

# Testing bodies for their deflocculation behaviour

### **1. Preparation of the sample**

The material to be examined is dried at 60 to 65  C in a dryer and comminuted using a jaw crusher and a cross hammer mill (screen insert 1 mm). Afterwards, depending on available amounts, the material is homogenized for 10 minutes either in a mixer or in a barrel using a roller. The time needed to prepare the samples is dependent upon the condition of the material at the time of delivery and the type of body.

### **2. Determination of the amount of mixing water required**

To determine the required amount of mixing water (mixing water = water content related to solid material) 300 g of the substance to be examined is slurried with:

120 g (= 40 %) water for plastic raw materials

60 g (= 20 %) water for non-plastic raw materials

in a plastic beaker using a Vollrath agitator, agitator head 60 mm at 750 rpm. Subsequently, 0.2 % deflocculant related to the solids content is added and homogenized for 10 minutes.

If the body remains stiff, the amount of mixing water is increased in 5% steps and the slip is homogenized for 5 minutes after each water addition. This procedure is continued until the mix becomes fluid. The amount of mixing water thus determined is held constant for the following examinations.

### **3. Determination of the casting curves**

The amount of mixing water that was determined in section 2 is filled into a container. Subsequently the body powder and deflocculant (0.1 % on solids content) are added.

After homogenizing for 10 minutes, the time of outflow is determined using a 100 ml pipette which has been calibrated with water at 20  C to a time of outflow of 3 seconds.

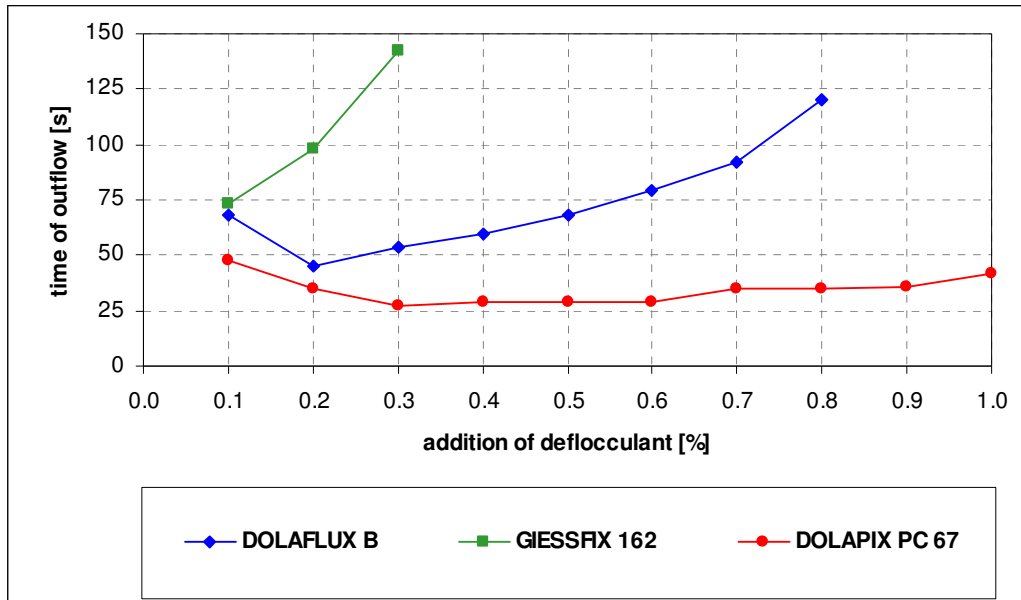
Then deflocculant is added in steps of 0.1 % up to a total amount of 1 % on solids content. If required, the deflocculant may also be added in 0.01 % steps.

For each composition, the time of outflow is determined after 10 minutes of agitation in the Vollrath agitator, with a 60 mm agitator head at 750 rpm.

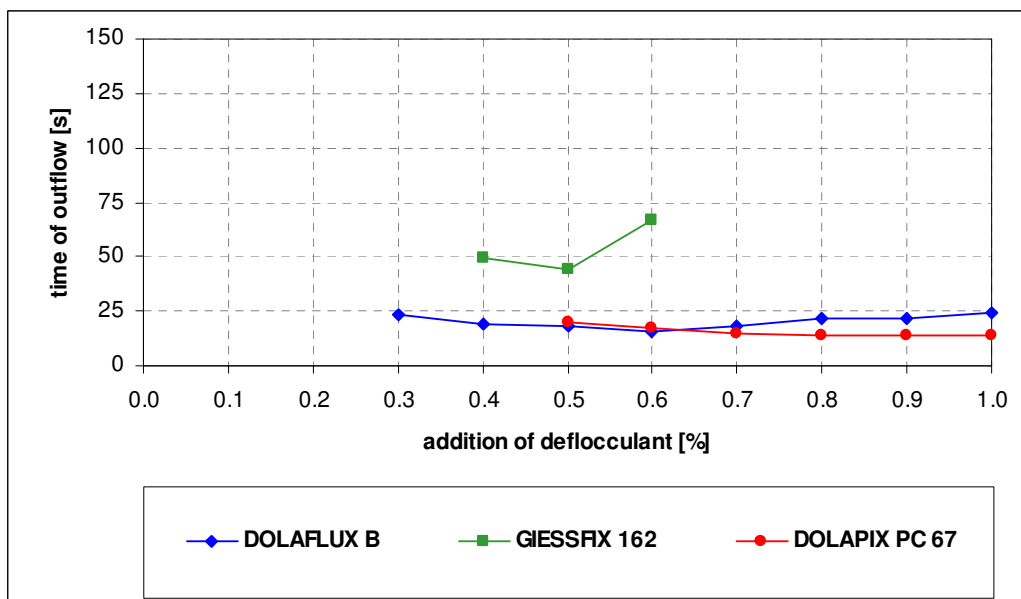
Thixotropic slips will not flow out completely. Thus, if the flow stops before the pipette is empty, the test has to be terminated. Thixotropic slip behaviour cannot be recorded in a casting curve as described below.

The times of outflow measured are recorded in a table, which is then represented graphically in a diagram. Diagrams of a sanitary ware body and of a tile body are given below as examples. From such diagrams, the most suitable deflocculant and its addition rate can be deduced.

### Casting curves of a sanitary ware body



### Casting curves of a tile body



The graphic of the **sanitary ware body** shows that in this particular case the best result was achieved with DOLAPIX PC 67, where the widest deflocculation range was achieved. There is a relatively high tolerance when processing the slip, so that variations in the production process can be compensated more easily. Moreover, the shortest time of outflow (which means lowest viscosity) is already achieved at an addition of 0.1 %.

For the **tile body** tested, DOLAFLUX B was best. Once again, a wide deflocculation range was found. DOLAFLUX B achieves deflocculation at 0.3 %, whereas deflocculation with DOLAPIX PC 67 requires an addition of 0.5 % or more.

The deflocculants to be tested are selected according to our long-standing experience with a wide variety of ceramic bodies.

To work out more details after determining the casting curves, viscosity is also measured with various other methods by examination of larger test mixes.

#### **4. Examination of the test mixes**

The calculated amounts of mixing water and deflocculant as obtained in section 3 are mixed with 500 g of the dry substance to be examined. The body is homogenized for 30 minutes using a Vollrath agitator with a 60 mm agitator head at 750 rpm. Afterwards the viscosity values are measured using a full pipette, a viscometer type Lehmann, as well as a rotation viscometer Viskotester (Haake), Gallenkamp, and/or Brookfield.

##### Measurement with pipette

100 ml of slurry is taken up into the pipette. The time of outflow is then determined in seconds.

##### Measurement with a viscometer according to Lehmann

The measurement using a viscometer type Lehmann is usually made with a 3.2 mm nozzle. Depending on the substance respectively on customer's requirements, a differently sized nozzle can be chosen (e.g. 6.38 mm for chamotte bodies). During the measurement, care must be taken that the temperature is maintained at 20  C and that the flow cup (Lehmann) is filled to the 500 ml mark.

Upon opening the outlet (turning the clamp) a stop watch is started simultaneously; it is stopped when exactly 100 ml slip is collected in a 100 ml flask (= Lehmann 1 ). The time of outflow measured serves as a measure of viscosity.

This slip quantity is used at the same time to determine the density. For this purpose, the previously tared flask, filled with slip, is accurately weighed to three decimal places.

After a standing time of 10 minutes, a second time of outflow (= Lehmann 2) is determined. The difference between the two values Lehmann 1 and Lehmann 2 is the measure for the thixotropy of the slurry.

Alternatively, the ratio of Lehmann 1 to Lehmann 2 can also be used for evaluation purposes.

Measurement using a rotating viscometer

The measurements using a rotating viscometer (Haake, Gallenkamp, Brookfield) are to be made according to the operating instructions of the specific manufacturer.

Using these testing methods, valid results for the deflocculation behaviour of the slurries can be determined. However, in order to obtain an exact analysis of the slurry rheology, the described procedures are not sufficient as they rely exclusively on singlepoint measurements.

In order to determine properties such as rheopexy, dilatancy, structural viscosity and Newtonian flow, a measurement over a range of shear gradients is necessary.

At Zschimmer & Schwarz, a rotating viscometer from the company Physica is available for use. Either rotation frequency or shear stress can be kept constant. Viscosity, flow behaviour and yield point of the slurry to be examined can thus be determined. The slurry is kept at a defined temperature using a thermostat.

If required, further tests concerning deflocculation and slip properties can be carried out, e.g. evaluation of a possible slip degradation over time, of sedimentation, and of casting rate. These results can be completed by testing the modulus of rupture of dried specimens.