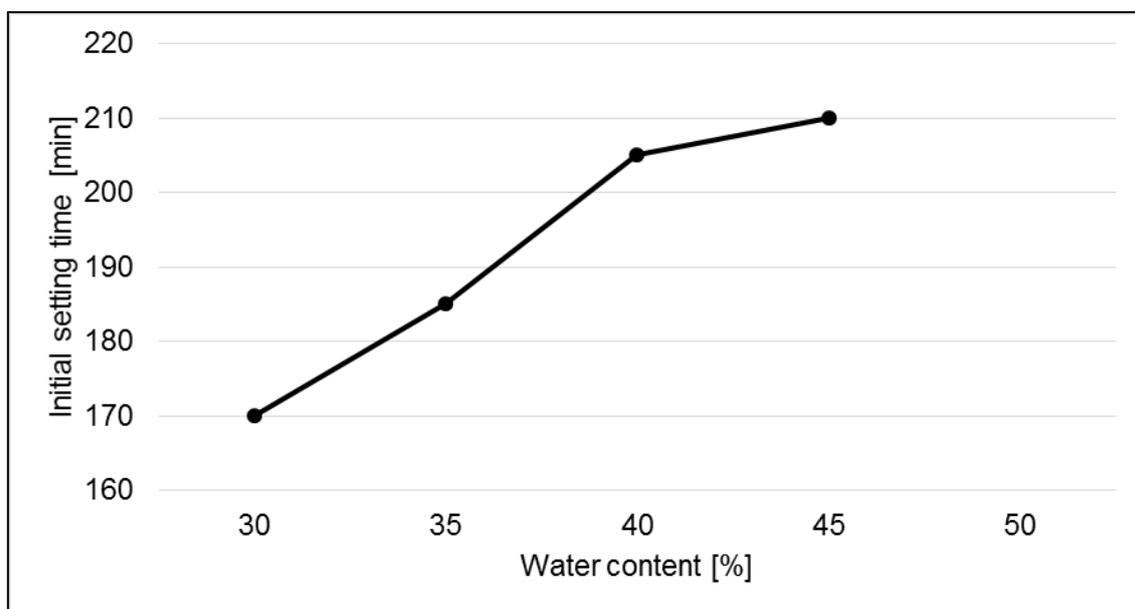
Figure 8 shows the temperature profiles inside the control specimen for samples GB 1.0 – 30 to GB 1.0 - 50 with different water contents, which were placed in an air-conditioned chamber with a constant temperature of 25 °C. Figure 8 shows the increase of temperature in the middle of the control vessel to 29 °C in the case of the GB 1.0 – 30 sample followed by gradual decrease to the tempering temperature. The highest maximal temperatures were recorded for samples with a lower amount of water, while the lowest maximal temperatures were recorded for the samples with higher water content. Similar measurements were performed e.g. by Novais<sup>25</sup>, who measured the dependence of the temperature of the solidifying binder on the water content. His work describes the same trend, i.e. a rapid rise in temperature with gradual temperature decrease. It is clear that geopolymerization is an exothermic process. The heat generated by geopolymerization is used to increase the material temperature. Due to the high specific heat capacity of water and the inhibition of the geopolymer reaction, geopolymers with a higher water content do not reach such temperatures of geopolymers with lower water content.



**Figure 8: Dependence of sample temperature on time for samples GB 1.0 – 30 to GB 1.0 – 50.**

### **Determination of the initial setting time using velocity of the flow through a needle**

The results of the initial setting times measurements are shown in Figure 9. The initial setting times for binders with water content of 40 and 45 % are very similar. There are larger differences between binders with water content of 30, 35 and 40 %. It was not possible to determine the initial setting time for a binder with water content of 50 %. The binder was so thin that in case of a longer delay time before the measurement started, the undissolved aluminosilicate raw material sedimented to the needle, and thus the needle became blocked. The reason is that the lower viscosity results in lower resistance of the environment to the particle movement and in turn accelerates the sedimentation of the solid phase. However, geopolymer with 50 % water is very unsuitable for practical use, therefore this method is also suitable for most low-viscosity geopolymer binders applicable in practice. The results show that with increasing water content in the binder, the time corresponding to the initial setting time also increases and the binder is applicable for longer time.



**Figure 9: Dependence of the initial setting time of the binders on the water content given by the needle apparatus**

Determining the initial setting time using a needle apparatus described the state of the binders with different amounts of water, when these were still processable. It was not a static process as in the case of measurement with the Vicat device, nor permanent mixing as in the case of the rotary viscosimeter. Therefore, the results of this method can be considered to be more corresponding to practical use.

### **Comparison of the initial setting times for various measurement methods**

A comparison of the resulting initial setting times of geopolymer binders shows significant differences depending on the measurement method used. Figure 10 shows the initial setting times of the binders for all three methods mentioned in this work. The shortest initial setting times are obtained for the measurement of the flow through the needle (170 – 210 min for binders with 30 – 45 % water), followed by a rotary viscosimeter (248 – 287 min for 30 – 50 % water) and the highest values were measured with the Vicat apparatus (260 – 393 min for 30 – 50 % water). The Vicat device shows true solidification when the geopolymer is already beyond the workability time limit. The results from the rotary viscosimeter are affected by the thixotropy of the geopolymers with high water content (45 and 50 %) and do not correspond to the usual conditions of geopolymer aging at rest. On the other hand, the measurements confirm the expectation, that agitation of the mixture can prolong the workability time of the geopolymer. In comparison to the results from the static Vicat test, the curves for 45 and 50 % water (Figure 4) from

the rotary viscosimeter indicate that even after a long time there would be no complete solidification during mixing, which correlates with the verification in practice.

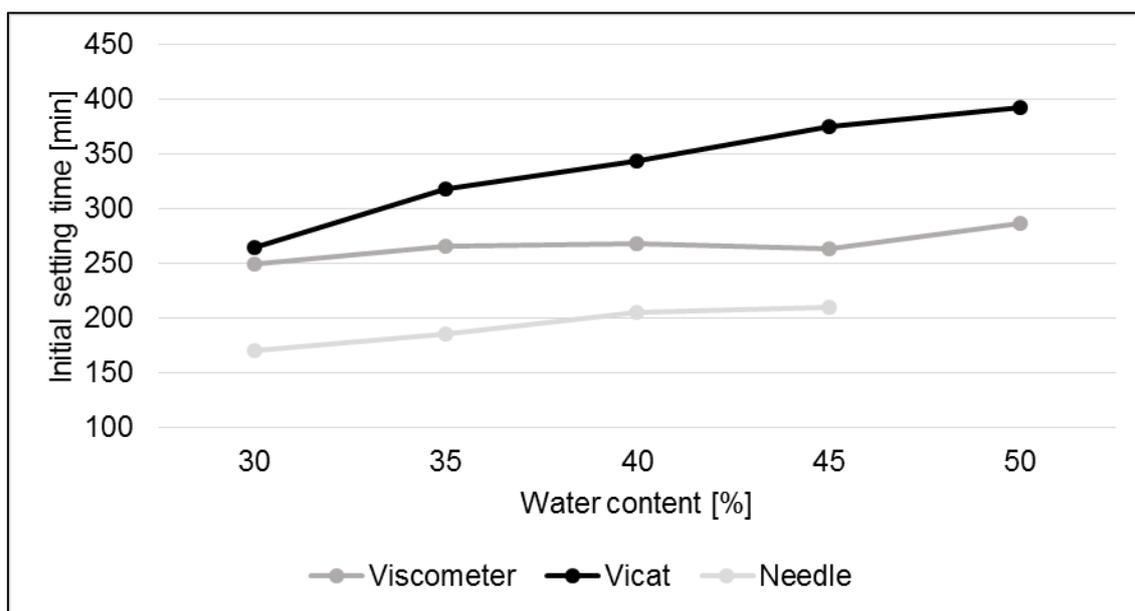


Figure 10: Initial setting times of the binders for three different measurement methods

## Conclusions

In this work, the rheological properties of low-viscosity stabilizing geopolymer binders with different water content were investigated by three methods - monitoring the flow rate of the binder through the needle under defined conditions, determining the viscosity with a rotary viscosimeter and determining the solidification rate with a Vicat device. The tests were supported by monitoring the temperature of the geopolymer binder during solidification. The results of all the above tests are consistent and confirm the already known assumptions:

- with higher water content, the viscosity of the binders decreases and the initial setting time is prolonged, the workability time is prolonged as well.
- as the binder solidification is an exothermal process, heat is generated which results in increased temperature of the mixture. This temperature decreases with increasing water content which is probably related to high specific heat capacity of water.
- long-term mixing of the geopolymer binder prolongs the initial setting time.

The rheological tests used so far made it possible to monitor the rheological properties of geopolymeric binders in the fresh state (rotary rheometer) or in the almost solidified state (Vicat's apparatus). The newly developed method of measuring the flow rate of the binder through a needle of appropriate diameter at suitable overpressure together with modifications of the standard viscosity measurement procedure allow investigation of the change in rheological properties of geopolymer binders during the whole solidification process from mixing the precursors to the geopolymer solidification. The developed method of binders evaluation using the flow through the needle seems to be the most suitable for determining the real workability time of a geopolymer binder.

The tested binders were processable for a long time from the mixing of the basic raw materials. The solidification initial setting time of the GB 1.0-30 binder measured by means of the Vicat instrument corresponded to the time when the rheometer recorded the maximum viscosity of the binder.

According to these measurements, the workability of the most viscous binder was determined for at least 250 minutes from the mixing of the basic raw materials. However, this does not correspond to the

real situation that the workability time is significantly lower and corresponds to the time measured by the designed needle apparatus (approximately 160 minutes).

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## Reologické vlastnosti geopolymerních pojiv a nová metoda měření

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### Souhrn

Cílem této práce bylo charakterizování změn reologických vlastností na počátku tuhnutí stabilizačních geopolymerních pojiv s využitím dvou standardních a jedné nově navržené metody. Standardně bylo použito měření dynamické viskozity rotačním viskozimetrem a stanovení počátku tuhnutí Vicatovým přístrojem. Za účelem lepší charakterizace procesu tuhnutí geopolymerních materiálů byla navržena a konfrontována metoda měření rychlosti průtoku pojiva jehlou za definovaných podmínek. Výše uvedené metody byly také využity pro stanovení vlivu obsahu vody na počátek tuhnutí geopolymerního pojiva.

Bylo zjištěno, že rotační viskozimetr není vhodný pro měření počátku tuhnutí geopolymerních pojiv s ohledem na jejich tixotropii. Měření pomocí Vicatova přístroje dává dobré výsledky časů počátku tuhnutí, ale měří již technologicky nezpracovatelnou formu pojiv. Pro praktické využití, respektive určení doby zpracovatelnosti geopolymerních pojiv, nejlépe vyhovuje navržená metoda, při níž je měřena změna rychlosti průtoku pojiva jehlou.

**Klíčová slova:** geopolymer, pojivo, reologie, počátek tuhnutí, viskozita.