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# **Two-component grout: a research study on the influence of bentonite and activation time**

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# Abstract

Two-component grout is one of the most popular backfilling techniques in shielded TBM projects thanks to its versatility and ability to minimize surface settlements compared to other backfilling technologies. A main component in two-component grout is bentonite, hence, a laboratory test campaign was performed in order to determine its effects regarding the technological properties and mechanical strengths of grout. For this purpose, twenty mix designs were tested, each one with a different type of bentonite and activation time. Bleeding, gel time, surface compressive strength (SCS), unit weight, and viscosity were measured, and the results are presented and discussed considering also the operational point of view.





*A Roberta*



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# 1 INTRODUCTION

Purpose of this research study is to analyse the influence of bentonite and activation time on properties of two-component grout.

Two-component grout is extensively used in backfilling operation when shielded tunnel boring machines (TBMs) are employed in tunnelling projects.

Two-component grout is obtained mixing component A with component B; first one is a cementitious mix made mainly of water, cement, bentonite, and retarding/fluidifying agent whilst component B is an accelerator agent (usually made with sodium silicate). The obtained mixture is called gelled grout because when the two components are mixed a gelation process occurs. Afterwards, the grout solidifies in few seconds (usually in the range of 5 – 15 seconds) and is named hardened grout.

To accomplish the research on the behaviour of two-component grout, a laboratory test campaign was carried out to assess the fluctuation of the characteristics of component A, gelled grout, and hardened grout as type of bentonite and time of activation change. Bleeding, unit weight, and viscosity were tested on Component A, whilst gel time was assessed on gelled grout; surface compressive strength was evaluated on hardened grout cured for 1 hour and 3 hours. These tests are commonly carried out in construction sites to verify the proper characteristics of batched grout, furthermore they are easy to perform and do not require any complex equipment. Finally, liquid limit and swell index were measured on bentonites to evaluate a correlation between its properties and characteristics of two-component grout.

This research study is a continuation of the PhD thesis of Todaro et al., 2021, where an extensive study of the properties of two-component grout in relation with activation time and three types of bentonites was performed.

In this laboratory test campaign, five types of bentonites were tested (two more respect Todaro et al., 2021) and named, for commercial reasons Type 1, Type 2, Type 3, Type 4, and Type 5. For each type of bentonite, four times of activation were used, and they are: 0 minutes, 2 minutes, 4 minutes, and 7 minutes. Therefore, twenty different mix designs of two-component grout were obtained by combining the types of bentonites and the times of activation.

The procedure used to prepare the component A, to assess the gel time, and to measure the surface compressive strength, are all innovative methods proposed in papers of Todaro et al., 2019 and 2020.

Hereinafter, general aspects of full-face mechanized tunnelling method using shielded machines are briefly discussed (2). Then, backfilling is introduced, discussing about its general aspects, backfilling injection methods and backfill materials employable (3). Chapter 4 is entirely dedicated to explaining what two-component grout is, how is obtained, how it works, what its components and its properties are, and the main differences with mono-component grout. The influence of bentonite in cementitious mixes is discussed in Chapter 5. Finally, laboratory test campaign and the obtained results are illustrated (6, 7, 8, 9).

## 2 FULL FACE MECHANIZED TUNNELING METHOD USING SHIELDED MACHINES

In full face mechanized tunnelling method, the excavation is carried out using full-face Tunnel Boring Machines (TBMs) that operate in a pseudo continuous way, where the excavation, stabilization, and mucking operations are performed by the machines themselves. The tunnel face is excavated on its entire surface and a circular shaped tunnel is obtained. According to the type and condition of the ground to excavate the machines are commonly divided into rock TBM and soil TBM (Bilotta et al., 2022).

### 2.1 Rock TBMs

In rock tunnel boring machines, the excavation is performed by a cutter head pressed against the tunnel face and that rotates along an axis coincident with that one of the tunnel. The cutter head is normally equipped with disc cutters (rolling cutters) which are able to detach rock chips thanks to the cooperation of adjacent cutters. Rock chips are formed by the intersection of cracks in the rock mass caused by intense tensile and shear stresses provided by disc cutters. Rock debris are then collected by buckets placed in the cutterhead and discharged on conveyor belt installed inside the machine. The muck is then discharged on tunnel transport conveyor belt or any other transport devices such as trains or trucks (EFNARC, 2005; Bilotta et al., 2022).

Rock TBMs can be classified in three main types: open (or gripper), single shield, and double shield TBM.

Open (or gripper) rock TBMs are employed in that rock masses characterized by stable conditions that allow to complete the operation of excavation without the necessity to

provide any support to the cavity or the face. A gripper system is installed in the machine to provide a lateral anchoring to the surrounding ground and to allow the thrusting of the cutterhead. The operative sequences are very similar to those employed in conventional method such as the installation of the first phase lining and of the final lining (if necessary) therefore no segmental lining neither backfilling is employed (Figure 2.1) (EFNARC, 2005; Bilotta et al., 2022).

In shielded TBM (both single and double shield) the excavation is performed under the protection of the shield, which provides temporary support of the cavity, and the final lining, which is made of segments, is directly installed under it (Bilotta et al., 2022).

In single shield TBM, the installation of the segmental lining is alternated with the excavation phase since the machine advances thanks to thrust jacks pushing against the side of the lining. This entails that the machine works in a cyclic way than a continuous one (Figure 2.1) (Bilotta et al., 2022).

In double shield TBM both thrust jack system and gripper system are installed in combination with a front, a telescopic, and a tail shield. This allows to perform the excavation and installation of segmental lining simultaneously (Bilotta et al., 2022). Gripper system allows the thrusting of the cutterhead whilst the segments are installed under the tail shield. The telescopic shield extends to cover the advancing of the machine connecting the front shield to the tail one. When the segments are installed, thrust jacks are pushed against the lining to advance the rear of the machine and the grippers are replaced (Figure 2.1).

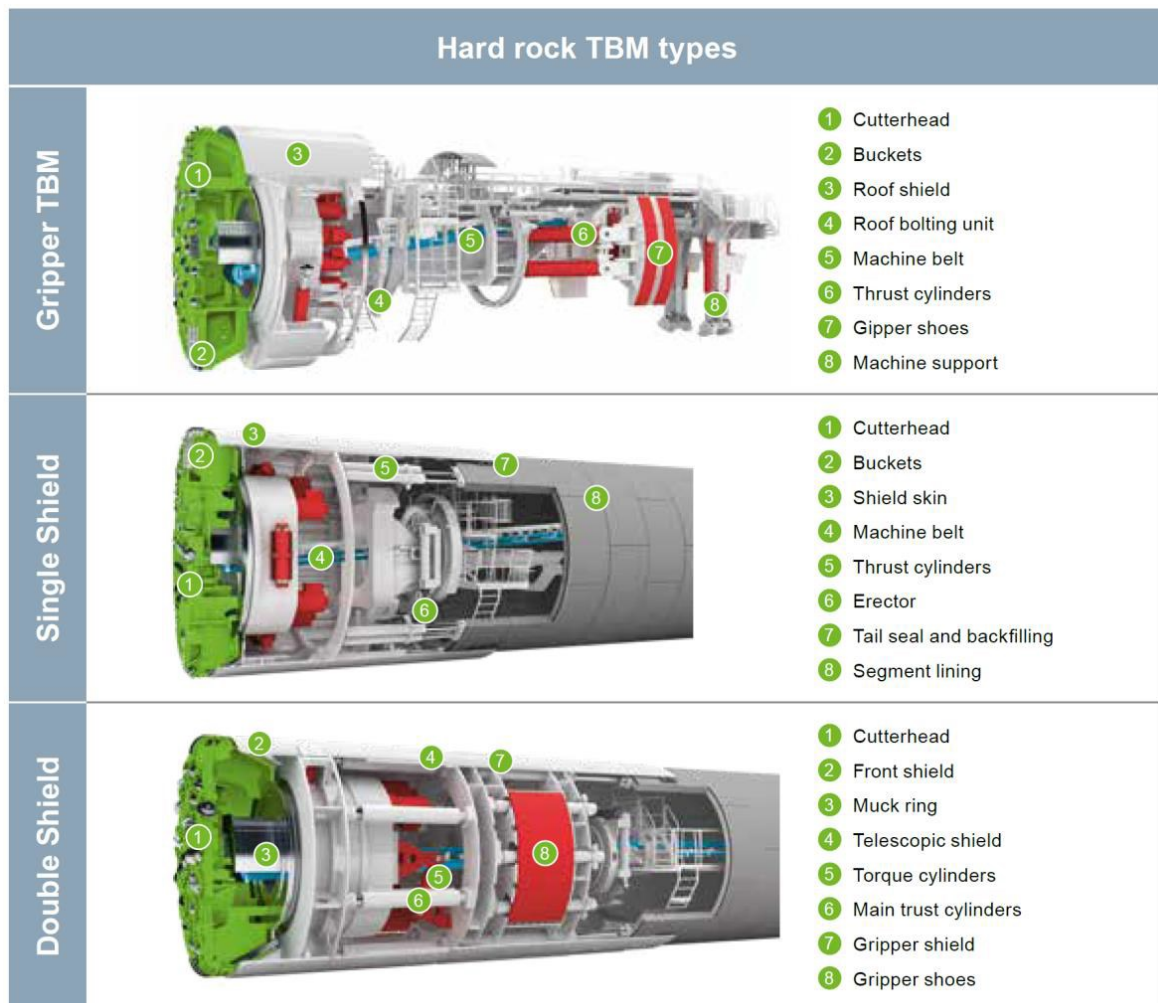


Figure 2.1: Schemes of rock TBMs: gripper (or open), single shield, and double shield TBM (Bilotta et al., 2022; website Herrenknecht AG).

## 2.2 Soil TBMs

Soil tunnel boring machines are employed in soil or in rock masses where it is necessary to provide stability to the tunnel face and cavity. Stability of the cavity is guaranteed by the shield whilst the stability of the face is provided by the face pressure applied in the excavation chamber of the machine (located behind the cutterhead) and obtained by a fluid or by the excavated soil properly conditioned. TBMs that provides stability to the face using a fluid are generally called slurry shield whilst the machines that condition the soils are named Earth Pressure Balance Tunnel Boring Machines (EPB TBMs) or EPB shields (Peila et al., 2011; Bilotta et al., 2022).

In slurry shield machines, the face stability is achieved by pumping bentonite slurry in the excavation chamber which is located behind the cutterhead. The machine is made with a rotating cutterhead equipped with cutter tools, such as scrapers, teeth or rippers, that detach the soil which enters in the excavation chambers, through large apertures in the cutterhead, from where is transported outside the tunnel with slurry by a pipe system. Slurry helps to maintain an even pressure at the tunnel face and also to transport the excavated soil. Final lining is installed under the shield and is made up of segments. The installation of the segmental lining is alternated with the excavation phase since the machine advances thanks to thrust jacks pushing against the side of the lining (Figure 2.2) (EFNARC, 2005; Peila et al., 2011; Bilotta et al., 2022).

In EPB machines, the face stability is provided by the excavated soil properly conditioned which is obtained by injecting chemical additives ahead of the cutterhead or into the excavation chamber or into the screw conveyor. Chemical additives change the characteristics of the soil leading to a pulpy and plastic behaviour which is able to provide an even pressure at the face. EPB machines are made with a rotating cutterhead equipped with cutter tools, such as scrapers or disc cutters according to the type of ground to excavate (soil or weak rock mass). The excavated soil enters in the excavation chamber where is collected by a screw conveyor and then discharged on tunnel transport conveyor belt or any other transport devices. EPB machines can work both in close and open mode. Open mode is used when the face is stable and consists of left the excavation chamber void and the operative sequences are similar to those ones of a single shield TBM. Close mode is used when the support of the face is required and consists of left the bulk chamber filled with excavated material to provide a proper support pressure at the tunnel face. The required support pressure is achieved by thrusting the cutterhead forward or by regulate

the screw conveyor rotation. Final lining is installed as for slurry shield under the protection of the shield (Figure 2.2) (EFNARC, 2005; Peila et al., 2011; Bilotta et al., 2022).

Variable density machines are multimode machines able to change the operation mode from a rock TBM mode, to an EPB TBM and to a slurry shield TBM mode in a continuous way (Figure 2.2) (Bilotta et al., 2022).

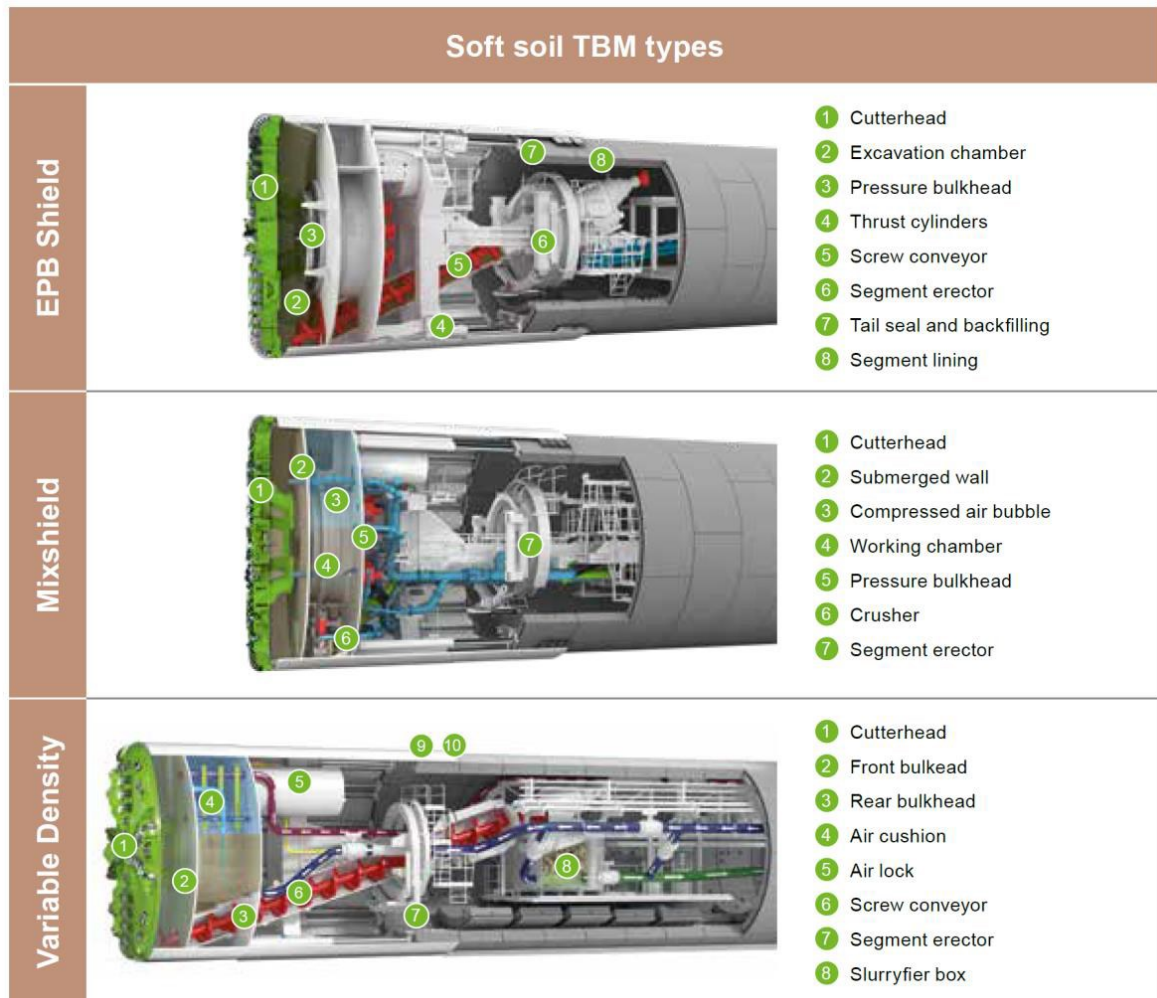


Figure 2.2: Schemes of soil TBMs: EPB shield, mixshield (or slurry shield), and variable density TBM (Bilotta et al., 2022; website Herrenknecht AG).





### 3 BACKFILLING GROUTING

#### 3.1 General aspects

During the construction of a tunnel, in full face mechanized method, a void is created after the advancement of shielded tunnel boring machines (TBMs) and the consequent setting of new prefabricated lining segments. The void is unavoidable, it is delimited by the outer diameter of the lining and by the surface of the excavated ground, and it is the result of over excavation, necessary to permit the advancement of the machine, conicity of the shield, thickness of the shield, and design of seal brushes. This annular void has to be continuously filled, and this fundamental operation is named backfilling (Figure 3.1; Figure 3.2) (EFNARC, 2005; Thewes et al., 2009; Peila et al., 2011).

The aim of backfilling is to prevent ground convergence, surface settlement, and to guarantee a symmetrical and homogeneous distribution of the loads along the segmental lining. In detail, backfilling operation is needed to (EFNARC, 2005; Peila et al., 2011; ITAtech, 2014):

- Prevent ground convergence and consequently surface settlement (especially in short-depth tunnel) ensuring a uniform, homogeneous, and immediate contact between the segmental lining and the excavated ground.
- Lock the segmental lining into position allowing the bearing of thrust forces, hoop stresses, and loads in general, transmitted by the TBM during its advancement and by the TBM back-up, furthermore, it allows the optimal transmission of these loads and stresses to the surrounding ground.
- Guarantee a symmetrical and homogeneous distribution of ground bearing loads along the segmental lining avoiding punctual loads.

- Collaborate with gaskets to prevent any water inflow by realizing a continuous waterproof layer.

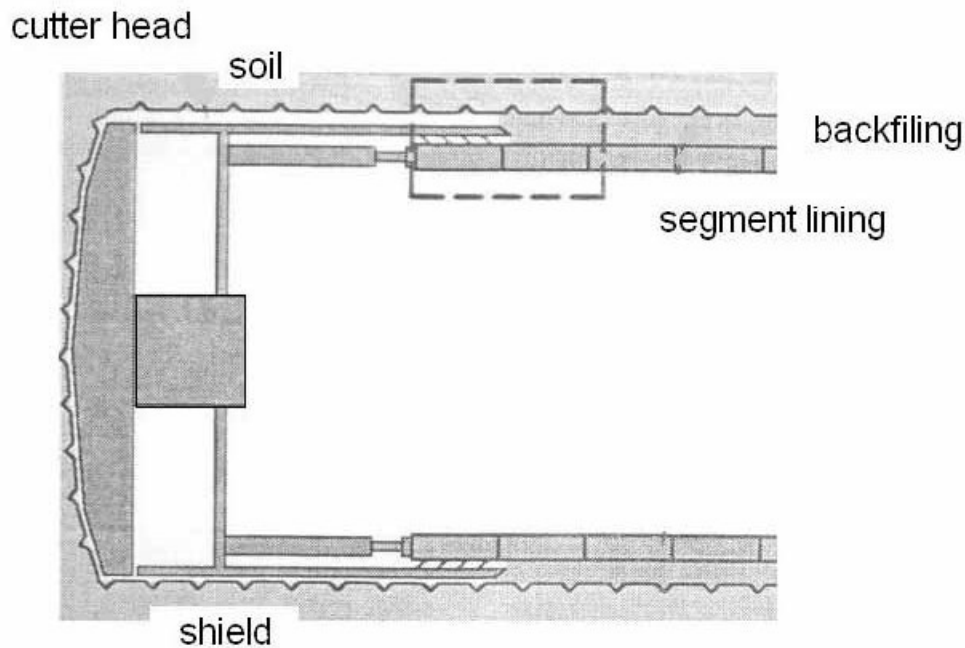


Figure 3.1: backfilling operation (Peila et al., 2011).

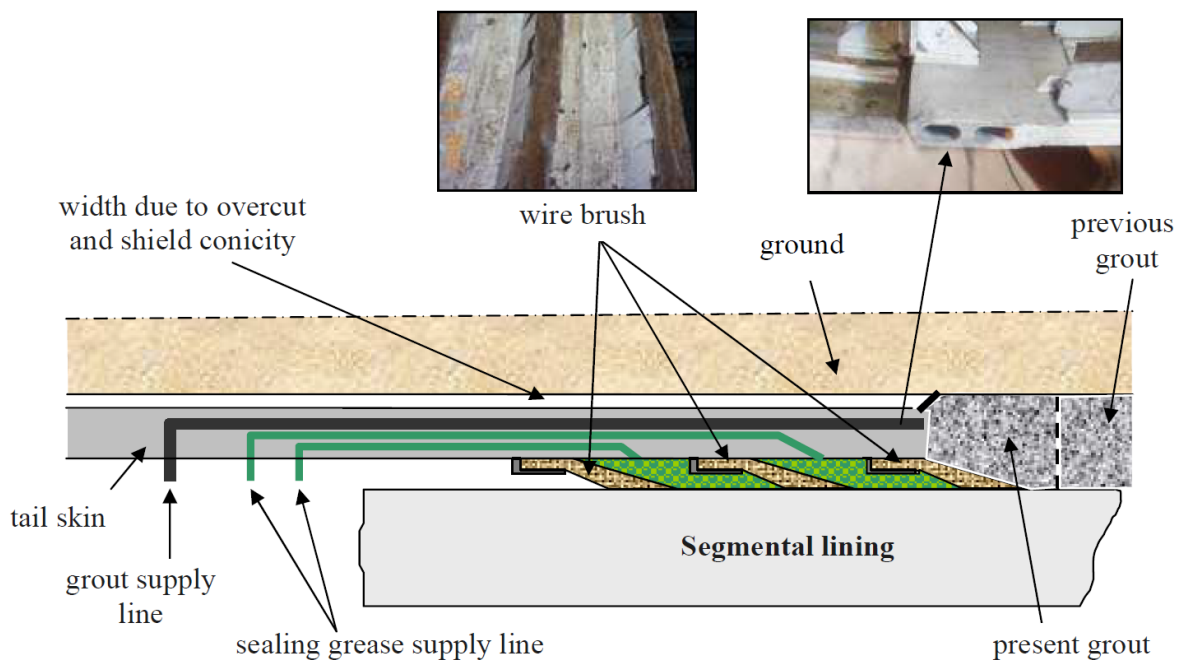


Figure 3.2: detail of backfilling operation. It is visible the annular void filled with a backfilling injection. The gap is given by the overcut, shield thickness, shield conicity, and dimension of seal brushes (or wire brushes) (Pelizza et al., 2010).

Wire brush sets (or seal brush sets) are installed on the inner side of the tail skin (the final section of the shield) with the aim to impede the enter of backfilling injection inside the working chamber of the machine. Grease fills the gap between wire brushes sets and it is fundamental to guarantee the efficiency and waterproof of the whole system. On the outer side of the tail skin, an excluder ring, typically in the form of fish scale seal, is mounted to avoid the overcoming of the backfilling injection over the shield and, consequently, the cementation of the shield to the surrounding ground (Figure 3.3) (ITAttech, 2014).

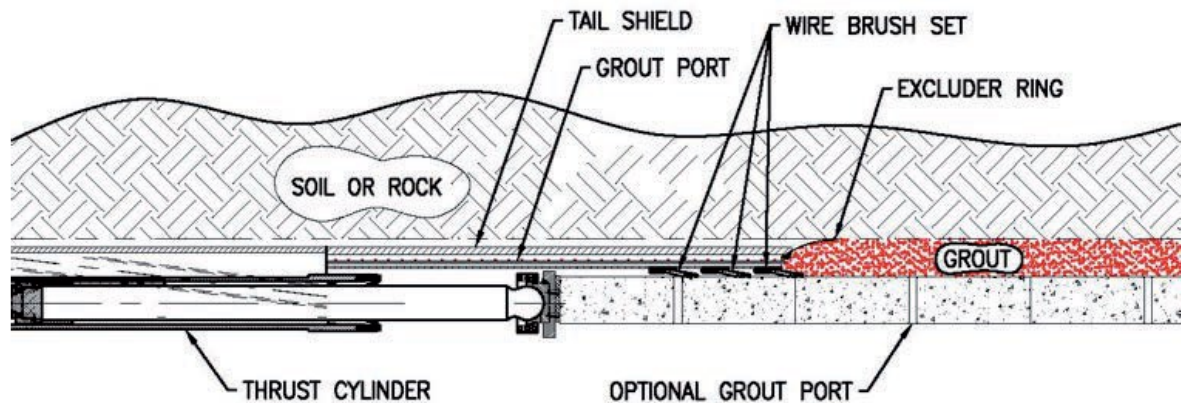


Figure 3.3: detail of wire brush sets and excluder ring in backfilling operation (ITAttech, 2014).

To perform a good backfilling operation and achieve the goals previously described, it is necessary that the annulus gap is completely and instantaneously filled as it is created reducing the ground displacement and surface settlement. Hence, the backfilling is usually injected from apertures in the tail skin. Furthermore, it is fundamental to create a homogenous and regular layer, also with respect to the physical and mechanical behaviour, and optimally linked with the surrounding ground. According to the environmental condition, the backfilling injection may be designed to set quickly providing a rapid support to the segmental lining, without causing clogging of the injection pipes, or to be resistant

to wash-out phenomena in presence of ground water flows (EFNARC, 2005; Peila et al., 2011).

Regarding logistic requirements, the material employed for backfilling should be designed to maintain its transportability, pumpability, and workability for a long period, usually up to 72 hours, avoiding clogging of the injection pipes during the normal stops of the TBM, and then, to permit a restarting of the backfilling operation at any time. Furthermore, during the transport phase from the batching plant to the injection point, phenomena such as bleeding and segregation should not take place (EFNARC, 2005; Peila et al., 2011).

### 3.2 Backfill injection methods

The injection of backfilling material inside the void occurs through ports located in the tail of the shield or through grout ports in the segmental lining. The choice of a method than the other one depends mainly on the backfilling material employed. Take in mind that tail skin injection is the only method which permit a completely and continuously filling of the gap and maximize the benefits of the backfilling operation (Thewes et al., 2009; ITAtech, 2014).

#### 3.2.1 BACKFILL INJECTION THROUGH PORTS IN THE SHIELD

Backfill injection through ports in the shield, also known as tail tube grouting, occurs at the end of the tail shield thanks to pipes that run in the thickness of the tail shield. This method permits a continuously filling of the void providing a good embedment and stabilization of the segmental lining reducing the risk of settlement and ground displacement.

To avoid the clogging of the injection tail tube, cementitious mixes like mono component grouts without aggregates, such as sand and gravel, and two-component grouts are employed (Thewes et al., 2009; ITAtech, 2014).

### 3.2.2 BACKFILL INJECTION THROUGH PORTS IN THE SEGMENT LINING

Backfill injection through ports in the segment lining occurs at a certain distance from the tail of the machine and this means the void is not filled as it is formed and could be remained unfilled for a longer time. This results in a higher risk of settlements, ground displacement, and segmental lining displacement. The backfill material is injected through predesigned and cemented grout sockets in the segment which are easily drilled after the setting of the segment. These apertures can be equipped with non-return valves or plugs to impede the leakage of the just injected backfilling. This method allows the injection of cementitious mixes, also with aggregates, and pea gravel since, due to the large dimension of the apertures, there is no risk of clogging.

### 3.3 Backfill material

Several materials can be employed in backfilling operation to fulfil the previously described requirements. They can be categorised in three main groups: mono component grout, inert mix, and two-component grout, each one with different properties, field of application, and required equipment (Table 3.1).

#### 3.3.1 MONO COMPONENT GROUT

Mono component grout is a cementitious mix made up of water, cement, bentonite, fly ashes, aggregates (such as sand and gravel), and chemical agents such as retarder, fluidifier, to improve its technological properties like the water/binder ratio, the mechanical strength, the initial and final setting time, and the pumpability. The mix should be fluid at least 3 - 4 hours to avoid clogging of the pipes due to stop of the machine during its normal working phase.

The mix is commonly batched outside the tunnel and transported to the machine with grout cars and finally injected through ports in the tail skin allowing to fill completely and continuously the created void providing a good embedment of the segmental lining and reducing the settlements. Injection through ports in the segmental lining is also possible, especially if aggregates are employed, such as sand and gravel, to reduce the risk of clogging of the tail-skin pipes. On the other hand, the injection through the segmental lining does not provide the benefits of a tail-skin injection (EFNARC, 2005; Thewes et al., 2009; Peila et al., 2011; ITAtech, 2014).

Mono component grouts are characterised by a slow set time (due to the hydration process of the cement), which involves a delay in the ability to support the lining and transmit efficiently the loads and stresses derived from the machine and the ground, on the other hand, once their set is complete, the mechanical strength can reach high values in the range of 15 - 20 MPa at 28 days. Mono component grouts are not applicable when high groundwater flow is present due to the dilution of the grout before it could reach the hardening phase (EFNARC, 2005; Thewes et al., 2009; Peila et al., 2011; ITAtech, 2014).

Mono component grouts can be classified into three categories: active systems, semi-inert systems, and inert systems (EFNARC, 2005; Peila et al., 2011).

- Active systems are characterized by a full hydration of binder component and have a cement content over 200 kg/m<sup>3</sup>.
- Semi-inert systems are characterized by a partial hydration of binder component and have a fraction of cement varying from 50 up to 100 kg/m<sup>3</sup>.
- Inert systems do not contain any cement and belong to inert mix (3.3.2).

Mono component grouts are usually delivered using piston pumps, which are able to regulate the injected volume thanks to the pace of the piston. Double piston pumps are usually installed in TBMs due to their compact design (Thewes et al., 2009).

### 3.3.2 INERT MIX

Inert mixes are characterised by the absence of any binding agent such as cement, avoiding any risk of clogging of the injection system, and they are made up mainly by sand, with adding of filler and fly ashes, transported in water. To reduce irregularities and heterogeneities of the mix, the sand is properly selected and graded. The injection happens through ports in the segmental lining due to the risk of clogging in the ports of the tail skin (Peila et al., 2011).

#### 3.3.2.1 *Pea gravel*

Backfilling using pea gravel (or a mix with sand and pea gravel) is applied in rock masses excavated with double or single shield TBM. Pea gravel has a diameter varying from 8 up to 12 mm and is obtained by washed and screened alluvial pebbles or crushed stone. To avoid any damage to the wire brush sets, the injection occurs from ports in the segmental lining at an appropriate distance from the tail of the machine to prevent the contact between the pea gravel and the seal brush sets. The injection of pea gravel follows its natural angle of repose, hence, only a partial filling of the annular gap is performed leaving an unfilled void at the top. This unfilled void, which can last for a long period of time, is usually filled with a secondary injection operation of a mono component grout through ports in the segment lining. The pea gravel works greatly as drainage layer in presence of flows towards the tunnel (Thewes et al., 2009; Peila et al., 2011; ITAtech, 2014).

Pea gravel is injected using compressed air, and powerful compressors are required to guarantee the spreading of the material and the filling of the annular gap (Thewes et al., 2009).

### 3.3.3 TWO-COMPONENT GROUT

Two-component grout is explained in 4.

Table 3.1: main field of application and pumping system required for various type of backfilling (redrawn from Thewes et al., 2009 and Peila et al., 2011).

Backfill material		Application range		Backfilling system		Required equipment			
		Hard rock	Soil	A	B	a	b	c	d
Mono component grout	Active system	X	X	X	X	X			
	Semi-active system	X	X		X	X			
Inert mix			X		X	X			
Pea gravel		X		X					X
Two-component grout			X	X	X		(X)	X	

*X applicable; (X) limited applicability.*

*A: backfilling through ports in the segmental lining, B: backfilling through ports in the tail skin.*

*a: piston pump; b: peristaltic pump; c: progressive cavity pump; d: pressurized air.*



## 4 TWO-COMPONENT GROUT

Two-component grouts were employed for the first time in 1982 in the construction of the Osaka Subway in Japan, achieving good results regarding the control of settlement. Since then, the two-component grouts were largely used in Japan and later in the rest of the world, especially in America, Asia, and Europe (Hashimoto et al., 2006; Reschke et al., 2011). Two-component grouts consist of a component A and a component B mixed a moment before the injection, at the nozzle, which harden in few seconds (normally 5-15 seconds) providing a rapid and uniform stabilization of the annular gap (Hashimoto et al., 2006; Peila et al., 2011). When component A is mixed with component B, a semi-solid plastic gel is obtained, and this operation is called gelation. This gelled grout maintains this property for about half an hour before to harden and set (gel time, harden time, and set time can be changed by acting on the mix design and additive agents) (Figure 4.1) (Hashimoto et al., 2006). Component A is made up of water, cement, bentonite, eventually with adding of fly ashes, and chemical agents such as retarder, fluidifier, etc., to extend the workability, stability, and pumpability. Component B, instead, is an accelerating agent, usually made with sodium silicate (Hashimoto et al., 2006; Reschke et al., 2011; ITAtech, 2014).

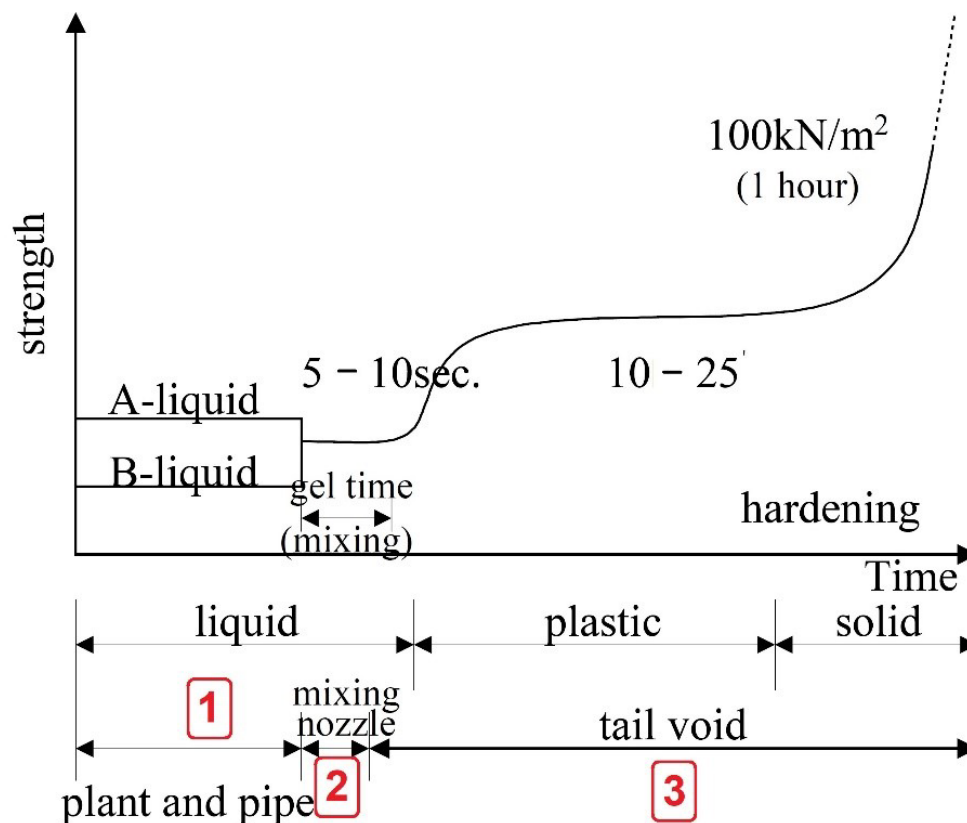


Figure 4.1: behaviour of two-component grout when is injected over time. It is illustrated also the relation with its mechanical strength (redrawn from Hashimoto et al., 2006).

#### 4.1 Injection method

Component A is transported from the batching grout plant to the TBM via a pipe system and injected under pressure throughout the annular gap through pipes embedded inside the tail shield. At a certain distance from the injection port, component B is added and mixed along this length thanks to a turbulence phenomenon. As the grout is injected, the TBM (or only the tail shield) advances leaving a void which is immediately filled. Component B is usually transported via pipe system too. Usually, in the last 100 mm of advance of the tail shield, only component A is injected avoiding chocking of the ends of the pipes due to gelation of two components. A high-pressure water flushing system is present to remove the possible volumes of gelled grout in the pipe system. Furthermore, to minimize the risk

of choking a proper mix design and specifically designed equipment should be chosen, also a normal cleaning regime and regular maintenance procedures should be established (Figure 4.2) (Hashimoto et al., 2006; Peila et al., 2011; Reschke et al., 2011; ITAtech, 2014).

Injection ports are usually evenly distributed on tail shield perimeter with addition also of spare ports; their number depends on a variety of factors such as the type of grout, advancing rates, machine cutterhead diameter, type of ground to excavate, and dimension of the grouting ports. Each grout port should have an independent pump to control the injection volume and pressure, and to prevent clogging (Figure 4.3) (Peila et al., 2011; Reschke et al., 2011; ITAtech, 2014).

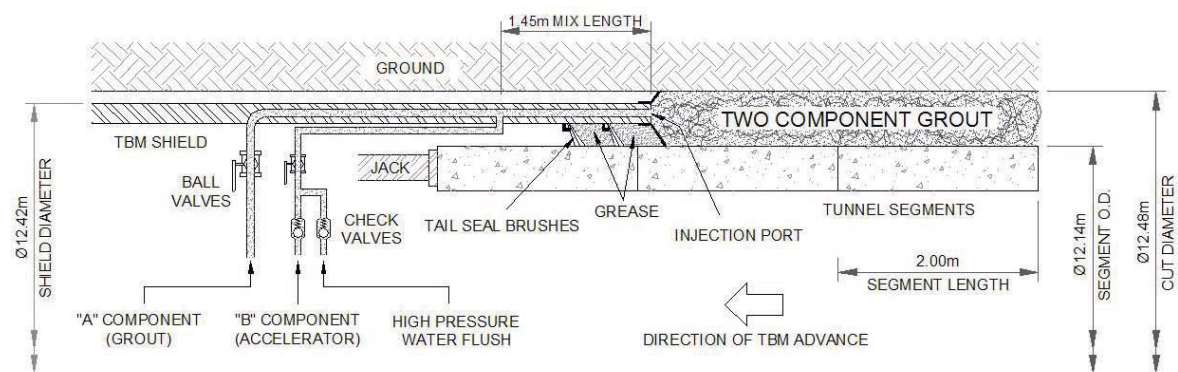


Figure 4.2: detail of a two-component injection system. A Herrenknecht EPB machine with incorporated annular two-component type grouting through the tailskin is shown. This machine was used in Brisbane's Airport Link Project, Australia (Reschke et al., 2011).

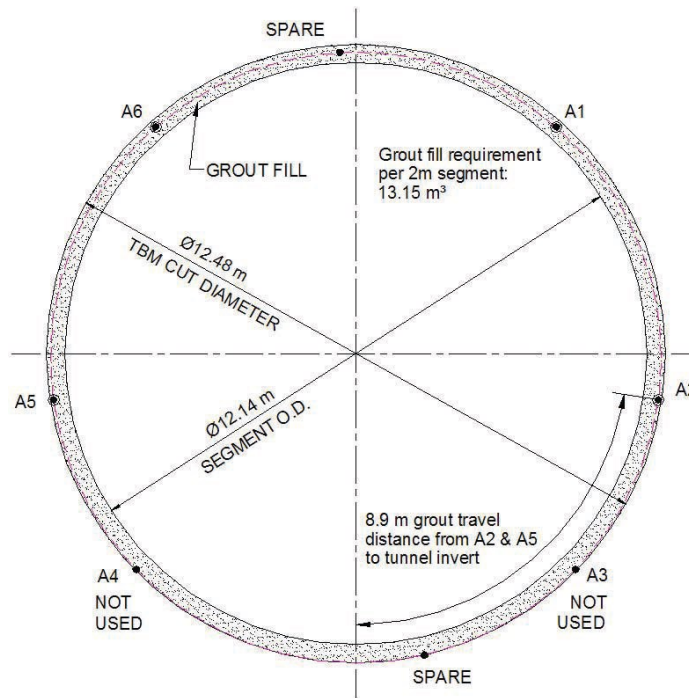


Figure 4.3: detail of grouting ports embedded in the tail shield. A Herrenknecht EPB machine with incorporated annular two-component type grouting through the tailskin is shown. This machine was used in Brisbane’s Airport Link Project, Australia (Reschke et al., 2011).

This system requires a certain degree of sophistication since the pressure of both components must be maintained constant and sufficient to achieve a complete filling of the annular gap and penetration into any voids of the surrounding ground. Two-component grout is able to penetrate also into the ground, according to ground permeability. Instead, an excess of pressure could cause leakage throw tail seals (Peila et al., 2011; Reschke et al., 2011; ITAtech, 2014).

Volume of both components must be also controlled to maintain the correct mix proportion between A and B. The injected volume could be bigger than the theoretical volume, i.e., presence of a void in the surrounding ground, therefore, the batched volume should be always greater than theoretical one without leaving an unfilled portion of the

annular gap (the crown) which will not be able to support any loads and leading to potential consequences (Peila et al., 2011; Reschke et al., 2011).

#### 4.2 Uncompressible annular bubble

Two-component grout has to be considered as an uncompressible fluid since its mainly made up of water (which is incompressible) and because of the accelerator which give its thixotropic consistency. Consequently, the annular gap is a closed annular bubble which is continuously filled with an uncompressible fluid as the tail shield advances. To proper create this uncompressible annular bubble, the grout should gel quickly, but not instantaneously, otherwise there will be a lack of homogeneity (Peila et al., 2011).

If a displacement of the surrounding ground or a movement of the lining happens, causing a pressure or a force acting on the bubble, this last reacts with an evenly distributed pressure along its surfaces and volume avoiding any type of deformation and any type of punctual loads. To achieve this behaviour it is necessary to follow the following conditions (Peila et al., 2011):

- Injected grout must remain uncompressible.
- Grout should not permeate through the surrounding ground or leakage through lining gaskets or through the space between the outer surface of the tail shield and the excavated ground (that is avoided thanks to tunnel face pressure or to the excluder ring (ITAtch, 2014)).
- The convergence of the surrounding ground should be controlled since an excessive displacement towards the bubble could cause an increase of the pressure required to move forward the TBM. This condition is usually balanced with tunnel face pressure and injection of bentonite slurry on the outer surface of the tail shield.

- The segmental lining should be properly installed avoiding any deformation of segments due to their own weight.
- The injected grout should not be diluted by underground water.

#### 4.3 Durability and tests on injected two-component grout

All the hydrostatic and geostatic loads are borne by the segmental lining and the incompressible annular bubble does not provide any structural contribution inasmuch its purposes are to mitigate external loads and pressures, to avoid any formation of point loads, to block the segmental ring in its projected position, and to avoid deformations due to the TBM thrust and cutting-wheel rotation. Because of this, two-component grout should not be affected by degradation to permanently guarantee the properties of the annular bubble. Durability is guaranteed in normal humidity conditions of the ground where no water is lost due to evaporation or filtration maintaining stable and undeformed the backfilled material. Moreover, permeability of the ground lower than  $10^{-8}$  m/s assures no loss of water through the ground (Peila et al., 2011).

Mechanical strength of grout is regularly measured over time (usually at 0.5 h, 8 h, and over 24 hours) to assess the proper gelation of two-component grout, and consequently the proper creation of the incompressible annular bubble, and to observe if any decomposing phenomenon have occurred. The measures can be carried out using, i.e., a pocket penetrometer. The most reliable and significant way to measure the strength and check the filling of the annular gap is the direct way, by extracting core samples through the lining (the extraction happens when the grout reaches a sufficient strength to allow the coring operation) (Peila et al., 2011).

#### 4.4 Elements of two-component grout

Cement is commonly a Portland Type I with a high mechanical strength, usually in the range 42.5-52.5 R, due to the absence of aggregates.

Water is commonly provided by local aquifer; however, its chemical and physical properties should be always checked because could degrade two-component grout.

Bentonite reduces bleeding phenomena and permeability of component A (see Chapter 5).

Accelerator agent (or component B) causes the gelation of component A in few seconds (usually 5-15 seconds) which starts to develop mechanical strength almost instantaneously, furthermore, the obtained gel is homogeneous with a thixotropic consistency and hence avoids point loading phenomena over segmental lining (Peila et al., 2011).

Retarding agent has the purpose to inhibit the setting of component A and extends its workability up to 72 hours after batching. This is useful during stops of machine or in case of unforeseen working delays with the possibility to stockpile and use at a later time (Peila et al., 2011).

#### 4.5 Batching grout plant

The equipment used to mix and place the grout directly influences its properties and quality, furthermore, the capacity of the batching grout plant and of storage tanks should be sufficient to grout a complete ring without stopping and should be designed according to the TBM advance rate and segment erection rate (EFNARC, 2005).

Component A is commonly batched using a lower shear paddle mixer or a high shear colloidal mixer, the latter is widely recognised as the most efficient method of mixing cement-based grouts because of reduced bleeding and increased strength. The colloidal mill is characterized by a discar which spins over 2000 rpm to cause a violent turbulence

and high shearing action to break down clusters of dry cement particles. All the elements are rapidly mixed inside the tank thanks to the vortex action obtaining a grout which exhibits colloidal properties such as minimal settling and bleeding (EFNARC, 2005; Reschke et al., 2011).

Water is weighed using a fast feed piping system followed by a slow feed system to achieve a high weigh accuracy (accuracy  $\pm 1\%$  of its designed weight). Before to add the other elements, the mixer is cleaned using this water which circulates in the system. After that, the stabiliser agent is weighted via a diaphragm pump and measured by a flow meter (accuracy  $\pm 5\%$  of its designed weight). Bentonite and cement, instead, are accurately weighted controlling the frequency drives of screw conveyors via fast then slow feed rates (accuracy  $\pm 2\%$  of their designed weights) (EFNARC, 2005; Reschke et al., 2011).

The mixing time shall be as short as possible consisting with complete mixing of the ingredients. Bentonite is added first and mixed for the desired amount of time (i.e., 30 seconds – 7 minutes) before the cement and later the additive agents are added. Mixing continues for an additional minute after that the batched grout is transferred to an agitation tank. Batched component A is pumped via a pipe system to a tank on the TBM. Component B is also pumped via a pipe system to another tank on the TBM. Pressure in both lines is constantly monitored especially in that for component A to avoid clogging of the pipe system due to segregation of cement (EFNARC, 2005; Reschke et al., 2011).



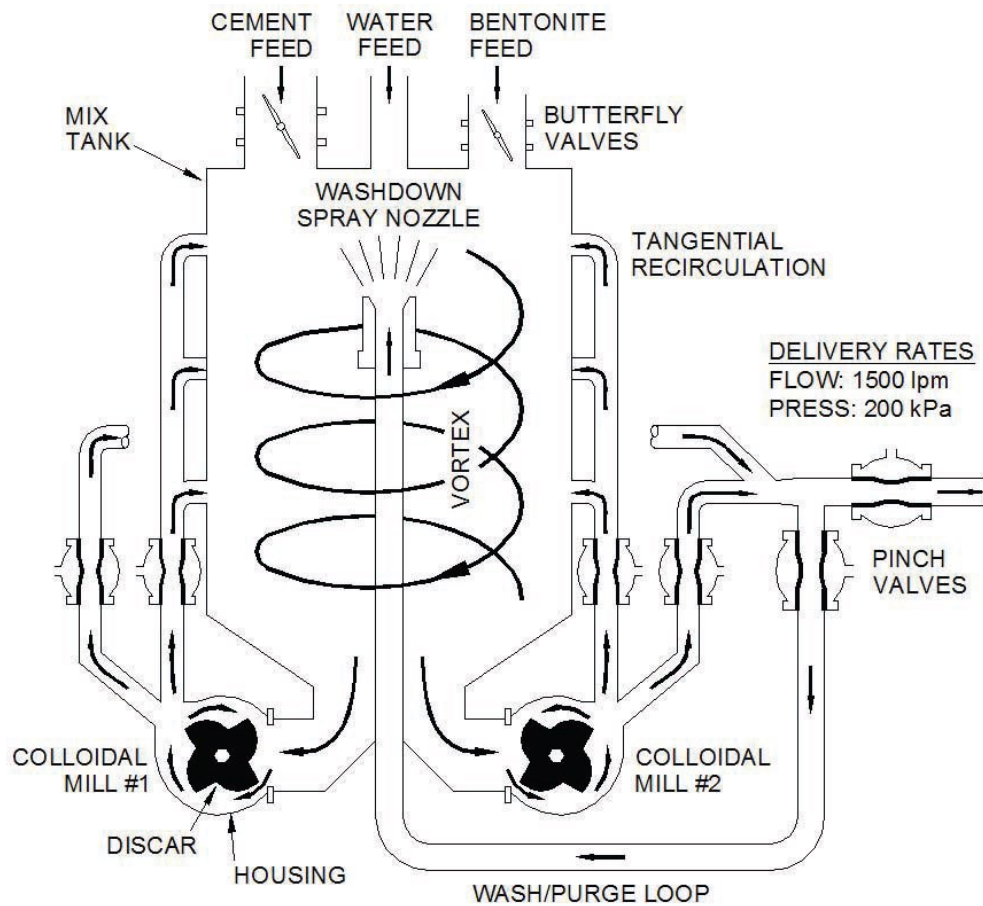


Figure 4.4: a high energy colloidal mixer. Here is shown the colloidal mixer used in Brisbane's Airport Link Project, Australia (Reschke et al., 2011).

#### 4.6 Pumping equipment

Progressive cavity pumps and peristaltic pumps are commonly employed to transport the liquid components of two-component grout. The first ones are positive displacement pumps and are made with a horizontal spiral within a tube where a little space permits the transportation of the material due to the friction at the spiral blade. This kind of pump permits a continuous flow of the material. Peristaltic pumps are positive displacement pumps and are made with a flexible tube which is compressed by the rotation of rollers or wipers mounted on the rotor. This compression causes a vacuum inside the flexible tube achieving the flowing of the material (EFNARC, 2005; Thewes et al., 2009).

## 4.7 Comparison between mono component grout and two-component grout

Hereafter the main differences between a mono component grout and a two-component grout are summarized (Reschke et al., 2011; ITAtech, 2014) (Table 4.1).

### 4.7.1 STRENGTH

Mono component grout shows a higher mechanical strength thanks to the higher set up time, which allows a more homogeneous and regular harden, and to the presence of aggregates in the admixture. The desired strength can be easily designed for single component grout by modifying the components of the mix. Actually, final strength is not a leading parameter since it is usually required that grout develops a sufficient strength to support the segmental lining and bearing the loads from the surrounding grounds and from the TBM.

### 4.7.2 COST

Mono component grout, generally, has a lower cost than a two-component grout. Other parameters to take into account are the volume of grout needed and thrown out which could lead towards the choice of two-component grout. The chosen method of injection influences the volume to fill, injection through segmental lining required a lower bore diameter than injection through the tail skin, thus reducing the volume of grout needed, on the other hand, the risk of settlement is increased.

### 4.7.3 GROUND WATER EFFECTS

In presence of groundwater flows, dilution of the grout can occur in mono component grouts whilst two-component grouts are not affected due to their rapid setting time. Furthermore, two-component grout has low permeability because of its content of

bentonite, providing an effective waterproofing of the tunnel. To improve the resistance of mono component grout it is possible to add anti-washout agents.

#### 4.7.4 EARLY SUPPORT OF LINING

Thanks to the quick setting time of two-component grout, ground convergence and settlement are minimised, furthermore, successfully results can be assessed also in difficult ground conditions and sensitive locations. Two-component grout guarantees the lock in place of the segments as they are set, avoiding the displacement and deformation of the segmental lining. The grout reaches a high early strength which is able to resist loads and stresses coming from the ground, from thrusting the machine forward, and from moving the TBM back-up. This condition leads to a reduction of maintenance costs and repair works. The slow set time of mono component grouts, caused by hydration phenomena of cement, does not produce the previously described benefits and moving or distortion of lining can occur.

#### 4.7.5 FLUIDITY/PUMPABILITY AND TRANSPORTATION

Mono component grout has a lower fluidity and its transportability and pumpability results more difficult than component A and component B. Component A and B can be easily pumped for long distances, from the batching grout plant to the injection point, whilst mono component grout is commonly transported by grout cars. Mono-component grout requires high pressure concrete type pumps which can lead to detrimental effects on surrounding ground if excessive pressure is used. Smooth flow, positive displacement pumps can be used to have a good control of pressure in two-component grout thanks to its low viscosity.

#### 4.7.6 BATCHING

The batching of mono component grout and component A can be done in automated grout plants obtaining good mixtures.

#### 4.7.7 MAINTENANCE

Component A is designed to remain fluid (usually up to 72 hours) and pumpable until it is mixed with component B (which is not affected by solidification phenomena) at the nozzle. This reduces the risk of clogging of the injection system and to waste grout, especially during stops of the machine or unforeseen delays. Mono component grout, instead, remains fluid for a shorter time with the possibility to set in the injection pipes causing clogs and their resulting substitution.

Table 4.1: comparing chart for mono component grout and two-component grout (redrawn from ITAttech, 2014).

Consideration	Mono component grout	Two-component grout
Strength	X	
Cost	X	
Groundwater resistance		X
Early set time		X
Early support of lining		X
Fluidity/pumpability		X
Transportation		X
Batching		X
Maintenance		X

## 5 BENTONITE

Bentonite is a clay mineral originated by alteration process of volcanic ashes and characterized by a high percentage of montmorillonite and a high plasticity (plasticity index goes from 50% up to 100% and the liquid limit goes from 300% up to 700%) (Lancellotta, 2012). It may be associated with minor amounts of other minerals such as quartz, mica, feldspar, calcite, etc. (EFNARC, 2005). Bentonites are generally grouped into sodium bentonite, calcium bentonite, and calcium-sodium bentonite. The first one is mostly used in cement-based grouts because of its higher swelling capacity when dispersed in water (its swelled volume is 10-25 times the original dry volume). The other two types have a low and medium swelling capacity respectively. Bentonite particles, which have a plane-rectangular shape, adsorb water on their surfaces therefore stabilising the grout and slow down the sedimentation of cement. Calcium bentonite can be chemically treated to obtain an activated sodium grade (Garshol, 2003; EFNARC, 2005; Mesboua et al., 2018).

### 5.1 Clay minerals

Clay minerals are hydrous aluminium silicates belonging to phyllosilicate, a subclass of the silicate class according to the Nickel-Strunz classification. This classification categorizes all minerals according to their chemical composition and crystal structure into ten main classes which are subdivided into divisions, families, and groups. A simplified scheme is reported in Table 5.1. The definition of mineral is given by the International Mineralogical Association: “A mineral is an element or chemical compound that is normally crystalline and that has been formed as a result of geological processes” (Nickel, 1995).

Table 5.1: A simplified scheme of the Nickel-Strunz classification.

Class	Subclass	
1		Native elements
2		Sulfides and Sulfosalts
3		Halogenides
4		Oxides
5		Carbonates and Nitrates
6		Borates
7		Sulfates, Selenates, Tellurates
8		Phosphates, Arsenates, Vanadates
9		Silicates and Germanates
	9.A	<i>Nesosilicates</i>
	9.B	<i>Sorosilicates</i>
	9.C	<i>Cyclosilicates</i>
	9.D	<i>Inosilicates</i>
	9.E	<i>Phyllosilicates</i>
	9.F	<i>Tectosilicates without zeolitic H<sub>2</sub>O</i>
	9.G	<i>Tectosilicates with zeolitic H<sub>2</sub>O; zeolite family</i>
	9.H	<i>Unclassified silicates</i>
	9.J	<i>Germanates</i>
10		Organic Compounds

Clay minerals (to not be confused with clay which states particles with size lower than 2  $\mu\text{m}$ ) are hydrous aluminium phyllosilicates, having a lamellar shape and a colloidal dimension. The structure of clay minerals is characterised by overlapped two-dimensional lattice sheets belonging to one of the two fundamental units: tetrahedral or octahedral unit (Lancellotta, 2012).

In tetrahedral unit, silicon is in the centre and is bound to four oxygen ions placed to the vertices. Tetrahedral units are arranged in hexagonal lattice sheets thanks to bonds between oxygen ions (Figure 5.1) (Lancellotta, 2012).

In octahedral unit, aluminium ion (or magnesium ion) is in the centre and is bound to six hydroxyl ions. Octahedral units are arranged in lattice sheets, with their axis inclined of  $45^\circ$  to the sheet, forming two levels made of hydroxyl ions and a level made of aluminium ion (Figure 5.2) (Lancellotta, 2012).

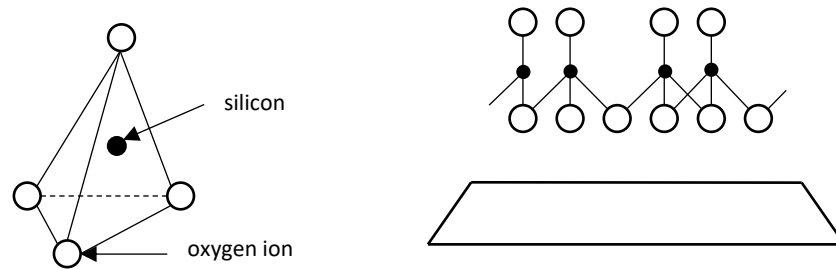


Figure 5.1: tetrahedral unit (left) and its symbolic representation (right).

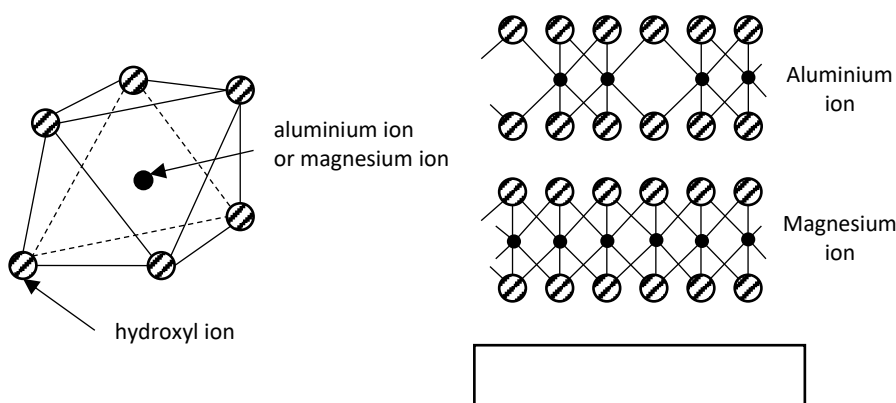


Figure 5.2: octahedral unit (left) and its symbolic representation (right).

Fundamental units are joined by strong bonds, as ionic bonds, to form fundamental packets; fundamental packets are connected by weak bonds, as weak ionic bonds and hydrogen bonds, to form clay particles (Lancellotta, 2012).

Kaolinite, which is one of the most common clay minerals in nature, consists of tetrahedral sheets alternated by octahedral sheets. Montmorillonite, which is the most common mineral in Smectite group, consists of an octahedral sheet placed between two tetrahedral sheets. Interstitial spaces are filled by absorbed water molecules bonded by sodium or calcium cations. Water molecules can double cause the swelling phenomenon of montmorillonite. Illite has a structure similar to that one of montmorillonite where fundamental packets are bound by potassium atoms, however it is not affected by swelling phenomena (Figure 5.3) (Lancellotta, 2012).

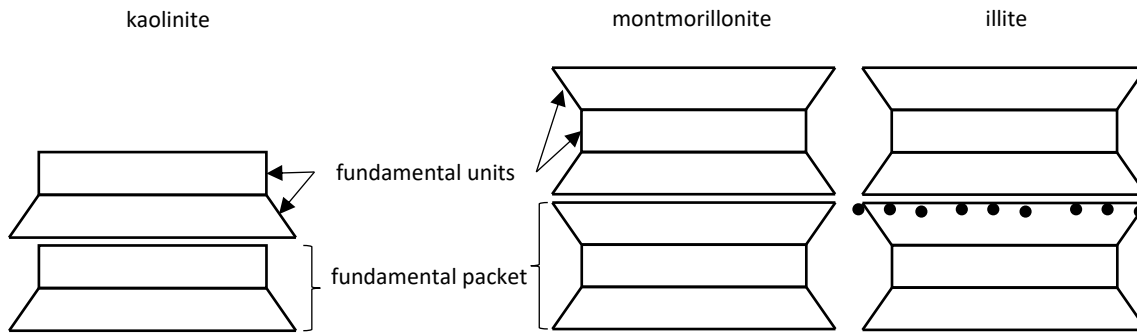


Figure 5.3: symbolic representation of kaolinite (left), montmorillonite (centre) and illite (right).

Clay minerals are characterized by lattice sheets where bond forces between packets are weak therefore particles are flattened and characterised by a high specific surface. Kaolinite has a specific surface in range of 10-20 m<sup>2</sup>/g whilst in montmorillonite it is up to 800 m<sup>2</sup>/g (Lancellotta, 2012).

## 5.2 Influence of bentonite in cementitious mixes

The commercial importance of bentonite employed in engineering field depends more on its physic-chemical properties rather than its chemical composition. Addition of bentonite in cement-based grout leads to the increase of the viscosity, a change of the rheological parameters such as yield stress and plastic viscosity (bentonite has a thixotropic behaviour) and reduce the bleeding (Mesboua et al., 2018).

The addition of bentonite causes a reduction of the fluidity of cement grout due to the high specific surface of bentonite and to its exceptional structure. Clay particles tends to absorb water on their surface reducing the lubrication of particles, whilst montmorillonite particles have a plane-rectangular shape forming structures similar to playing cards and named “card-house” and “card pack” where their development depends mainly on chemical properties of bentonite and its pH (Figure 5.4) (Mesboua et al., 2018).



Bleeding in cementitious mix can be reduced by adding bentonite, which absorbs water, and reduces segregation of cement thanks to its structure which embeds cement particles. 3-5% of Bentonite, related to the cement weight, provides a good stabilising effect on the grout (Figure 5.5). Furthermore, bleeding can reduce mobility and pumpability of the grout due to segregation of cement (Garshol, 2003; Mesboua et al., 2018).

Bentonite increases homogeneity and waterproof of cementitious mix (less than  $10^{-8}$  m/s), helping to achieve a thixotropic consistency and in the gelling process for two-component grout (Peila et al., 2011).

Compressive strength of cement grouts decreases slightly with increasing of bentonite content (Mesboua et al., 2018).

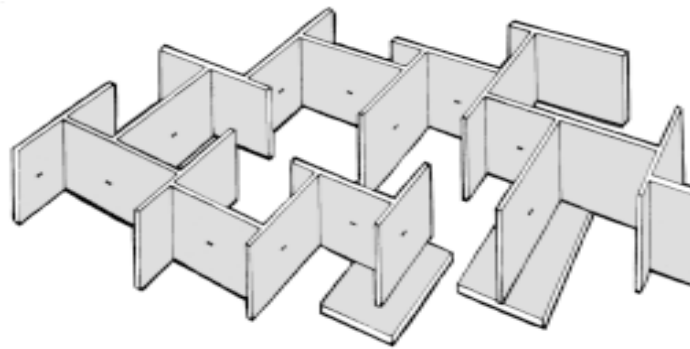


Figure 5.4: plane-rectangular shape of clay particles forming structures similar to playing cards and named “card-house” or “card pack” (Garshol, 2003).

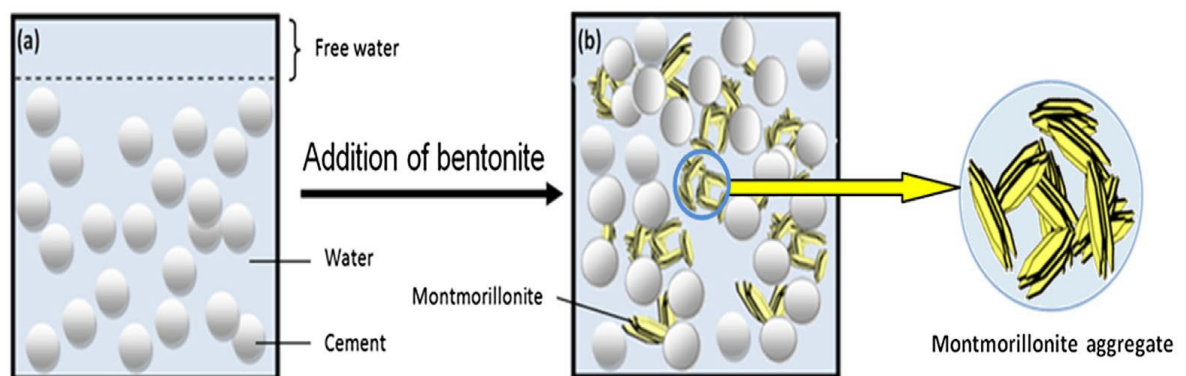


Figure 5.5: bleeding phenomena: segregation of cement without bentonite (left) and with bentonite (right) (Mesboua et al., 2018).



## 6 LABORATORY TEST CAMPAIGN

Laboratory test campaign is based on the analysis of the properties of two-component grout as type of bentonite and activation time change. Tests were performed on the component A such as bleeding, unit weight, and viscosity; on the gelled grout such as gel time; on the hardened grout such as the surface compressive strength, hereinafter SCS; and on the bentonite such as liquid limit, moisture content, and swell index. All above-mentioned tests, except for tests on bentonite, are commonly carried out on construction field to assess regularly two-component grout properties therefore these tests have a close operational nature.

Five types of bentonites and four times of activation were selected and by combination, twenty different mix designs were obtained (Table 6.1). The five types of bentonites are named as follow for commercial reasons: type 1, type 2, type 3, type 4, and type 5, whilst the times of activation are: 0 minutes, 2 minutes, 4 minutes, and 7 minutes.

Table 6.1: mix designs obtained by combination of bentonite type and activation time.

		Time of activation (minutes)			
		0	2	4	7
Type of bentonite	Type 1	mix 1	... 2	... 3	... 4
	Type 2	... 5	... 6	... 7	... 8
	Type 3	... 9	... 10	... 11	... 12
	Type 4	... 13	... 14	... 15	... 16
	Type 5	... 17	... 18	... 19	... 20

Two-component grout is obtained by mixing component A and component B obtaining a gelled grout. This process is called gelation and has an order of magnitude of few seconds, generally varying from 5 up to 15 seconds, and it is requested a certain degree of dexterity to correctly cast the grout samples.

Component A is prepared following a laboratory scale procedure proposed in the paper of Todaro et al., 2019, which emulates the batching grout plants in construction site.

Laboratory test campaign was performed from late-October 2021 to early-March 2022, and all tests, casting of samples, and curing of samples were carried out at room temperature and room humidity even though some fluctuations of these parameters occurred. To prepare component A, tap water was normally employed until mid-December 2021 when its temperature excessively cooled down, thus water was first stored in a tank to reach a thermal balance with room temperature (about 19 degrees) before to utilise it.

It was not possible to carry out all the tests of a mix design (bleeding, unit weight, viscosity, gel time, 1h SCS, and 3h SCS) with the same batched grout due to limitation of the maximum volume (3 litres) that can be prepared with the employed laboratory equipment. Therefore, tests referred to a same mix design were performed in different days even with some fluctuations of room temperature and room humidity.

Hereinafter the materials, the mix designs, the preparation of component A and B, and the tests are illustrated.

## 6.1 Materials employed

### 6.1.1 CEMENT

The employed cement is obtained by the grinding of clinker (at least 95 %) and gypsum and it is suitable in all that field where a high mechanical strength is requested, also for low curing time. Its unit weight is  $3.1 \text{ g/cm}^3$  and a characteristic mechanical strength of 52.5 MPa. The cement is in compliance with the European Standard EN 197/1 (about compliance policy, chemical, physical and mechanical requirements and requirements of the components) and the European regulation 305/2011 (CPR) (Figure 6.1, left).

### 6.1.2 BENTONITE

Five different types of bentonites were selected to prepare two-component grouts and hereinafter a brief description for each one is provided from data acquired from technical data sheets.

#### 6.1.2.1 Type 1

Type 1 is a sodium bentonite developed for the stabilization of cementitious suspensions. It appears as a beige dust having a unit weight of  $2.5 \text{ g/cm}^3$ , a bulk density of  $0.6 \text{ g/cm}^3$  and a swell index equals to  $15 \text{ ml/g}$  (Figure 6.1, right).



Figure 6.1: cement (left); Type 1 (right).

#### 6.1.2.2 Type 2

Type 2 is a sodium bentonite suitable to prepare cementitious mixes utilised in ground injection or in slurry walls. It is also found application as component for drilling fluids. Its colour is light yellow, and its unit weight ranges from  $2.5$  up to  $2.6 \text{ g/cm}^3$ , whilst the bulk density ranges between  $0.9$  and  $1.0 \text{ g/cm}^3$  (Figure 6.2, left).

#### 6.1.2.3 Type 3

Type 3 is a sodium bentonite suitable to prepare cementitious mixes utilised in ground injection or in slurry walls. It is also found application as component for drilling fluids. It appears as a light-yellow dust having a unit weight ranging from 2.5 up to 2.6 g/cm<sup>3</sup>, a bulk density varying from 0.9 up to 1.0 g/cm<sup>3</sup> and a swell index greater than 22 ml/2g (Figure 6.2, right).



Figure 6.2: Type 2 (left); Type 3 (right).

#### 6.1.2.4 Type 4

Type 4 is a natural sodium bentonite suitable to prepare self-hardening and waterproofing mixtures. It appears as a brown dust and its unit weight is 2.5 g/c m<sup>3</sup> (Figure 6.3, left).

#### 6.1.2.5 Type 5

Type 5 is a natural sodic bentonite suitable to prepare cementitious suspensions having high viscosity, low permeability and lubricating power employed for ground injection, back filling operation and as drilling fluids. It has a unit weight of 2.7 g/cm<sup>3</sup> (Figure 6.3, right).

### 6.1.3 RETARDING/FLUIDIFYING AGENT

The retarding/fluidifying agent utilised is the Mapequick CBS System 1 produced by Mapei (Figure 6.4, left). It is a liquid retarding agent with plasticizing effect which inhibits the



setting time of cementitious mixes for long period achieving a workability up to 72 hours after the mix batching. The agent reduces the viscosity of the mix and improves its stability which results in a reduction of the bleeding effects. It appears as a brown liquid; it has a density of  $1.22 \pm 0.03 \text{ g/cm}^3$  at  $20^\circ\text{C}$  and a pH value of  $7 \pm 2$  (technical data sheet of Mapequick CBS System 1).

#### 6.1.4 ACCELERATING AGENT

The accelerating agent utilised is the Mapequick CBS System 3 produced by Mapei (Figure 6.4, right). It is a liquid accelerating agent of cement-based mixes for injection, particularly suitable for extremely fluid mixes with a very high content of water (as in the case of the two-component grout which is its main field of application). It appears as a colourless/transparent liquid; it has a density of  $1.35 \pm 0.03 \text{ g/cm}^3$  at  $20^\circ\text{C}$ , a viscosity lower than 250 cps at  $20^\circ\text{C}$  and a turbidity lower than 30 N.T.U. at  $20 - 25^\circ\text{C}$  (technical data sheet of Mapequick CBS System 3).

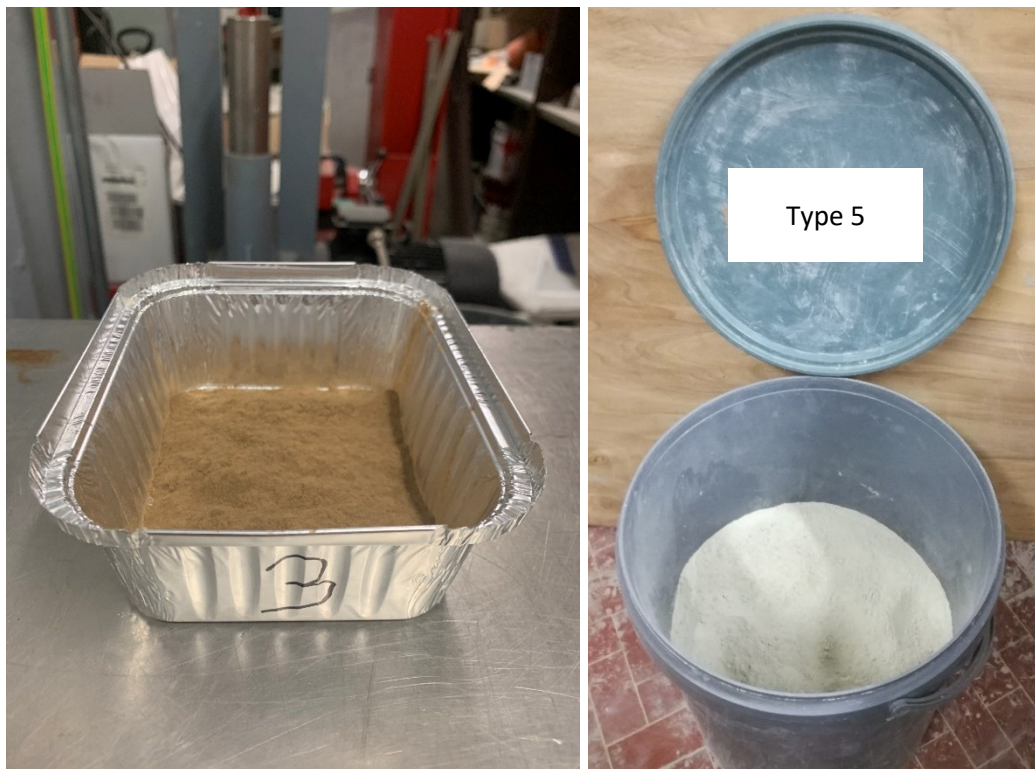


Figure 6.3: Type 4 (left); Type 5 (right).



Figure 6.4: Maapequick CBS System 1 retarding/fluidifying agent (left); Maapequick CBS System 3 accelerating agent (right).

#### 6.1.5 WATER

The employed water was that provided by the water supply network of the Metropolitan City of Turin.

### 6.2 Mix design

Two-component grout was prepared following the mix design proposed by Todaro et al., 2019. This mix design was obtained by testing several two-component grouts varying the proportions of the components until to reach an optimum mix in agreement with requirements and stakeholders (Table 6.2). Further information can be found in the cited paper.



To properly calculate the mix design, it is requested to know the quantities in weight and the unit weights of the components such as cement, bentonite, retarding/fluidifying agent, and accelerating agent, necessary to prepare a cubic metre (or a thousand litres) of grout. The volume of each element is calculated dividing the weight by the related unit weight. The required volume of water is calculated as completion to reach a thousand litres; subtracting a thousand litres by the summation of the volumes of the other elements (accelerating agent, bentonite, cement, and retarding/fluidifying agent) volume of water is obtained. Dividing the volume of each element by the overall volume (1000 l), the related fraction of each element is obtained. At this point, to evaluate the quantities required to prepare a desired volume of grout, e.g., three litres, it is necessary to multiply the fraction of each element by the volume to prepare.

Table 6.2: mix design proposed by Todaro et al., 2019.

Element	Weight (kg)
Cement	230
Bentonite	30
Water	853
Retarding/fluidifying agent	3.5
Accelerating agent	81

#### 6.2.1 MIX DESIGNS UTILISED

Hereinafter the mix designs adopted according to the type of bentonite utilised are shown, following the procedure reported previously.

The unit weights of Type 2 and Type 3 were obtained as mean value of the density reported in technical data sheets. Weight values of the components required to prepare three litres of grout are expressed in grams and rounded to first decimal digit. As illustrates from Table 6.3 to Table 6.7 the differences between one mix design and another one are limited only for the quantity of bentonite and water, which vary of few grams.

Table 6.3: mix design for Type 1.

Element	Weight (kg)	Unit weight (kg/l)	Volume (l)	Fraction (%)	Volume for 3 l (l)	Weight for 3 l (g)
Cement	230.0	3.1	74.2	7.42	0.2226	690.0
Bentonite	30.0	2.5	12.0	1.20	0.036	90.0
Retarding/fluidifying agent	3.5	1.22	3.0	0.30	0.009	11.0
Accelerating agent	81.0	1.37	60.0	6.00	0.18	246.6
<b>TOTAL</b>	/	/	<b>149.2</b>	/	/	/
Water	850.8	1.0	850.8	85.08	2.5524	2552.4

Table 6.4: mix design for Type 2.

Element	Weight (kg)	Unit weight (kg/l)	Volume (l)	Fraction (%)	Volume for 3 l (l)	Weight for 3 l (g)
Cement	230.0	3.1	74.2	7.42	0.2226	690.0
Bentonite	30.0	2.55	11.8	1.18	0.0354	90.3
Retarding/fluidifying agent	3.5	1.22	3.0	0.30	0.009	11.0
Accelerating agent	81.0	1.37	60.0	6.00	0.18	246.6
<b>TOTAL</b>	/	/	<b>149.0</b>	/	/	/
Water	851.0	1.0	851.0	85.10	2.553	2553.0

Table 6.5: mix design for Type 3.

Element	Weight (kg)	Unit weight (kg/l)	Volume (l)	Fraction (%)	Volume for 3 l (l)	Weight for 3 l (g)
Cement	230.0	3.1	74.2	7.42	0.2226	690.0
Bentonite	30.0	2.55	11.8	1.18	0.0354	90.3
Retarding/fluidifying agent	3.5	1.22	3.0	0.30	0.009	11.0
Accelerating agent	81.0	1.37	60.0	6.00	0.18	246.6
<b>TOTAL</b>	/	/	<b>149.0</b>	/	/	/
Water	851.0	1.0	851.0	85.10	2.553	2553.0

Table 6.6: mix design for Type 4.

Element	Weight (kg)	Unit weight (kg/l)	Volume (l)	Fraction (%)	Volume for 3 l (l)	Weight for 3 l (g)
Cement	230.0	3.1	74.2	7.42	0.2226	690.0
Bentonite	30.0	2.5	12.0	1.20	0.036	90.0
Retarding/fluidifying agent	3.5	1.22	3.0	0.30	0.009	11.0
Accelerating agent	81.0	1.37	60.0	6.00	0.18	246.6
<b>TOTAL</b>	/	/	<b>149.2</b>	/	/	/
Water	850.8	1.0	850.8	85.08	2.5524	2552.4

Table 6.7: mix design for Type 5.

Element	Weight (kg)	Unit weight (kg/l)	Volume (l)	Fraction (%)	Volume for 3 l (l)	Weight for 3 l (g)
Cement	230.0	3.1	74.2	7.42	0.2226	690.0
Bentonite	30.0	2.7	11.1	1.11	0.0333	90.0
Retarding/fluidifying agent	3.5	1.22	3.0	0.30	0.009	11.0
Accelerating agent	81	1.37	60.0	6.00	0.18	246.6
<b>TOTAL</b>	/	/	<b>148.3</b>	/	/	/
Water	851.7	1.0	851.7	85.17	2.5551	2555.1

In addition is illustrated the mix design without bentonite (Table 6.8). This mix design has no practical use however it is useful to compare properties of standard mix designs, such as mechanical strength.

Table 6.8: mix design for component A without bentonite.

Element	Weight (kg)	Unit weight (kg/l)	Volume (l)	Fraction (%)	Volume for 3 l (l)	Weight for 3 l (g)
Cement	230.0	3.1	74.2	7.42	0.2226	690.0
Retarding/fluidifying agent	3.5	1.22	3.0	0.30	0.009	11.0
Accelerating agent	81	1.37	60.0	6.00	0.18	246.6
<b>TOTAL</b>	/	/	<b>137.2</b>	/	/	/
Water	862.8	1.0	862.8	86.28	2.5884	2588.4

#### 6.2.2 COMPUTATION OF THE QUANTITY OF COMPONENT B IN RELATION TO COMPONENT A

The amount of component B (or accelerating agent) required to obtain a good gelation of any quantity of component A is calculated using the simple proportion reported in (Formula 6.1). The overall weight of component A ( $A_{\text{mix design}}$ ) is given by the summation of the weights of its component such as bentonite, cement, retarding/fluidifying agent, and water, as obtained from the mix design. Weight of component B ( $B_{\text{mix design}}$ ) is procured from the mix design. The required weight of component A or B is fixed whilst the weight of the other component is simply calculated.

$$A : A_{mix\ design} = B : B_{mix\ design} \quad (6.1)$$

*A<sub>mix design</sub> is the amount of component A obtained in the mix design (g);*

*B<sub>mix design</sub> is the amount of component B obtained in the mix design (g);*

*A is the desired amount of component A to combine with B (g);*

*B is the amount of component B to combine with A (g).*

### 6.3 Preparation of component A

Component A is prepared following a laboratory scale procedure proposed in the paper of Todaro et al., 2019, which emulates the batching grout plants in construction site. Purpose of this procedure is to obtain a component A most similar to that obtained on construction site regarding the homogeneity and the properties thus to obtain reliable data with laboratory tests.

Due to the dimension of the laboratory equipment, not more than three litres of component A can be prepared at a time whilst the minimum volume is 1.5 litres because of inadequacy of mixing and lack of homogeneity of the obtained grout.

#### 6.3.1 MIXING EQUIPMENT

To obtain a proper component A it is necessary to observe the following points about the properties of the mixing equipment (Todaro et al., 2019):

- The volume of the tank should be 2 – 3 times larger than the volume of the obtained component A.
- The propeller should be featured with sloping blades able to create large turbulence and should located in the centre of the tank during the grout preparation.

- The distance between the propeller and the bottom of the tank should be with a minimum that avoids impact during rotation (the distance has an order of magnitude of few millimetres). A larger distance will cause the formation of a thick not-mixed volume of grout below the propeller (Figure 6.5).
- The diameter of the tank should be two times larger than the diameter of the propeller to obtain a good turbulence in the tank.

Hereinafter a description of the required equipment is illustrated.

#### *6.3.1.1 Tank with lid*

A simple cylindrical tank with lid to insert and mix the elements to obtain the component A (Figure 6.7).

#### *6.3.1.2 Propeller*

A propeller to mix homogenously the elements (Figure 6.6 and Figure 6.7).

#### *6.3.1.3 Mixer*

A mixer able to rotate the propeller at different rotations per minute (rpm) at least in the range 800 - 2000 rpm (Figure 6.7).

#### *6.3.1.4 Vacuum cleaner*

A vacuum cleaner or any device able to suction dust generated during the addition of cement and bentonite in the tank (Figure 6.7).

#### *6.3.1.5 Containers*

Two containers to contain respectively the bentonite and the cement and two smaller containers for the retarding/fluidifying agent (Figure 6.8).

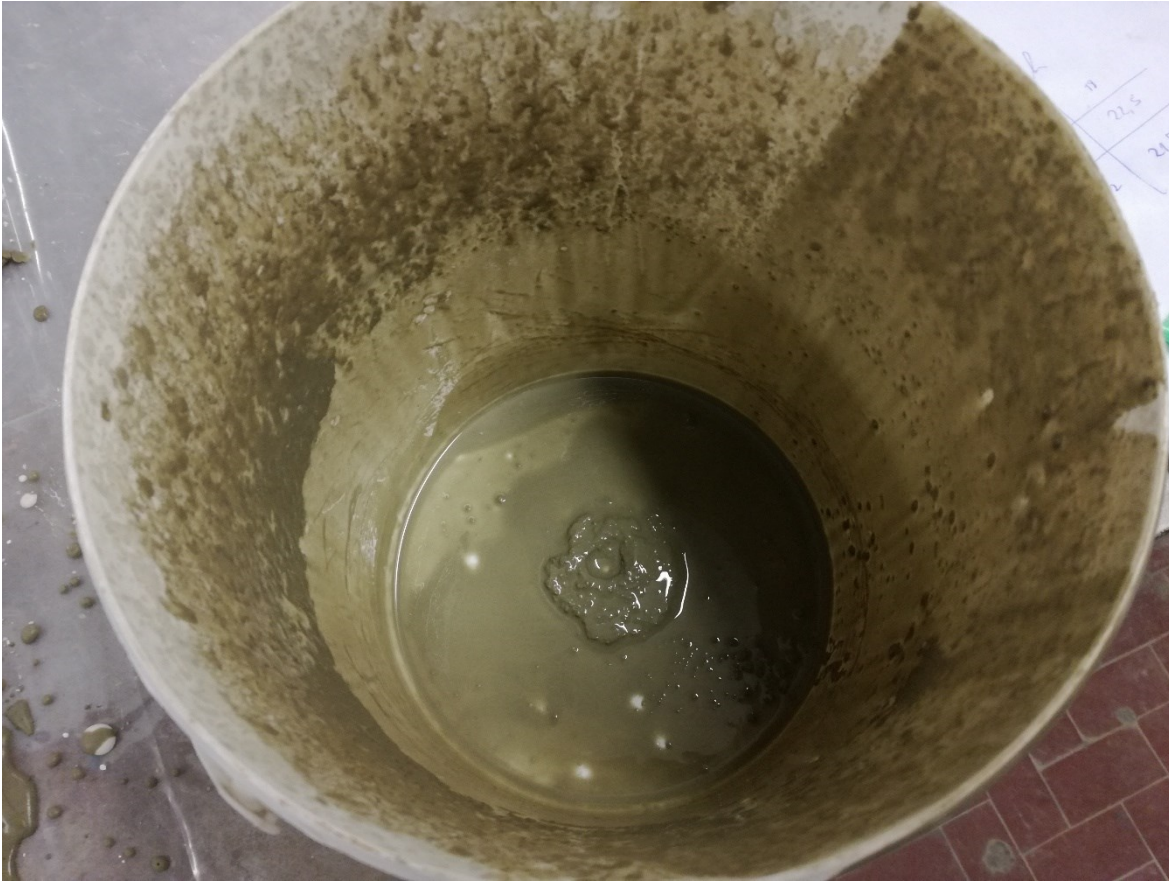


Figure 6.5: Thick not-mixed volume of grout on the bottom of the tank due to a non-proper installation of the propeller.

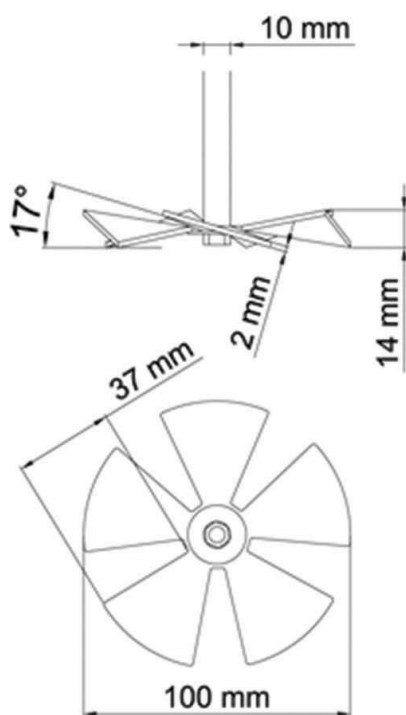


Figure 6.6: scheme (Todaro et al., 2019) (left) and photo of the utilised propeller (right).



Figure 6.7: photo of tank with lid (A), mixer (B), propeller (C) and vacuum cleaner (D).



Figure 6.8: Containers for cement and bentonite (left) and two small containers for retarding/fluidifying agent (left one is “wetted”) (right).

### 6.3.2 WEIGHING OF THE ELEMENTS

All the elements are weighted using a precision scales having an accuracy of  $\pm 0.01$  gram (Figure 6.9).



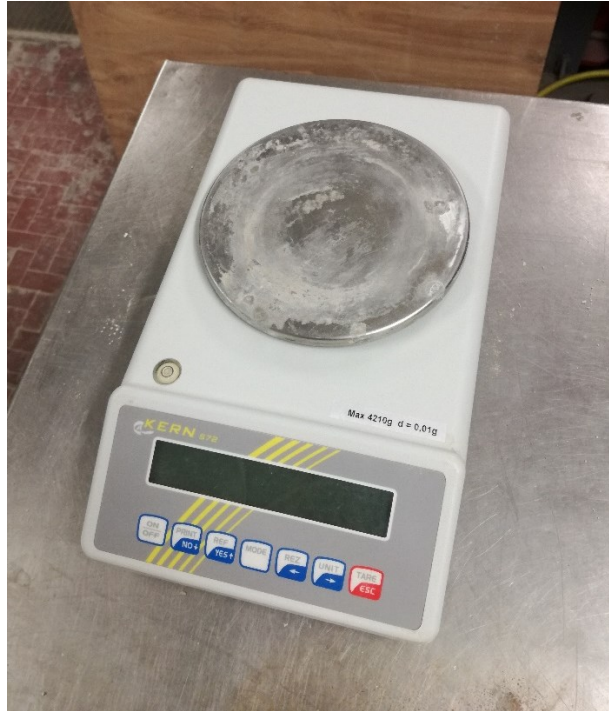


Figure 6.9: precision scales.

#### 6.3.2.1 *Water*

The tank is tared using a precision scale (0.01 g), then water is added until to reach the wanted weight.

#### 6.3.2.2 *Bentonite*

The container for the bentonite is tared and then bentonite is added till reach the wanted weight.

#### 6.3.2.3 *Cement*

The container for the cement is tared and then cement is added till reach the wanted weight.

#### 6.3.2.4 *Retarding/fluidifying agent*

To proper weight the retarding/fluidifying agent is requested to have two containers. First, a certain amount of the agent is inserted in the first container, then the quantity is poured in the second one ensuring to rotate the first container to wet completely its inner surface. At this point, the wetted container is tared and the retarding/fluidifying agent is added till



reach the wanted weight. This procedure is fundamental to add the correct quantity of agent into the admixture. If this procedure is not applied, during the addition of the retarding/fluidifying agent, some liquid will adhere on the inner surface of the container reducing the real amount inserted in the tank, moreover small changes in the quantity of the agent have large effects on the properties of the obtained grout (Figure 6.8).

### 6.3.3 MIXING PROCEDURE

Once the required elements are properly weighted as reported in 6.3.2, the preparation of the component A can start.

#### 6.3.3.1 Preliminary operation

The tank filled with water is placed below the mixer, the blender and the propeller are fixed. The mixer is initiated till reach 800 rpm, then increased to 2000 rpm and finally decreased to 800 rpm. This passage is fundamental to check the correct placement of the mixer and of the propeller; if the propeller is not correctly placed e.g., it is too close to the bottom of the tank and in contact with it, the entire system will vibrate.

#### 6.3.3.2 Mixing operation

With the propeller speed at 800 rpm, pouring the bentonite in the tank and gradually increase the propeller rotation speed until reach 2000 rpm. Mix for a time equal to the activation time (e.g., 2', 4', 7') before to add the cement in the tank and continue to mix for three minutes, finally, pour the retarding/fluidifying agent continuing to mix for two minutes (Table 6.9).

##### 6.3.3.2.1 MIXING OPERATION FOR ZERO MINUTES ACTIVATION TIME

The procedure to prepare a two-component grout with zero activation time is slighter different from the previous one: it is necessary to pour at the same time the bentonite and

the cement in the tank and gradually increase the propeller rotation speed till reach 2000 rpm. Mix for three minutes, then, pour the retarding/fluidifying agent in the tank and continue to mix for two minutes (Table 6.10).

#### 6.3.3.2.2 MIXING OPERATION FOR COMPONENT A WITHOUT BENTONITE

The procedure is identical to 6.3.3.2.1 except for pouring of bentonite (Table 6.11).

To safety reason remember to turn on the vacuum cleaner to suction dust generated during the addition of cement and bentonite in the tank.

Table 6.9: Mixing operation for component A with activation time not equal to zero minutes.

Mixing operation	Propeller rotation speed (rpm)	Duration (min)
Place the tank with water and start the mixer	800	/
Add bentonite and mix	800 → 2000	According to activation time (2, 4 or 7)
Add cement and mix	2000	3
Add retarding/fluidifying agent and mix	2000	2

Table 6.10: Mixing operation for component A with activation time equal to zero minutes.

Mixing operation	Propeller rotation speed (rpm)	Duration (min)
Place the tank with water and start the mixer	800	/
Add bentonite and cement and mix	800 → 2000	3
Add retarding/fluidifying agent and mix	2000	2

Table 6.11: Mixing operation for component A without bentonite.

Mixing operation	Propeller rotation speed (rpm)	Duration (min)
Place the tank with water and start the mixer	800	/
Add cement and mix	800 → 2000	3
Add retarding/fluidifying agent and mix	2000	2

## 6.4 Tests characterization

To evaluate the properties of the designed mixes, each one constituted with a different type of bentonite and batched with a different activation time, several tests were performed on component A, on gelled grout, on hardened grout, and on bentonite. Hereinafter the carried-out tests are illustrated.

### 6.4.1 TESTS ON COMPONENT A

#### 6.4.1.1 *Bleeding test*

Bleeding value is defined as the ratio, expressed in percentage, between the volume of exudate water and a fixed volume of component A. Exudate water natural occurs due to the sedimentation of cement over the time. The test is carried out in compliance with the standard UNI 11152-11:2005 (Sospensioni acquose per iniezioni a base di leganti idraulici - Caratteristiche e metodi di prova. Parte 11).

The standard establishes to insert a litre of component A in a standard graduated cylinder (its geometry is defined as reported in the standard) and to seal it with a stopper or an impervious element (e.g., a plastic adhesive tape). Do not accomplish the sealing operation mean to invalidate the test due to the evaporation of the exudate water (Figure 6.10). Using a standard graduated cylinder, it is possible to calculate the bleeding value by measuring the height of exudate water without the necessity to directly measure its volume by removing the impervious cap and by using a pipette; this last operation will tamper with the test impeding to perform measurements over the time. The measurements should be recorded at the following times: 10', 20', 30', 40', 50', 60', 90', 120', and 180 minutes.

The bleeding value is calculated according to its definition:

$$Bleeding = \frac{V_{exudate\ water}}{V} (\%) \quad (6.2)$$

$V_{\text{exudated water}}$  is the volume of exudated water ( $\text{m}^3$ );

$V$  is the fixed volume of one litre of component A ( $\text{m}^3$ ).



Figure 6.10: standard graduated cylinders (left) and a bleeding test (right). It is visible the impervious stopper (A), the exudate water (B), and the component A (C).

#### 6.4.1.2 Unit weight

The unit weight of the component A is measured in compliance with the standard EN 1015-6:1998 (Methods of test for mortar for masonry. Part 6: Determination of Bulk Density of Fresh Mortar), using a precision scales with a resolution of  $\pm 0.005 \text{ g/cm}^3$  as shown in Figure 6.11.

The container of the precision scales is filled with component A and the cap is placed. The surplus of component A pours out from the small hole in the centre of the cap. Closing the hole of the cap with a finger, the poured-out component A is removed with a delicate flow of water, then, the precision scales is located in its box to measure the unit weight.



Figure 6.11: precision scales in its box to measure the unit weight of component A, without the cap (left), with the cap (right).

#### 6.4.1.3 Viscosity

The viscosity of the component A is evaluated by measuring the time needed to a litre of component A to flow out from the standard nozzle of the Marsh cone. The test is carried out by using the Marsh cone procedure in compliance with the standard UNI 11152-13:2005 (Sospensioni acquose per iniezioni a base di leganti idraulici - Caratteristiche e metodi di prova. Parte 13).

Component A is poured inside the Marsh cone through a sieve until to reach a notch located on the inner surface. Air bubbles, if present, are removed tapping the outer surface of the Marsh cone, then, the admixture is left to flow out into a container and the time required to fill a litre is measured (Figure 6.12).

In this laboratory campaign the viscosity tests were performed using the fresh component A and no tests were performed on mixes cured 3, 24, and 72 hours as happens as usual on the construction site.

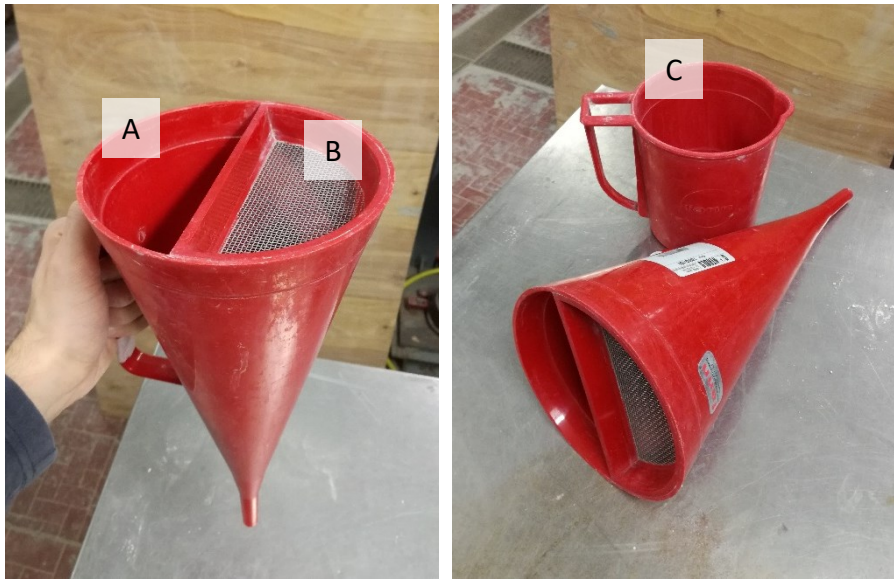


Figure 6.12: standard Marsh cone with the notch (A) and sieve (B) (left). Standard Marsh cone and container with the one litre notch (C) (right).

#### 6.4.2 TEST ON GELLED GROUT

##### 6.4.2.1 Gel time

Gel time is defined as the interval of time between the start of the mixing of the component A into the component B and the instant when the mixed grout stops to be fluid. The procedure adopted is that proposed by Todaro et al., 2019, since no standard procedures are available.

The proposed procedure consists of using two containers with a capacity of 0.4 litres, one is filled with 200 grams of component A, whilst the other one is filled with the related quantity of component B (see paragraph 6.2.2). The component A is quickly mixed with the component B by pouring them into the two containers. To obtain a good turbulence and consequently a homogeneous mixing of the two components is fundamental to start the mixing pouring first the component A into the component B. To obtain a reliable gel time is recommended to perform at least three different measurements and using the mean value as time of gelation (Figure 6.13).



Figure 6.13: six containers with capacity of 0.4 l suitable to perform three gel time tests. A stopwatch to measure the time of gelation.

#### 6.4.3 TEST ON HARDENED GROUT

##### 6.4.3.1 *Surface Compressive Strength (SCS) test*

To evaluate the short-term compressive strength of the two-component grout, the surface compressive strength (SCS) test proposed by Todaro et al., 2020 was adopted since no standards are available.

The test procedure is similar to penetrometer test and follows as guideline the standard ASTM C403/C403M-16 (Standard test method for time of setting of concrete mixtures by penetration resistance), opportunely modified to fit the characteristics of two-component grout which lacks aggregates compared to concrete. These changes are related, e. g., to the surface area of the bit and to the penetration thickness (Todaro et al., 2020).

The surface compressive strength was measured on two-component grout samples having a curing time of one hour and three hours. The test is performed leaving the samples inside

their mould and the latter must impede any displacement due to pressure applied during the test. The cast of samples is explained in 6.5.

A mould allows the casting of three samples, and three SCS tests are carried out along the surface of each sample providing a whole number of nine SCS tests per mould. The SCS values obtained for a same sample do not show any correlation or any dependence despite their close distance (Todaro et al., 2020).

The test was carried out using a SAUTER GmbH Ziegelei 1 D-72336 Balingen digital model dynamometer (max force of 1000 N and 0.5 N of resolution) equipped with a flat circular bit with an area of 177.9 mm<sup>2</sup> and 5 mm of thickness (Figure 6.14).

The dynamometer is placed perpendicularly to the sample casting surface and a growing pressure is applied manually with a constant advancement speed. If the applied pressure is not perfectly perpendicular to the surface the recorded strength will be lower than the actual one. The peak force that allows to penetrate the bit thickness for 5 mm into the two-component grout is recorded (Figure 6.15).

The surface compressive strength is a pressure, and it is the ratio between the measured peak force (N) and the area of the bit (177.9 mm<sup>2</sup>).

$$SCS = \frac{F}{A} \text{ (MPa)} \quad (6.3)$$

*SCS is the surface compressive strength (MPa);*

*F is the measured peak force (N);*

*A is the area of the bit (mm<sup>2</sup>).*





Figure 6.14: SAUTER GmbH Ziegelei 1 D-72336 Balingen digital model dynamometer (left); detail of the bit (Todaro et al., 2020) (right).



Figure 6.15: SCS tests performed on the surface of three samples inside their mould. The marks left by the test are visible.

#### 6.4.3.2 Uniaxial Compressive Strength (UCS) test

Uniaxial compressive strength tests, at 24 hours and 28 days curing time, were not performed during this laboratory campaign nonetheless they are commonly utilised in construction site to full characterize two-component grout therefore UCS test is described hereinafter.

Uniaxial compressive strength (UCS) tests are carried out in compliance with UNI EN 196-1:2016 (Methods of testing cement. Part 1: Determination of strength) with some adaptations to fit the characteristics of two-component grout which lacks aggregates compared to concrete and has a mechanical behaviour which is like a hard clay at short curing time and a weak concrete at long curing time (Todaro et al., 2019, 2020).

Samples are casted following the procedure described in 6.5, and are cured for 24 hours or 28 days according to UCS test required. Three-point flexural tests are performed on each cured sample, after demoulding, obtaining the flexural strength and splitting the sample in half, thereafter UCS tests are performed on each half. This procedure works well for samples with a curing time of 28 days, however for samples with a curing time of 24 hours it is not possible to obtain the two halves with three-point flexural test due to phenomena of supports indentations on the weak surfaces in addition to difficulty of demoulding operation. Hence, to obtain the two halves, a cutting blade is utilised without performing three-point flexural test. If UCS tests should be performed on samples with a curing time lower than 24 hours, it is not possible to cut in half the samples due to their weaknesses, hence it is necessary to utilise special moulds with waterproof partition walls to divide the mould into two or three parts obtaining respectively two or three mini-samples with their main dimension larger than 40 mm (Figure 6.16) (Todaro et al., 2019, 2020).

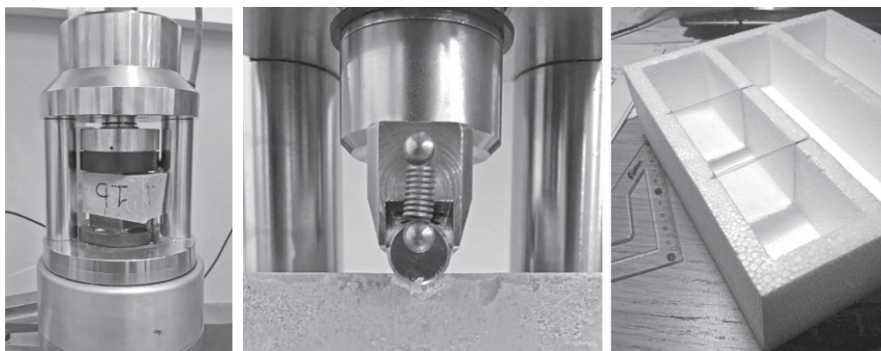


Figure 6.16: UCS test (left) (Todaro et al., 2020); Indentation phenomena (centre) and special mould with waterproof partition walls (right) (Todaro et al., 2020).

#### 6.4.4 TESTS ON BENTONITE

##### 6.4.4.1 *Liquid limit*

The liquid limit is the empirically established moisture content at which a soil passes from the liquid state to the plastic state (BS 1377-2:1990) (Methods of Test for Soils for Civil Engineering Purposes. Part 2: Classification tests). The determination of the liquid limit is carried out using the Casagrande apparatus method in compliance with the standard BS 1377-2:1990 (Figure 6.17, left).

A sample of about 300 grams of bentonite which passes the 425  $\mu\text{m}$  test sieve is taken and placed on a standard glass plate (all the five types of bentonites tested already respect this requirement). The sample is mixed with distilled water for at least 10 minutes using two palette knives (or two spatulas) until to obtain a homogeneous paste which gives a blow count of about 50 blows (Figure 6.17, right).

A portion of the mix is placed in the cup of the Casagrande apparatus taking care to not entrapping air. The surface of the paste is levelled off parallel to the base of the apparatus and divided into two equal parts using a standard grooving tool (Figure 6.18). The cup is lifted and dropped by turning the crank handle at the rate of 2 rotations per second (if the apparatus is manual) or by activating the apparatus (if automatic) until the two parts of the paste come into contact at the bottom of the groove along a distance of 13 mm. The number of bumps at which this condition occurs are recorded.

A little more of the prepared mix is taken from the glass plate and added and mixed with the paste in the cup and the procedure described in the previous paragraph is repeated until two consecutive runs give the same number of bumps. The mix on the glass plate should not dry out between one test and another one, otherwise, the number of bumps will increase as the paste dries, invalidating the test. About 10 grams of the paste, which

has just merged in the cup, is taken with a spatula and placed in a container for the determination of the moisture content (see 6.4.4.3). The paste in the cup is removed and the cup and the grooving tool are washed and dried.

Distilled water is added to the paste to increase its moisture content and the previously described operations are repeated until to obtain four or more tested mixes.

The four or more tested mixes are plotted on a semi-logarithmic chart, with the percentage of moisture contents as ordinates on the linear scales and the number of bumps as abscissa on the logarithmic scale. The number of bumps should be evenly distributed in the range from 50 up to 10 bumps. The data are fitted with a straight line and the moisture content, rounded to the nearest whole number, corresponding to the abscissa of 25 blows is the liquid limit.



Figure 6.17: Casagrande apparatus (left); standard glass and spatulas (right).



Figure 6.18: standard grooving tool (left); paste placed in the cup with groove (right).

#### 6.4.4.2 Swell index

The swell index is a parameter correlated to the swelling properties of a clay mineral when it is hydrated with distilled water. The determination of the swell index is carried out using a method in compliance with the standard ASTM D5890-18 (Standard Test Method for Swell Index of Clay Mineral Component of Geosynthetic Clay Liners).

The method utilises graduated cylinders with a capacity of 250 ml instead of the standard 100 ml glass cylinders reported in ASTM D5890-18, hence some changes are necessary to guarantee the compliance with the standard. Paper of Todaro et al., 2021, demonstrates the swell index values obtained using a 250 ml graduated cylinder are similar to those obtained with a standard glass cylinder if the following proportions are applied:

$$100 \text{ ml} : 250 \text{ ml} = 90 \text{ ml} : x_1 \text{ ml} \quad (6.4)$$

$$2 \text{ g} : 100 \text{ ml} = x_2 \text{ g} : 250 \text{ ml} \quad (6.5)$$

$$0.1 \text{ g} : 100 \text{ ml} = x_3 \text{ g} : 250 \text{ ml} \quad (6.6)$$

$x_1$  is the equivalent capacity of the 250 ml glass cylinder to fill (ml),  $x_1 = 225 \text{ ml}$ ;



$x_2$  is the mass of bentonite to be tested (g),  $x_2 = 5$  g;

$x_3$  is the mass of bentonite to insert per turn (g),  $x_3 = 0.25$  g.

About 100 grams of bentonite is grinded to have a 100 % passing in a 150  $\mu$ m (N. 100) U.S. Standard Sieve and a minimum of 65 % passing in a 75  $\mu$ m (N. 200) U.S. Standard Sieve (all the five types of bentonites tested already respect this requirement). The sample of bentonite is dried, and its moisture content is calculated (see 6.4.4.3). Using a weighing paper, 5 grams of bentonite are weighted with an accuracy of 0.01 g, then, they are subdivided in 20 samples of  $0.25 \pm 0.01$  g (Figure 6.19, left). The glass cylinder is filled with 225 ml of distilled water (Figure 6.19, right) and the first bentonite sample of 0.25 g is evenly dusted over the water surface over a period of about 30 seconds avoiding dirtying the cylinder inner surface. After 10 minutes, another 0.25 g of bentonite is dusted as previously described and this procedure is repeated till all the 5 grams are added. After the final sample is dusted and 10 minutes have passed, the graduated cylinder is filled to reach 250 ml and sealed with an impervious stopper (e.g., a plastic adhesive tape) (Figure 6.20). After a minimum time of 16 hours, the level (in millilitres) of settled bentonite is measured to the nearest 0.5 ml together with its temperature.

The swell index is calculated with the same equation utilised in case a standard 100 ml glass cylinder is employed, substituting coherently the mass of bentonite in 5 grams instead of 2 grams to accurately obtain the unit of measurement in ml / 2g:

$$SWI = \frac{V}{5} \text{ (ml/2g)} \quad (6.7)$$

$SWI$  is the swell index (ml / 2g);

$V$  is the volume of settled bentonite after 16 hours (ml);

$5$  is the whole mass of bentonite tested (g).



Figure 6.19: bentonite samples and weighing papers (left); 250 ml graduated cylinder filled with 225 ml of distilled water (right).

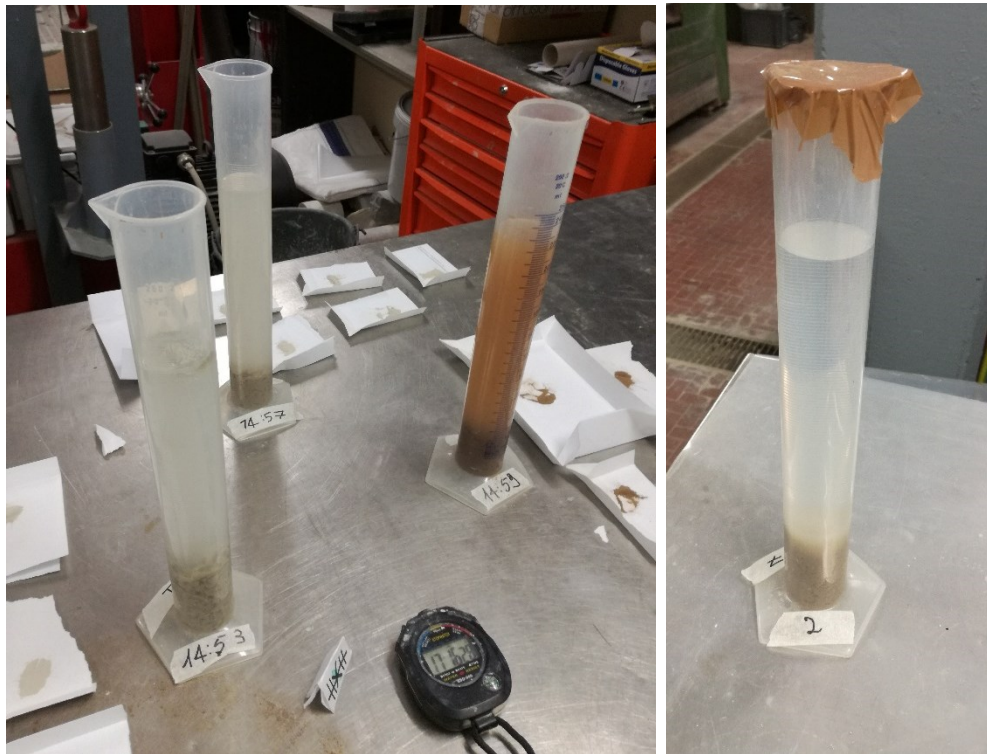


Figure 6.20: settled bentonite in the graduated cylinders during the test (left); graduated cylinder with impervious stopper (right).

#### 6.4.4.3 Moisture content

The moisture content is the amount of water expressed as a proportion by mass of the dry solid particles. A sample is defined dry when no further water can be removed at a temperature not exceeding 110 °C (BS 1377-2:1990) (Methods of Test for Soils for Civil

Engineering Purposes). The determination of the moisture content is carried out using the oven-drying method according to the standard BS 1377-2:1990.

A clean and dry container is weighted with a resolution of 0.01 grams. Then, a sample of bentonite is placed loosely in the container and weighted to the nearest 0.01 grams. The container should be sufficiently large to avoid the heap of the material. The container with the sample of bentonite is placed in a drying oven, capable to maintain a temperature of 105 °C to 110 °C, for a period sufficient to dry the sample (usually a time between 16 and 24 hours) (Figure 6.21). After that, the container with the dry sample of bentonite is placed in a desiccator (containing anhydrous silica gel) to cool and weighted to the nearest 0.01 grams.

The moisture content is calculated as a percentage from the following equation:

$$w = \frac{m_2 - m_3}{m_3 - m_1} 100 (\%) \quad (6.8)$$

*w is the moisture content (%);*

*m<sub>1</sub> is the mass of container (g);*

*m<sub>2</sub> is the mass of container and wet sample of bentonite (g);*

*m<sub>3</sub> is the mass of container and dry sample of bentonite (g).*



Figure 6.21: containers with samples of bentonite.



## 6.5 Sample casting

No standards are available to properly cast two-component grout samples since two-component grout have a mechanical behaviour which is like a hard clay at short curing time and a weak concrete at long curing time (Todaro et al., 2020). As proposed in Todaro et al., 2019 and Todaro et al., 2020, the standard UNI EN 196-1:2016 (Methods of testing cement. Determination of strength), opportunely modified, can be followed as guideline. In the past, before the spreading of the two-component technology, the mono-component grout (mainly water and cement) was widely utilised for backfilling operation and can be tested perfectly by the standard UNI EN 196-1:2016. Technology has changed however stakeholders and contract specifications still follow the standard UNI EN 196-1:2016 (Todaro et al., 2020).

### 6.5.1 CASTING PROCEDURE

Samples have a parallelepiped shape with dimension of 40 x 40 x 160 mm and are casted in non-deformable moulds. The casting phase required a high dose of dexterity to cast a homogeneous sample due to the short gelation time of two-component grouts (usually varying from 5 up to 10 seconds).

A standard clean mould with dimension to cast 40 x 40 x 160 mm samples is taken, if holes are present, they must be closed using, e.g., a paper scotch tape, avoiding the flow out of the grout. Two containers are filled, one with component A and the other one with component B. Component A is quickly poured into component B (if component B is firstly poured into A, a poor mixture is obtained due to lack of turbulence), and the obtained admixture is quickly poured into the emptied container and finally poured inside the mould. These operations should be performed very quickly to obtain a continuous and homogeneous grout flowing inside the mould. The mould must be filled with a single

attempt; it is not possible to fill a mould with subsequent flows because the obtained sample will be characterized by a stratified structure, unsuitable for testing. Once the mould is filled, and about 30 – 60 seconds have passed, the surface of the sample is scraped with a spatula in a single and continuous movement to obtain a flat surface, then, the mould is hermetically sealed to avoid water losses due to evaporation. The sample is left to cure for the desired time (Figure 6.22, Figure 6.23, and Figure 6.24).

#### 6.5.2 RECOMMENDATIONS

The volume of the mixture should be at least a 20 % greater than the volume of the mould to perform a good casting. The exceed volume permits to cast in a single flow the whole volume of the mould balancing unavoidable losses of material due to the adhesion on the inner surfaces of the containers and other losses due to an inaccurate pouring inside the mould. Furthermore, air bubbles inside the grout are commonly trapped during the casting phase due to the short gelation time. To reduce the amount of trapped air bubbles it is recommended to speed up the mixing and pouring phase, thus the trapped air bubbles can naturally ascend and reach the surface before the hardening of the grout blocks their movements.

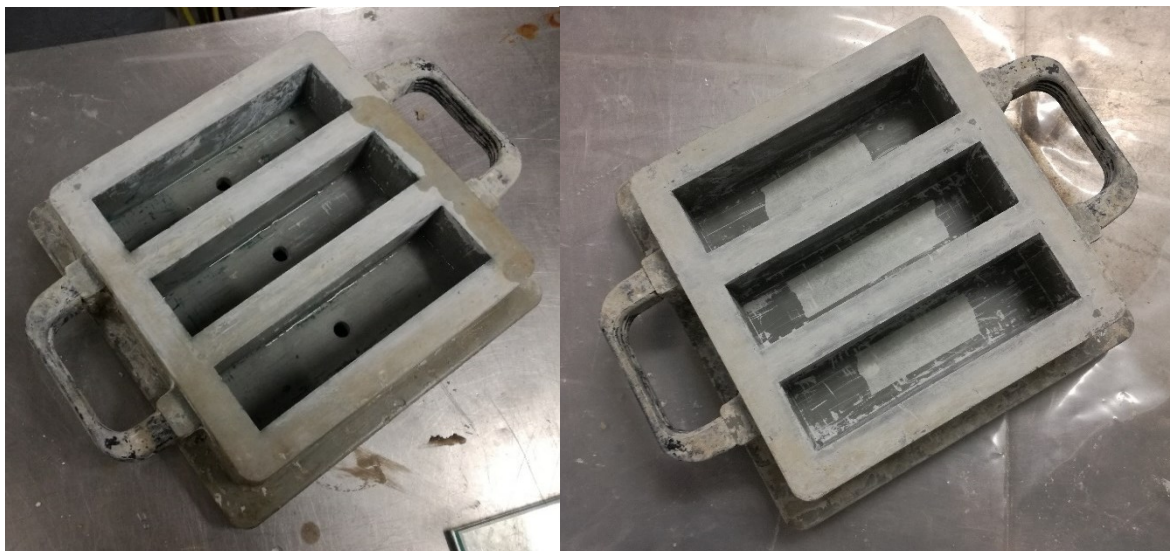


Figure 6.22: standard mould to cast three samples (left); standard mould with paper scotch tape to close the holes (right).



Figure 6.23: three samples of two-component grout just casted (left); a sample just scraped and a spatula for scraping operation (right).

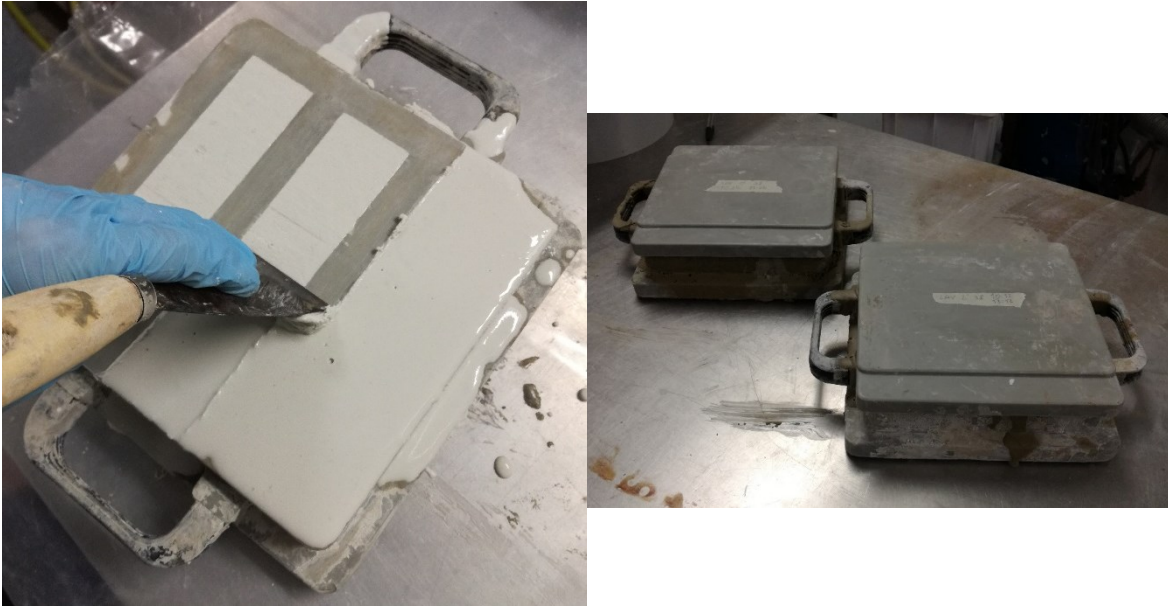


Figure 6.24: scraping operation (left); two mould hermetically sealed with their cap (right).

## 7 RESULTS OF LABORATORY TEST CAMPAIGN

### 7.1 Result of bleeding test

Hereinafter the results of bleeding tests performed on component A in relation to type of bentonite and time of activation are illustrated (Table 7.1 - Table 7.5 and Figure 7.1 - Figure 7.6).

Bleeding curves are characterized by a growing trend as time increases. Data shows a correlation between the bleeding and the time of activation: greater is the activation time of bentonite, lower is the bleeding obtained. Bleeding value is also affected by the type of bentonite inasmuch bentonites with same time of activation show different bleeding behaviours. All five bentonites with a time of activation of 0 minutes are characterised by a bleeding curve having high values which outdistances the other bleeding curves.

Type 2, Type 4, and Type 5 show a common trend in bleeding curves of component A: a large bleeding is observable for component A without activation whilst for component A with activation (2', 4', and 7 minutes) the bleeding curves have smaller values and are close each other. Type 1 and Type 3 show a similar trend too, however, in Type 3, bleeding curves for 2 minutes and 4 minutes are inverted. The difference between these curves is very small, about 0.5%, and it is possible to suppose that Type 3 is characterized by same bleeding behaviour with an activation time of 2' or 4 minutes. Differences in Type 1, instead, are more pronounced with a bleeding curve for two minutes characterised by high bleeding values, whilst bleeding curve for 7 minutes is close to 4 minutes curve, and this is similar to the behaviour of the other types of bentonites.

In Type 1, a second test on component A with a time of activation of 7 and 4 minutes was performed (named 7 min 2<sup>nd</sup> test and 4 min 2<sup>nd</sup> test) after that the first test has provided



not reliable data due to a rapid pouring of component A into the graduated cylinder causing turbulence.

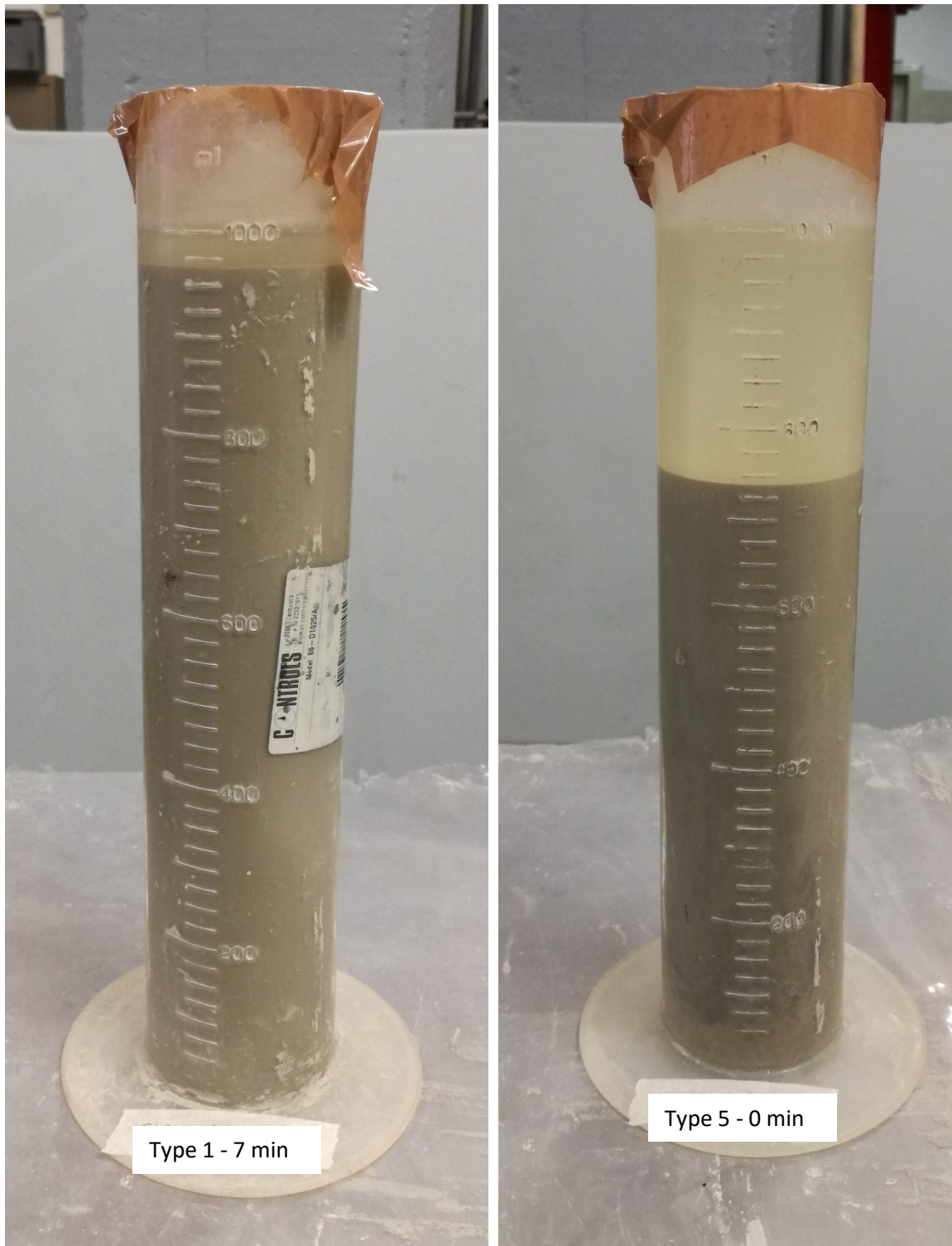


Figure 7.1: bleeding tests at 3 hours. Type 1 (2<sup>nd</sup> test) with time of activation of 7 minutes (left); Type 5 (2<sup>nd</sup> test) with zero time of activation (right).

Table 7.1: Bleeding values for Type 1 at different activation time.

Activation time 7 minutes*			Activation time 7 minutes (2 <sup>nd</sup> test)		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	1.5	0.52	10	1.0	0.35
20	4.0	1.38	20	1.5	0.52
30	6.0	2.08	30	2.0	0.69
40	7.5	2.60	40	2.5	0.87
50	9.0	3.11	50	3.5	1.21
60	10.0	3.46	60	4.0	1.38
90	13.0	4.50	90	6.0	2.08
120	14.5	5.02	120	7.0	2.42
180	19.0	6.57	180	9.0	3.11

Activation time 4 minutes *			Activation time 4 minutes (2 <sup>nd</sup> test)		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	3.5	1.21	10	1.0	0.35
20	9.5	3.29	20	2.5	0.87
30	19.0	6.57	30	3.0	1.04
40	32.5	11.25	40	4.0	1.38
50	41.5	14.36	50	5.0	1.73
60	47.5	16.44	60	5.0	1.73
90	57.0	19.72	90	6.0	2.08
120	63.5	21.97	120	7.5	2.60
180	71.5	24.74	180	10.5	3.63

Activation time 2 minutes			Activation time 0 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	2.5	0.87	10	13.0	4.50
20	6.5	2.25	20	25.0	8.65
30	11.5	3.98	30	35.0	12.11
40	19.0	6.57	40	45.5	15.74
50	25.5	8.82	50	57.0	19.72
60	32.0	11.07	60	65.0	22.49
90	55.0	19.03	90	92.0	31.83
120	64.0	22.15	120	116.5	40.31
180	72.0	24.91	180	159.5	55.19

\* not reliable data

*t*: time of measurement (min);

*H*: height of exudated water (mm);

*B*: bleeding value (%).

Table 7.2: Bleeding values for Type 2 at different activation time.

Activation time 7 minutes			Activation time 4 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	0.0	0.00	10	0.0	0.00
20	0.0	0.00	20	0.2	0.07
30	0.0	0.00	30	0.5	0.17
40	0.5	0.17	40	1.0	0.35
50	0.8	0.28	50	1.0	0.35
60	1.0	0.35	60	1.2	0.42
90	1.2	0.42	90	2.0	0.69
120	1.5	0.52	120	2.0	0.69
180	2.0	0.69	180	2.5	0.87

Activation time 2 minutes			Activation time 0 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	0.0	0.00	10	5.0	1.73
20	0.0	0.00	20	10.0	3.46
30	1.0	0.35	30	15.0	5.19
40	1.5	0.52	40	18.0	6.23
50	2.0	0.69	50	22.5	7.79
60	2.0	0.69	60	26.0	9.00
90	3.0	1.04	90	37.0	12.80
120	3.0	1.04	120	50.0	17.30
180	3.5	1.21	180	72.0	24.91

*t*: time of measurement (min);

*H*: height of exudated water (mm);

*B*: bleeding value (%).



Table 7.3: Bleeding values for Type 3 at different activation time.

Activation time 7 minutes			Activation time 4 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	1.0	0.35	10	2.0	0.69
20	1.5	0.52	20	2.5	0.87
30	2.0	0.69	30	3.5	1.21
40	2.5	0.87	40	4.5	1.56
50	3.0	1.04	50	5.0	1.73
60	3.5	1.21	60	5.0	1.73
90	4.5	1.56	90	8.0	2.77
120	5.5	1.90	120	10.0	3.46
180	7.5	2.60	180	13.5	4.67

Activation time 2 minutes			Activation time 0 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	1.0	0.35	10	10.0	3.46
20	2.0	0.69	20	17.0	5.88
30	2.5	0.87	30	21.5	7.44
40	3.0	1.04	40	27.5	9.52
50	4.0	1.38	50	33.5	11.59
60	4.5	1.56	60	38.5	13.32
90	6.5	2.25	90	55.0	19.03
120	9.0	3.11	120	73.0	25.26
180	11.5	3.98	180	111.0	38.41

*t*: time of measurement (min);

*H*: height of exudated water (mm);

*B*: bleeding value (%).

Table 7.4: Bleeding values for Type 4 at different activation time.

Activation time 7 minutes			Activation time 4 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	0.0	0.00	10	0.0	0.00
20	0.5	0.17	20	0.5	0.17
30	1.0	0.35	30	1.0	0.35
40	1.5	0.52	40	2.0	0.69
50	2.0	0.69	50	2.0	0.69
60	2.0	0.69	60	2.0	0.69
90	2.5	0.87	90	3.0	1.04
120	3.0	1.04	120	3.0	1.04
180	4.0	1.38	180	4.5	1.56

Activation time 2 minutes			Activation time 0 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	0.0	0.00	10	8.5	2.94
20	0.5	0.17	20	14.0	4.84
30	1.0	0.35	30	19.0	6.57
40	1.5	0.52	40	22.5	7.79
50	2.0	0.69	50	26.5	9.17
60	2.0	0.69	60	30.0	10.38
90	3.0	1.04	90	42.0	14.53
120	3.5	1.21	120	52.5	18.17
180	4.5	1.56	180	73.0	25.26

*t*: time of measurement (min);

*H*: height of exudated water (mm);

*B*: bleeding value (%).

Table 7.5: Bleeding values for Type 5 at different activation time.

Activation time 7 minutes			Activation time 4 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	0.0	0.00	10	0.0	0.00
20	0.0	0.00	20	0.0	0.00
30	0.0	0.00	30	0.0	0.00
40	0.0	0.00	40	0.5	0.17
50	0.5	0.17	50	1.0	0.35
60	1.0	0.35	60	1.0	0.35
90	1.2	0.42	90	1.5	0.52
120	1.8	0.62	120	2.0	0.69
180	2.0	0.69	180	3.0	1.04

Activation time 2 minutes			Activation time 0 minutes		
t (min)	H (mm)	B (%)	t (min)	H (mm)	B (%)
10	0.5	0.17	10	3.5	1.21
20	0.8	0.28	20	9.5	3.29
30	1.5	0.52	30	15.0	5.19
40	1.8	0.62	40	22.5	7.79
50	2.0	0.69	50	27.5	9.52
60	2.0	0.69	60	33.5	11.59
90	2.3	0.80	90	52.0	17.99
120	2.4	0.83	120	70.0	24.22
180	3.5	1.21	180	79.0	27.34

Activation time 0 minutes (2 <sup>nd</sup> test)		
t (min)	H (mm)	B (%)
10	6.0	2.08
20	12.0	4.15
30	16.0	5.54
40	20.0	6.92
50	24.0	8.30
60	28.0	9.69
90	40.0	13.84
120	51.0	17.65
180	73.5	25.43

*t*: time of measurement (min);

*H*: height of exudated water (mm);

*B*: bleeding value (%).

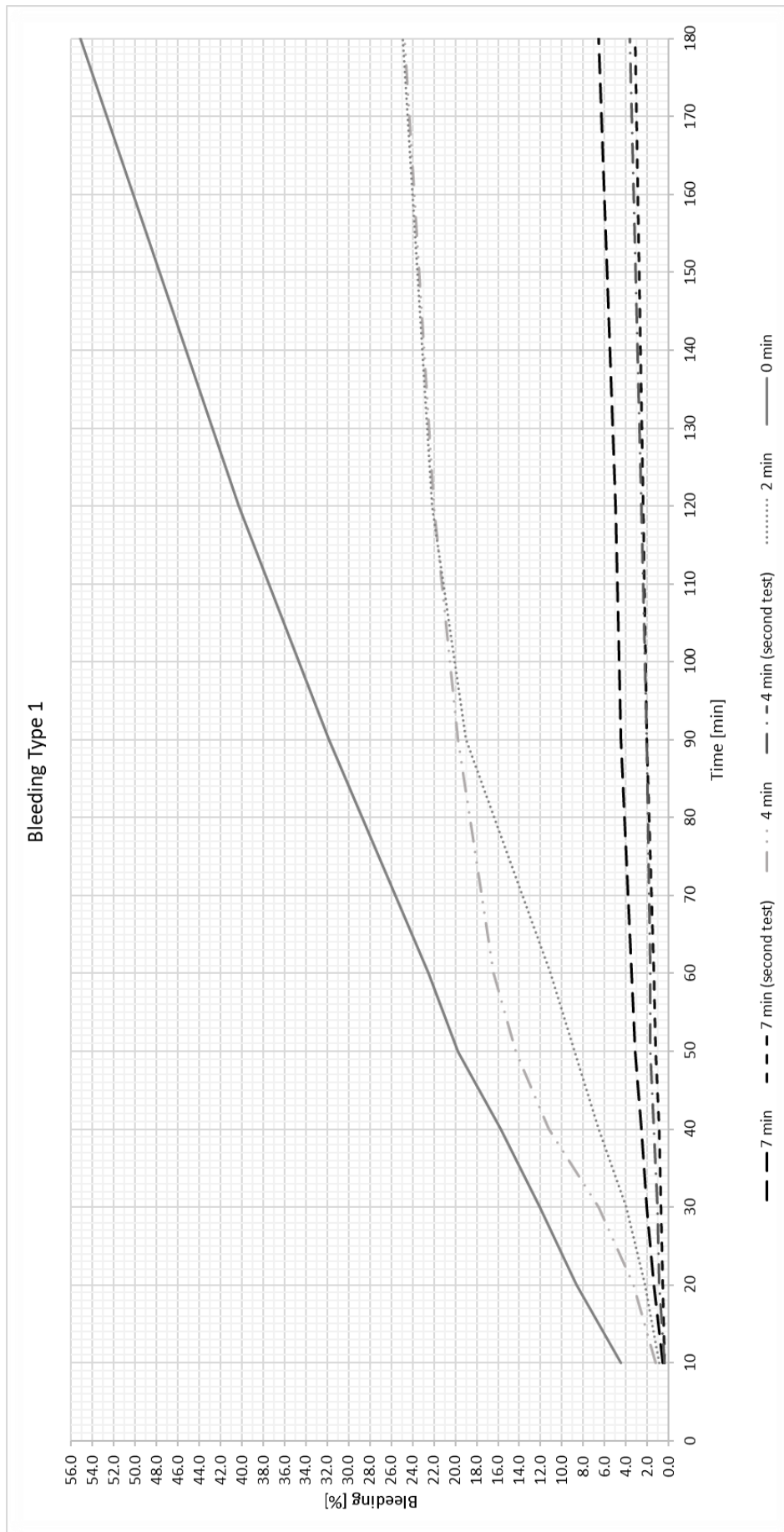


Figure 7.2: bleeding values Type 1. 7 and 4 minutes first test are not reliable.

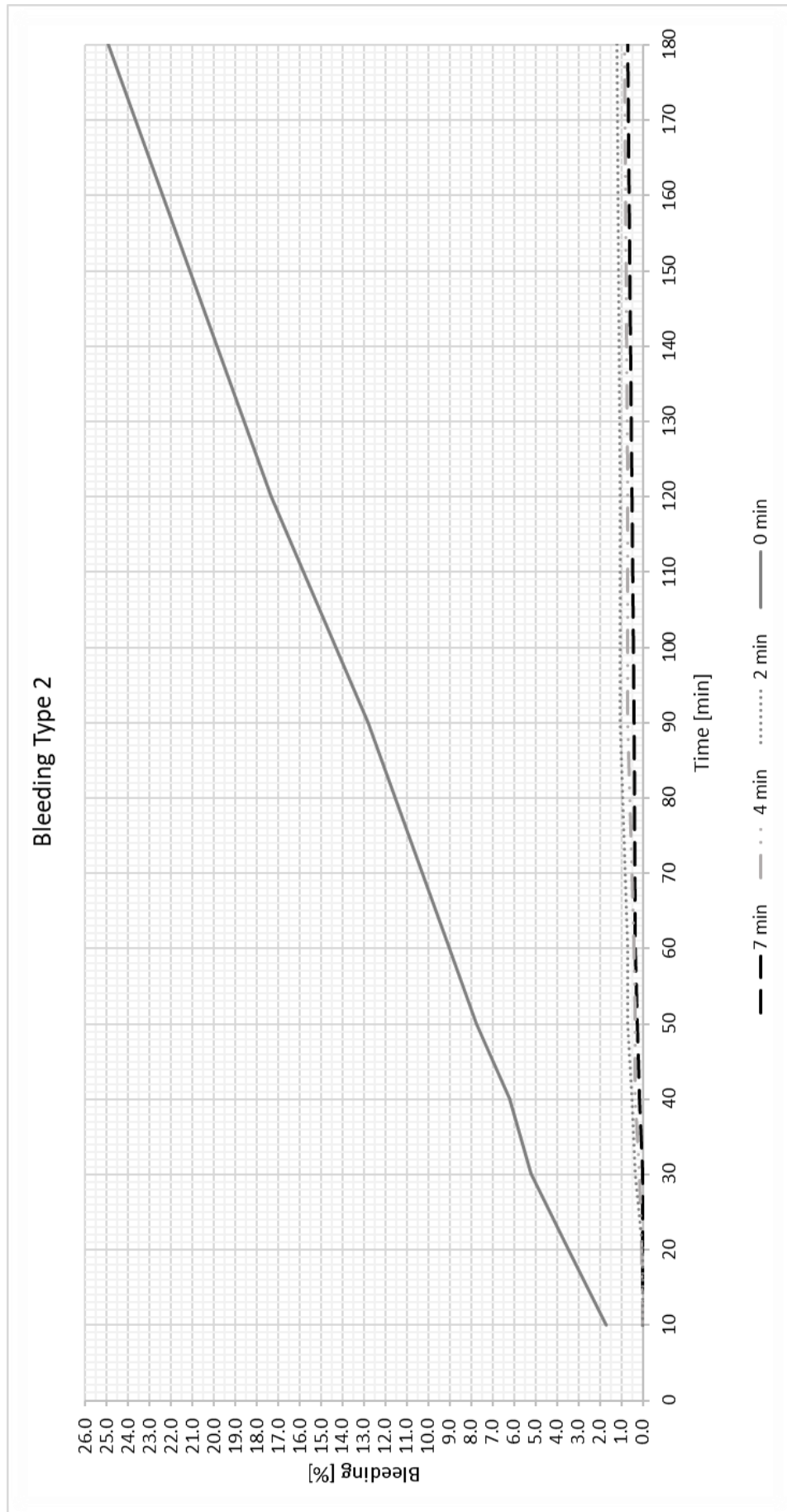


Figure 7.3: bleeding values Type 2.

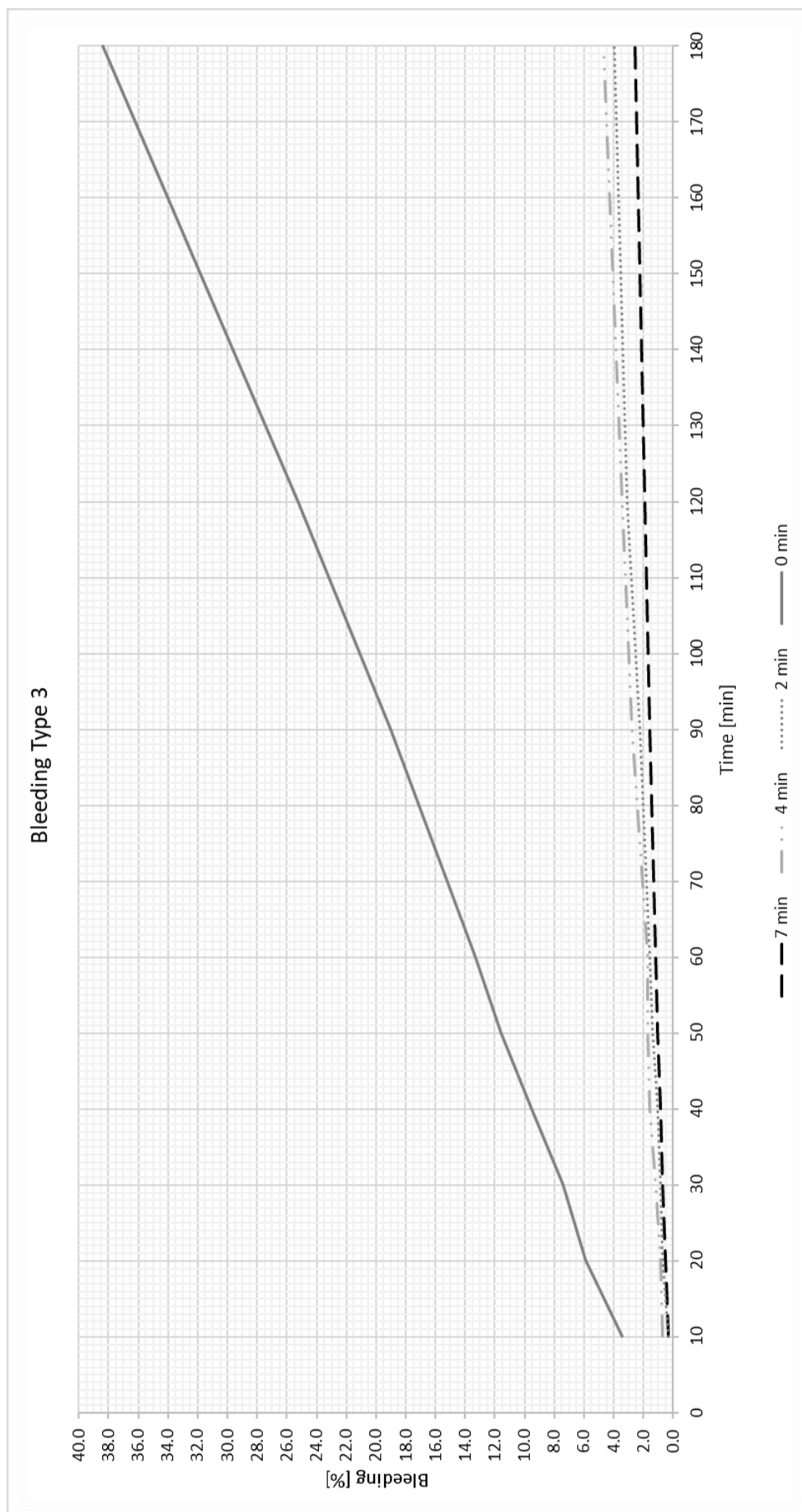


Figure 7.4: bleeding values Type 3.

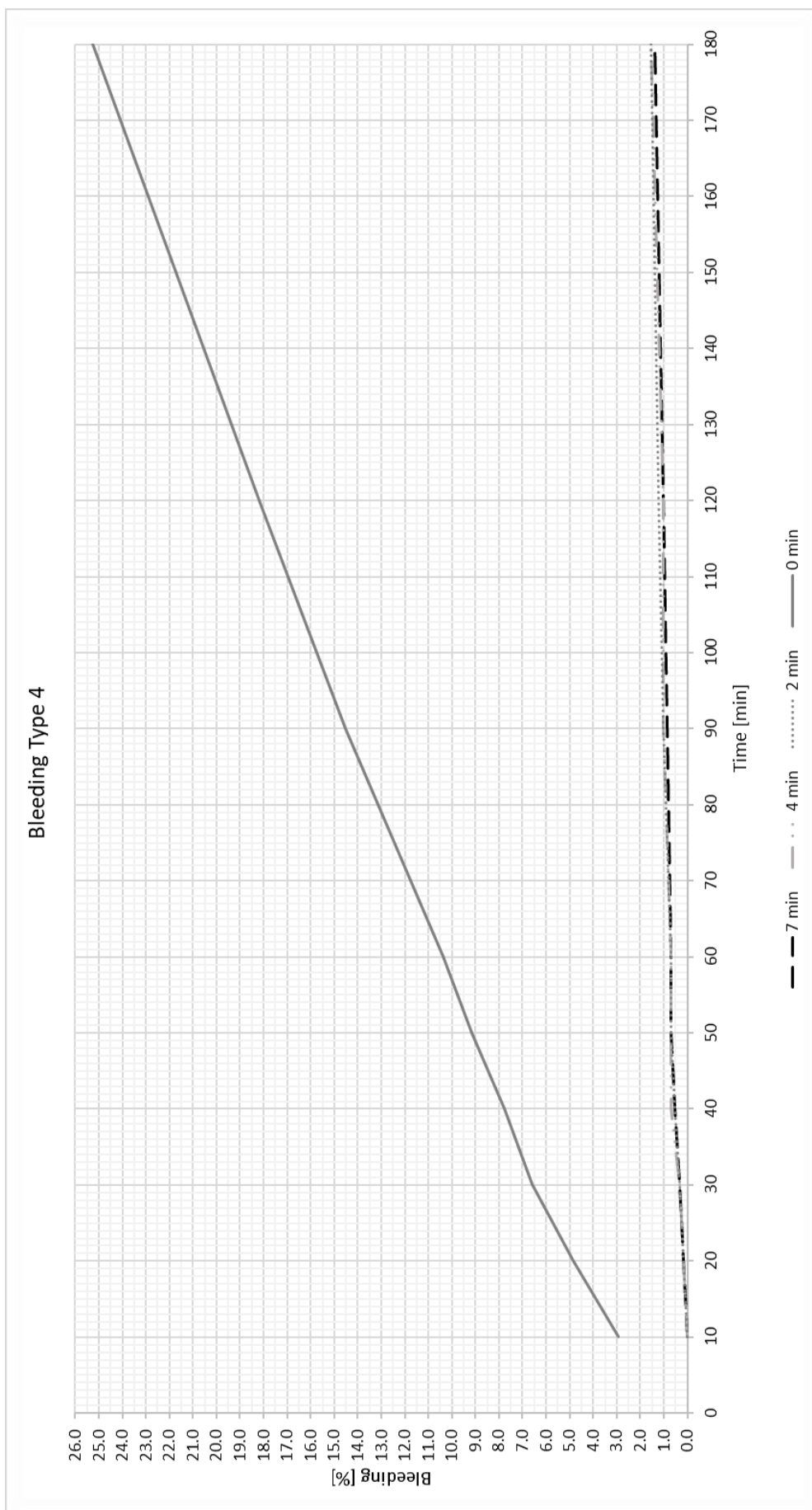


Figure 7.5: bleeding values Type 4.

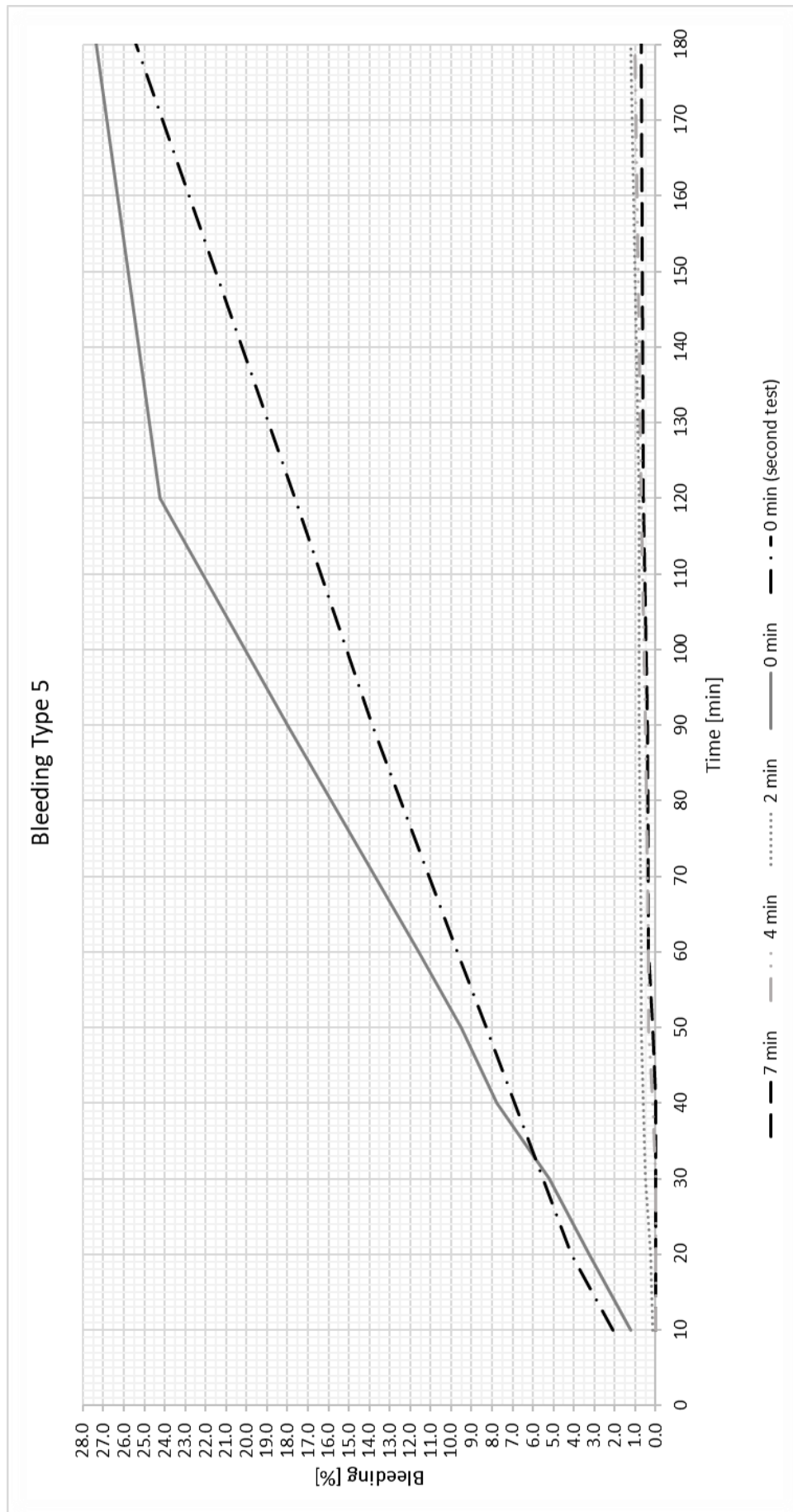


Figure 7.6: bleeding values Type 5.



## 7.2 Result of unit weight test

Unit weight of component A is not affected by type of bentonite neither by its time of activation. All component A samples have a constant unit weight ranging from 1.16 up to 1.19 g/cm<sup>3</sup> as illustrated in the following table (Table 7.6).

Table 7.6: unit weight (g/cm<sup>3</sup>) of tested bentonite related to activation time.

TYPE OF BENTONITE \ TIME OF ACTIVATION	0 min	2 min	4 min	7 min
Type 1	1.18	1.18	1.18	1.18
Type 2	1.185	1.18	1.16	1.17
Type 3	1.18	1.185	1.185	1.18
Type 4	1.19	1.19	1.185	1.185
Type 5	1.17	1.18	1.18	1.17

*values in g/cm<sup>3</sup>*

## 7.3 Result of viscosity test

From the analysis of viscosity data of component A, there is an upward trend of viscosity as time of activation increases even though it is not possible to identify a single trend to fit the behaviour of all five types of bentonites. Generally, component A without activation is characterised by flow time values ranging in the interval 28 – 29 seconds, whilst activated component A shows rising flow time values with a diversified behaviour in relation to type of bentonite. Activated Type 1, Type 3, and Type 4 have a similar behaviour since they are characterised by a flow time in the interval of 29.5 – 31.5 seconds whilst activated Type 2 and Type 5 have a flow time ranges in the interval 33 – 34 seconds. Therefore, a dependence with the type of bentonite is observable (Table 7.7 and Figure 7.7 - Figure 7.11).

Table 7.7: flow time (s) of tested bentonite related to activation time.

TYPE OF BENTONITE \ TIME OF ACTIVATION	0 min	2 min	4 min	7 min
Type 1	28.69	30.16	30.53	30.59
Type 2	28.84	33.97	32.97	33.87
Type 3	28.37	29.81	30.62	30.84
Type 4	28.35	30.47	31.47	31.00
Type 5	29.00	33.00	33.00	32.94

*values in s*

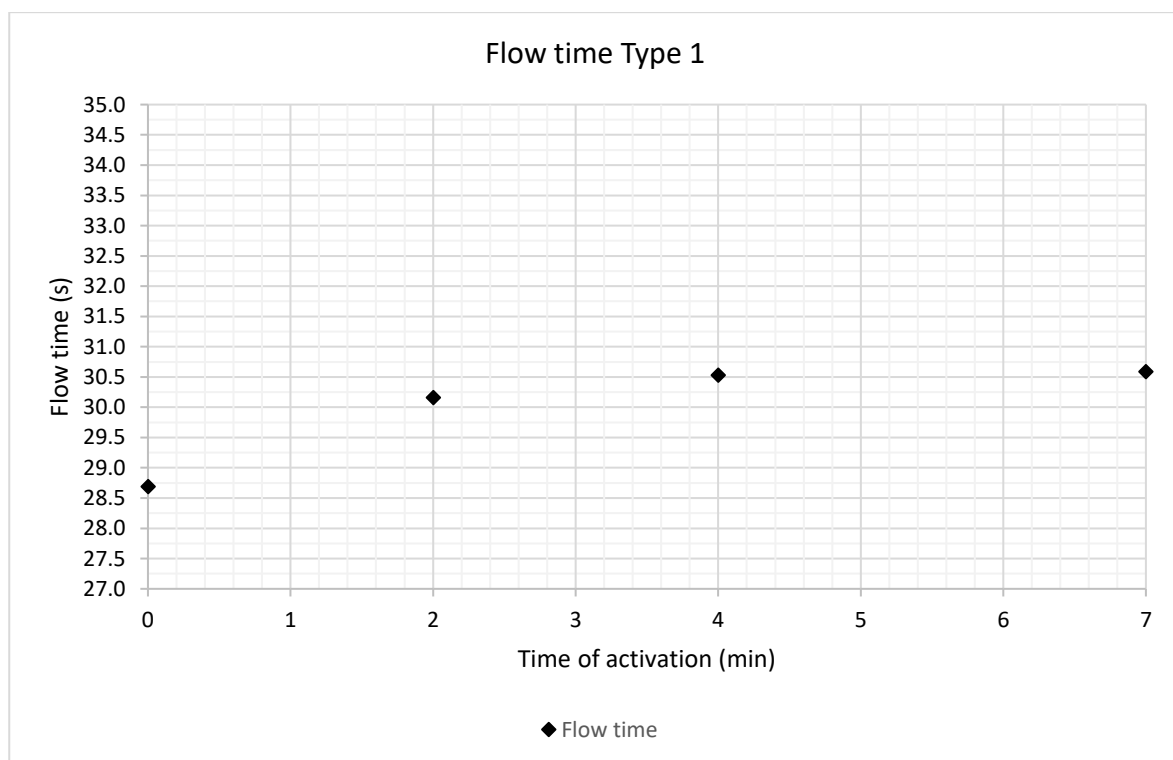


Figure 7.7: flow time Type 1.

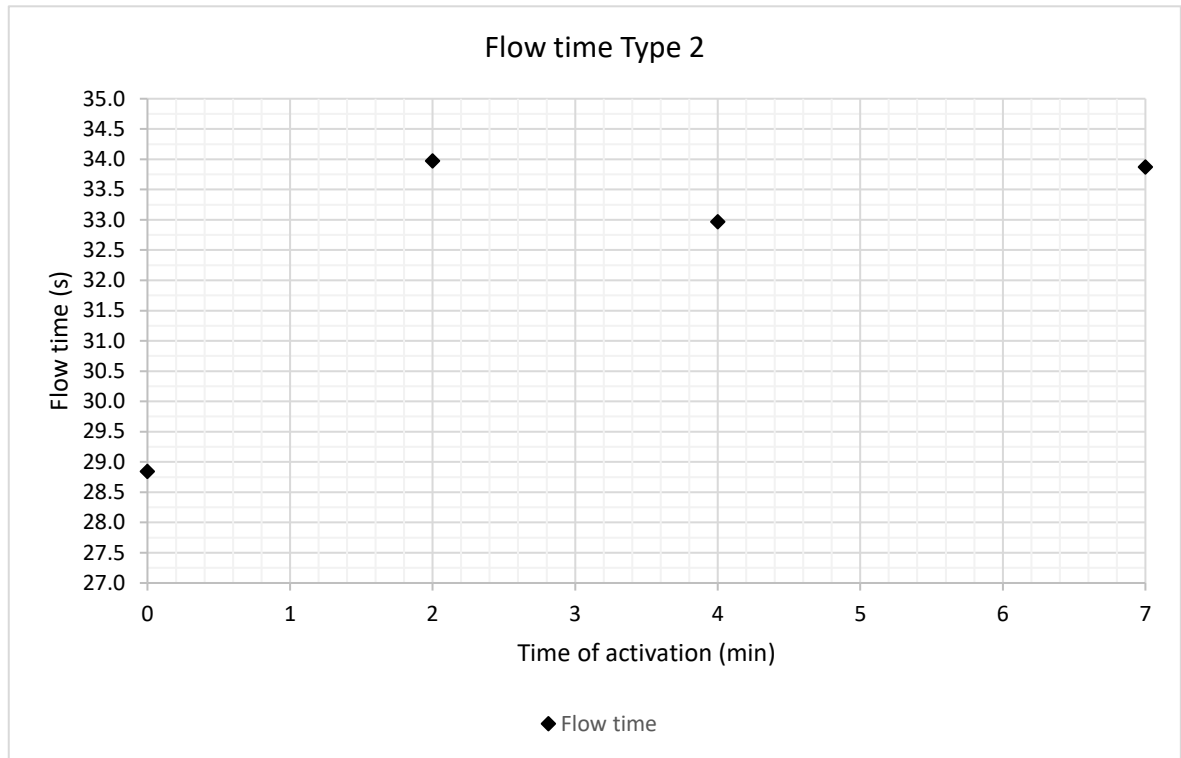


Figure 7.8: flow time Type 2.

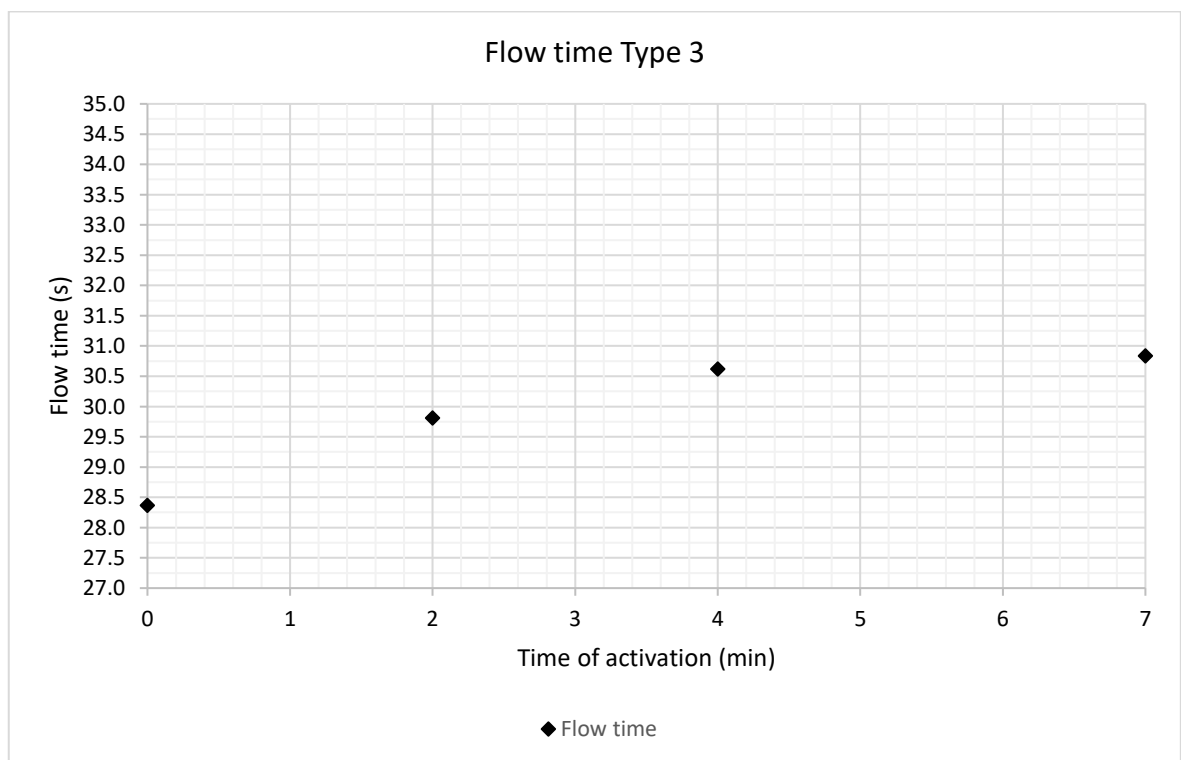


Figure 7.9: flow time Type 3.

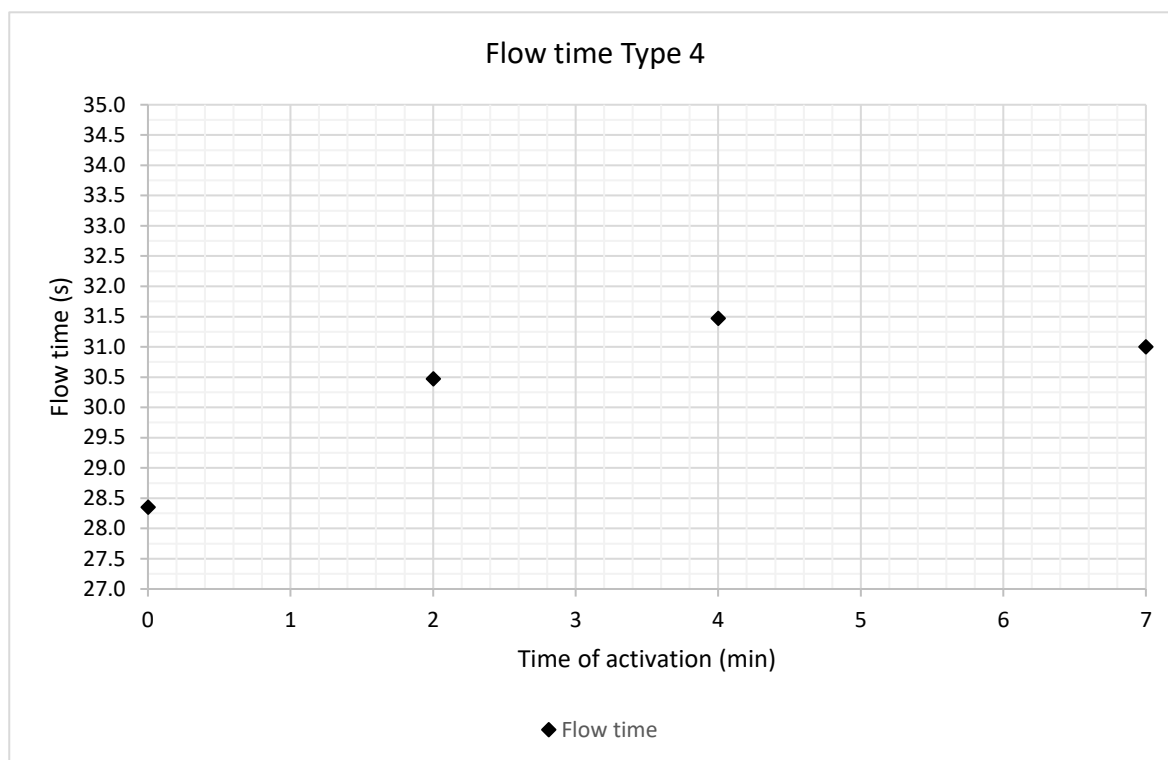


Figure 7.10: flow time Type 4.

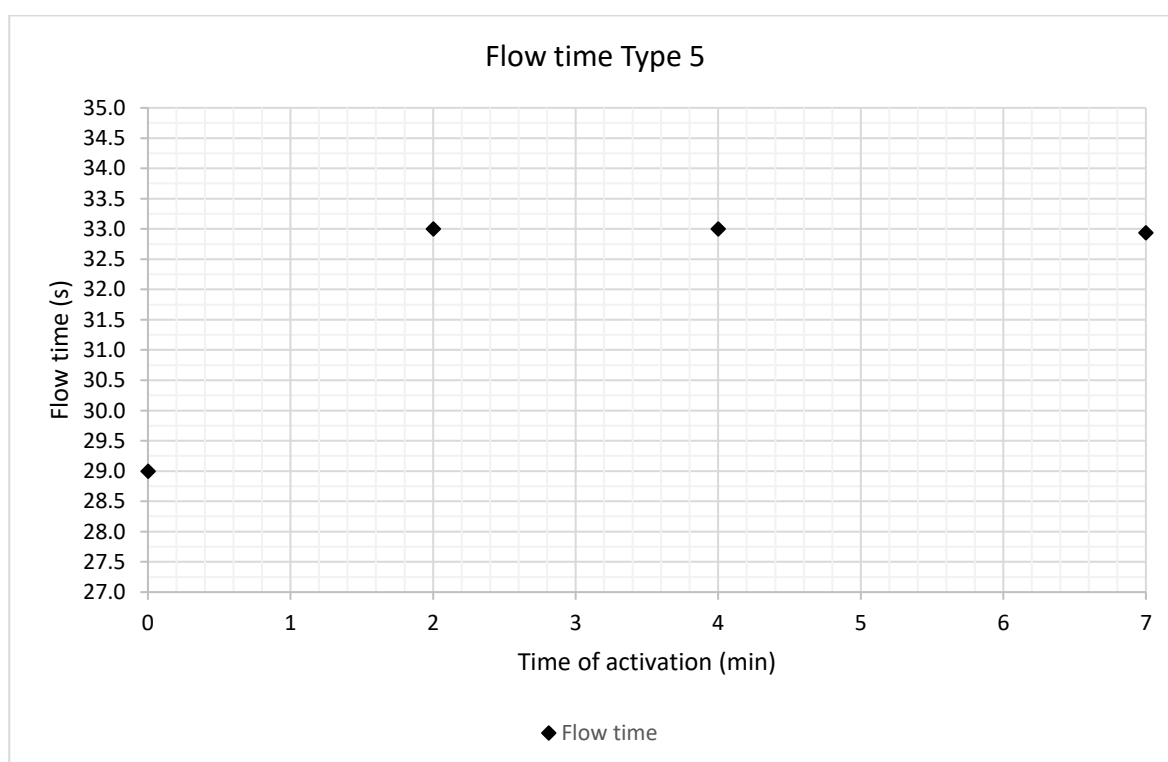


Figure 7.11: flow time Type 5.

## 7.4 Result of gel time test

From the analysis of data, gel time of two-component grout is not related with time of activation or type of bentonite (Table 7.8 - Table 7.12 and Figure 7.12 and Figure 7.16).

There is not any associable trend to gel time data because a difference of  $\pm 2$  seconds is not relevant and gel time curves can be assumed as constants.

Comparing gel time curves of two-component grout with different type of bentonite, their difference is inside the interval  $\pm 2$  seconds, and this entails type of bentonite does not influence gel time.

Hereinafter the data of gel time with the result of each test and the mean value to take into account as reference value for gel time are illustrated.

Table 7.8: gel time Type 1.

Time of activation (min)	Test 1 (s)	Test 2 (s)	Test 3 (s)	Mean value (s)
0	7.34	7.06	7.69	7.36
2	7.25	6.90	7.03	7.06
4	6.87	7.01	6.44	6.77
7	6.72	6.00	6.09	6.27

Table 7.9: gel time Type 2.

Time of activation (min)	Test 1 (s)	Test 2 (s)	Test 3 (s)	Mean value (s)
0	6.25	6.15	6.37	6.26
2	5.81	5.38	6.66	5.95
4	6.25	6.41	6.81	6.49
7	6.50	6.41	6.09	6.33

Table 7.10: gel time Type 3.

Time of activation (min)	Test 1 (s)	Test 2 (s)	Test 3 (s)	Mean value (s)
0	7.62	8.00	7.06	7.56
2	6.91	5.66	5.79	6.12
4	5.75	5.89	5.38	5.67
7	5.78	5.22	5.53	5.51

Table 7.11: gel time Type 4.

Time of activation (min)	Test 1 (s)	Test 2 (s)	Test 3 (s)	Mean value (s)
0	6.56	6.41	6.75	6.57
2	5.84	6.34	7.10	6.43
4	6.50	5.59	7.47	6.52
7	5.97	5.90	6.72	6.20

Table 7.12: gel time Type 5.

Time of activation (min)	Test 1 (s)	Test 2 (s)	Test 3 (s)	Mean value (s)
0	5.81	6.54	6.47	6.27
2	7.79	7.25	8.31	7.78
4	6.0	6.0	6.7	6.23
7	7.12	7.16	6.82	7.03

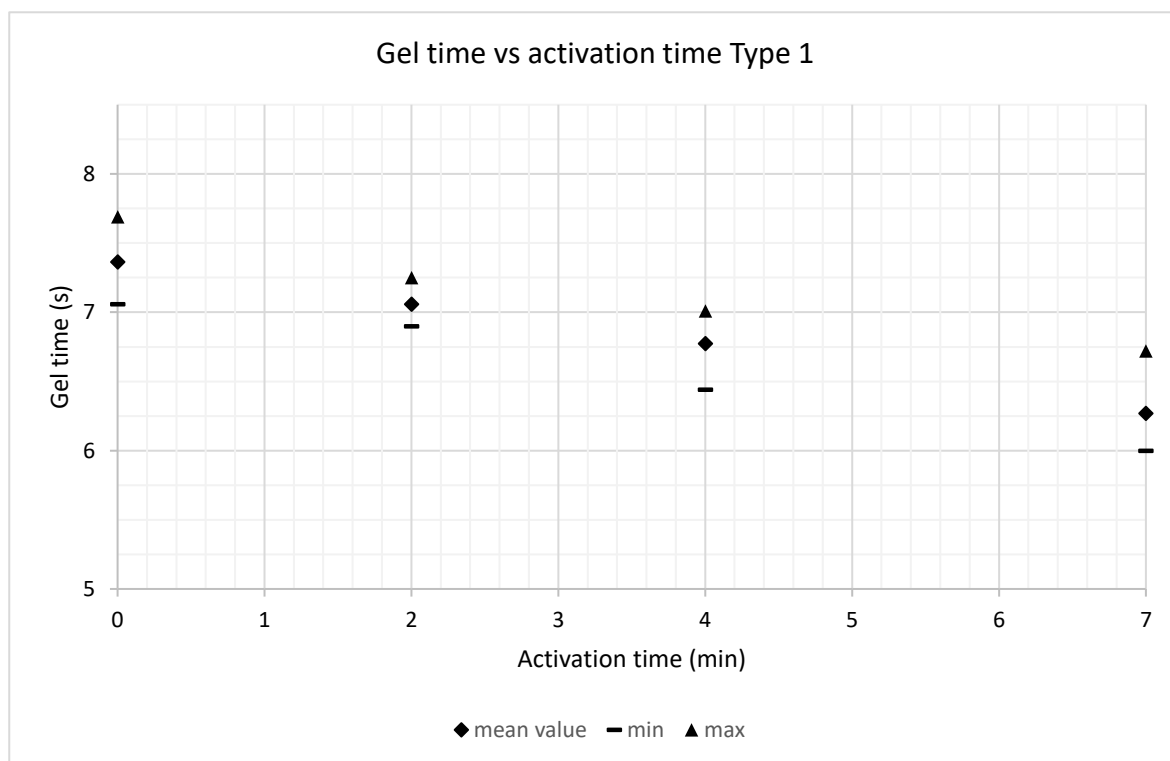


Figure 7.12: gel time vs time of activation Type 1.

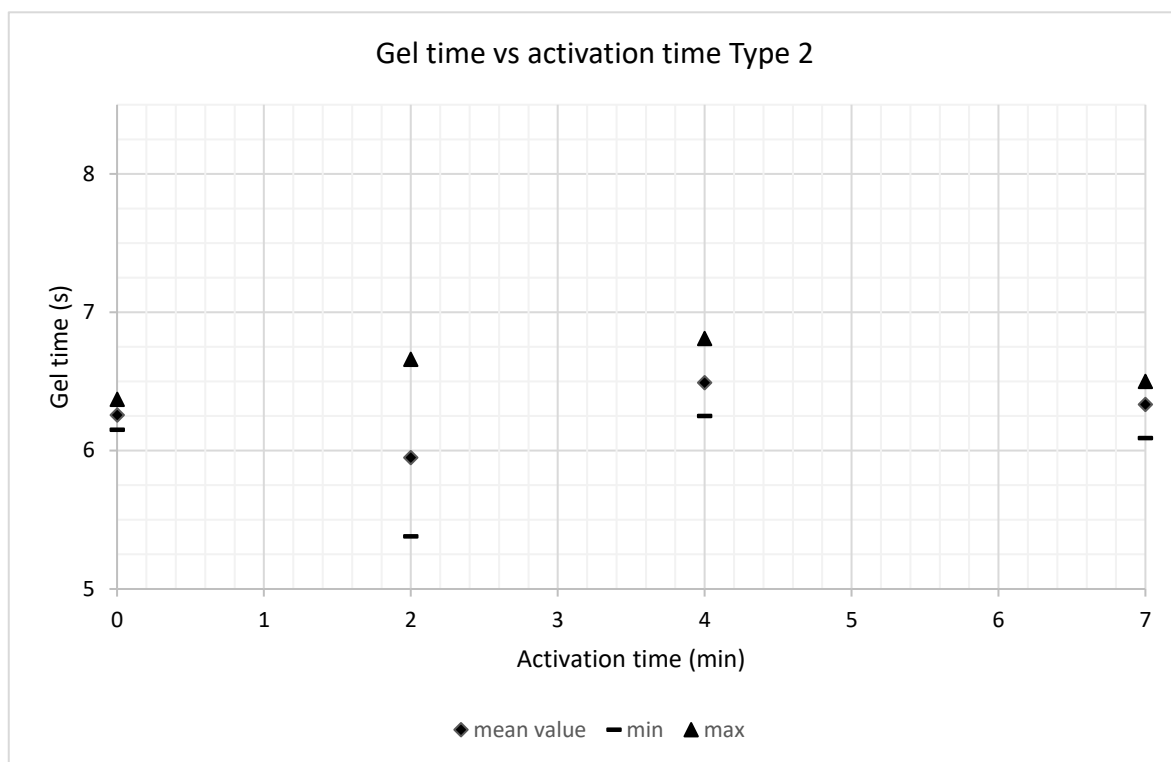


Figure 7.13: gel time vs time of activation Type 2.

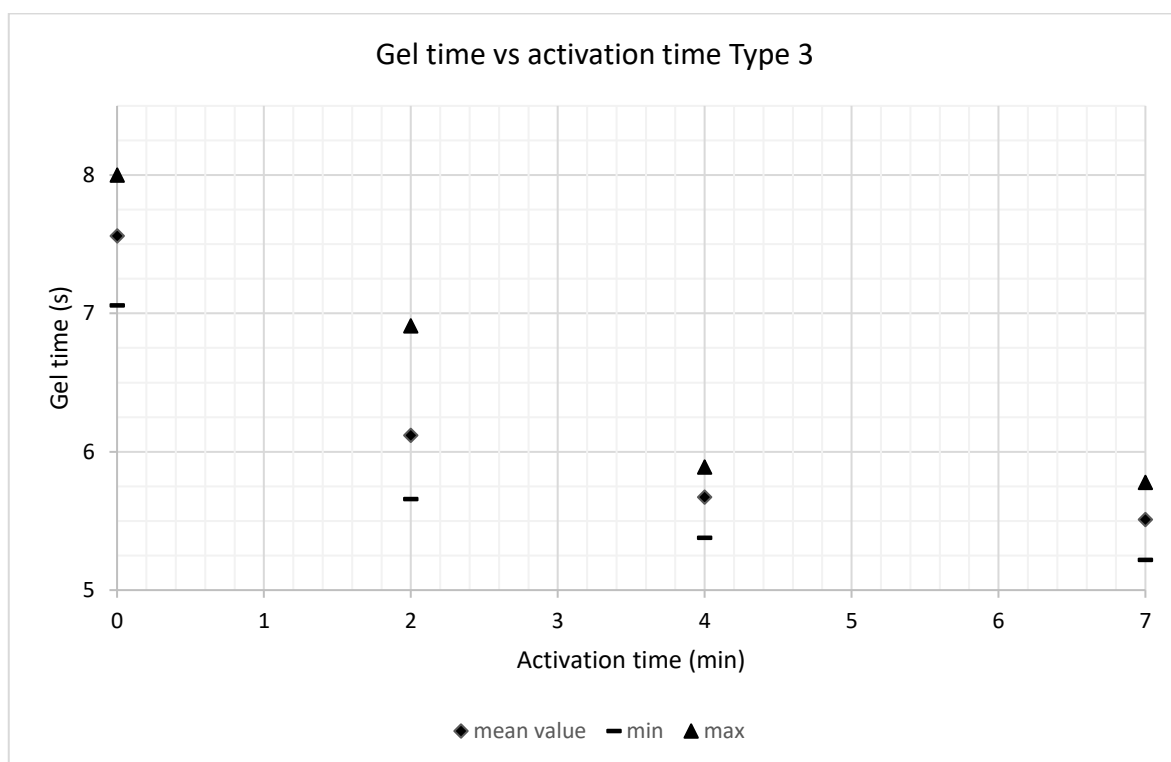


Figure 7.14: gel time vs time of activation Type 3.

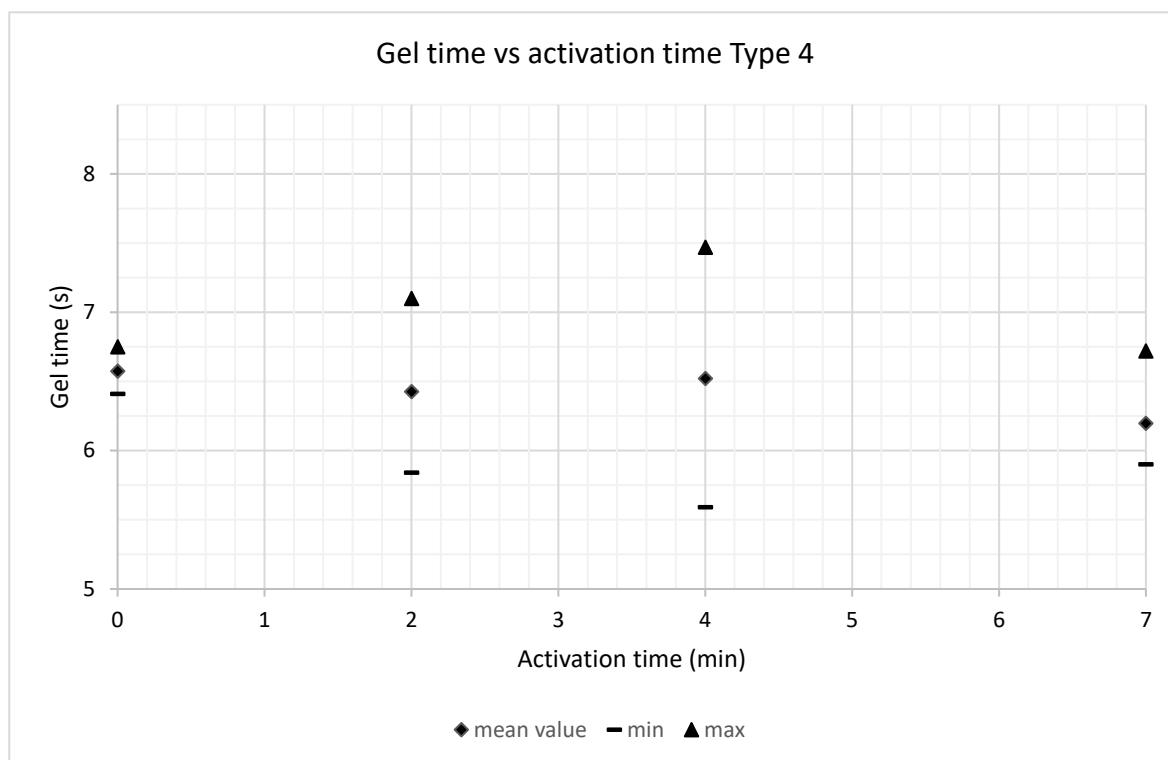


Figure 7.15: gel time vs time of activation Type 4.

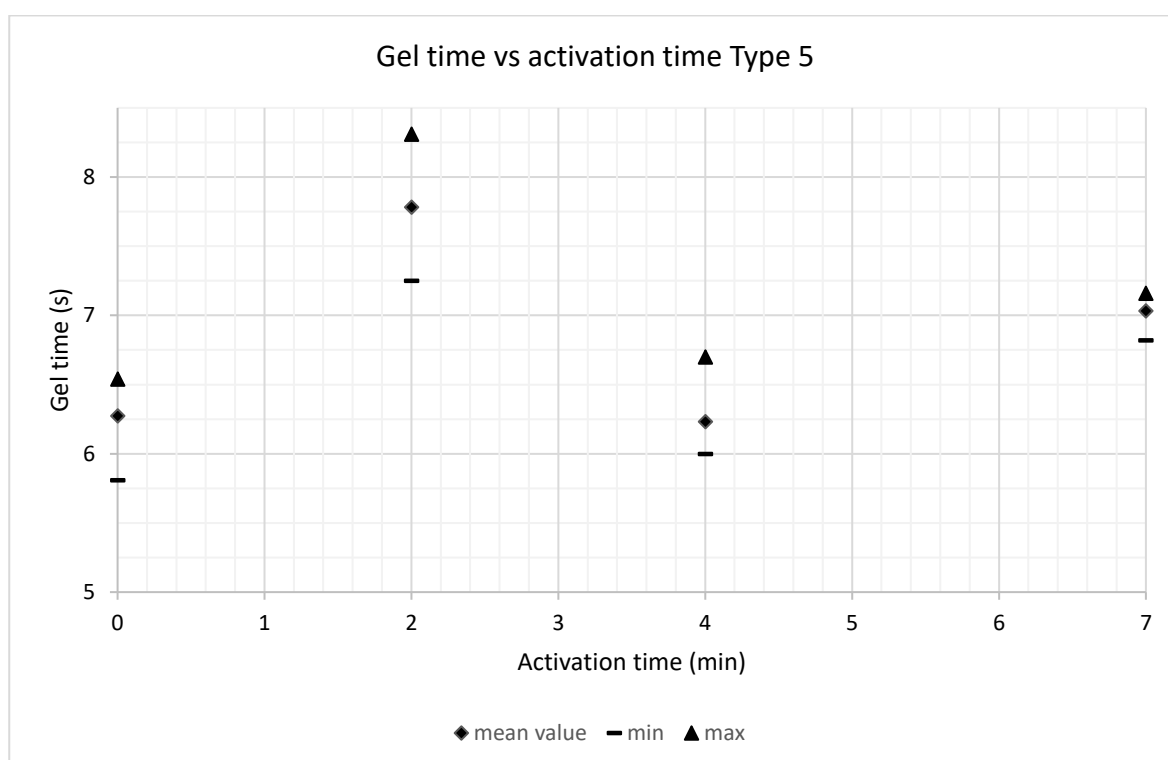


Figure 7.16: gel time vs time of activation Type 5.



## 7.5 Result of Surface Compressive Strength (SCS) test

From the analysis of data, there is an upward trend of SCS as the time of activation increases. The increasing of strength is more marked in samples cured 3 hours whilst samples cured 1 hour show a slight increase of the strength nevertheless the trend is more homogeneous. Type of bentonite has an influence on the strengths of the samples.

SCS values for samples with curing time of 3 hours are characterised by a high number of discarded values (values in quotation marks) where a certain number of them were caused by a not proper perpendicular penetration of the bit tool due to the achieved strength of sample surface. When the penetrometer is not maintained perpendicularly to the surface a rupture occurs laterally measuring a low SCS value. Samples with a curing time of 3 hours have a surface strength which made not easy the penetration operation, instead, samples cured 1 hour are not affected by this type of discarded values inasmuch the penetration is easy.

Room temperature and humidity, and temperature of mixture water influence strengths of samples:

- Type 1:
  - 1h and 3h SCS tests, with time of activation of 2 minutes, show strengths higher than expected because there was a room temperature higher than normal during the curing time of the samples.
- Type 2:
  - 1h SCS test, with time of activation of 2 minutes, shows strength lower than expected because the mixture water was cooler than normal.

- 1h SCS tests, with time of activation of 4 minutes, show strength higher than expected however it has not been possible ascertain the cause (or the causes).
- Type 3:
  - 1h and 3h SCS tests, with time of activation of 4 minutes, show strengths lower than expected because the samples were very wet during SCS due to a room humidity higher than normal during the curing time of the samples.
  - 3h SCS tests, with time of activation of 0' and 2 minutes, show strengths slightly lower than expected because of cold mixture water and wet samples, respectively.
  - 1h SCS 2<sup>nd</sup> test, with time of activation of 7 minutes, shows abnormal high strength on a sample, because this sample was obtained scraping the tank to recover enough component A causing a higher concentration of cement. Furthermore, minimum quantity of air bubbles was observed.
- Type 4:
  - Tests were carried out regularly.
- Type 5:
  - 3h SCS 2<sup>nd</sup> test, with time of activation of 7 minutes, shows strengths lower than expected because of cold mixture water.
  - 1h and 3h SCS tests, with time of activation of 0' and 4 minutes, show strengths lower than expected because there was a room temperature lower than normal during the curing time of the samples.

Hereinafter SCS values for samples cured 1 hour and 3 hours are illustrated. Values in quotation marks are affected by error such as air bubbles, wrong penetration, etc., even though they are shown, they were not considered in calculations. Spaces with slash mean absence of samples to assess, or discarded values affected by gross errors. Coloured values reported in graphs are affected by several problems specified case-by-case in captions (Table 7.13 - Table 7.65 and Figure 7.17 - Figure 7.23).

Table 7.13: SCS 1 h, Type 1, time of activation 0 minutes.

Time of activation 0 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
35.0	32.0	35.5	34.2	0.20	0.18	0.20	0.19
39.5	38.0	37.5	38.3	0.22	0.21	0.21	0.22
38.0	38.0	39.5	38.5	0.21	0.21	0.22	0.22
			37.0				0.21
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000186				standard deviation $\sigma$ (MPa): 0.0136

Table 7.14: SCS 1 h, Type 1, time of activation 2 minutes.

Time of activation 2 minutes* (* high room temperature)							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
59.5	53.5	50.5	54.5	0.33	0.30	0.28	0.31
56.5	58.5	63.0	59.3	0.32	0.33	0.35	0.33
67.5	62.5	64.5	64.8	0.38	0.35	0.36	0.36
			59.6				0.33
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000937				standard deviation $\sigma$ (MPa): 0.0306

Table 7.15: SCS 1 h, Type 1, time of activation 4 minutes.

Time of activation 4 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
51.0	43.5	45.5	46.7	0.29	0.24	0.26	0.26
52.5	53.0	45.0	50.2	0.30	0.30	0.25	0.28
57.5	56.5	56.5	56.8	0.32	0.32	0.32	0.32
			51.2				0.29
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000913				standard deviation $\sigma$ (MPa): 0.0302

Table 7.16: SCS 1 h, Type 1, time of activation 7 minutes.

Time of activation 7 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
50.0	50.0	53.5	51.2	0.28	0.28	0.30	0.29
59.0	57.5	62.5	59.7	0.33	0.32	0.35	0.34
62.0	63.0	65.0	63.3	0.35	0.35	0.37	0.36
			58.1				0.33
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00103				standard deviation $\sigma$ (MPa): 0.0320

Table 7.17: SCS 3 h, Type 1, time of activation 0 minutes.

Time of activation 0 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
164.5	171.0	"145.0"	167.8	0.92	0.96	"0.82"	0.94
161.5	188.0	175.0	174.8	0.91	1.06	0.98	0.98
188.5	167.5	159.0	171.7	1.06	0.94	0.89	0.96
			171.4				0.96
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.00403			standard deviation $\sigma$ (MPa): 0.0635

Table 7.18: SCS 3 h, Type 1, time of activation 2 minutes.

Time of activation 2 minutes* (* high room temperature)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
271.0	/	272.5	271.8	1.52	/	1.53	1.53
338.0	/	299.5	318.8	1.90	/	1.68	1.79
/	"280.0"	296.0	296.0	/	"1.57"	1.66	1.66
			295.5				1.66
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.0233			standard deviation $\sigma$ (MPa): 0.153

Table 7.19: SCS 3 h, Type 1, time of activation 4 minutes.

Time of activation 4 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
255.0	243.0	235.0	244.3	1.43	1.37	1.32	1.37
268.0	282.0	/	275.0	1.51	1.59	/	1.55
267.0	253.0	/	260.0	1.50	1.42	/	1.46
			259.8				1.46
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.00813			standard deviation $\sigma$ (MPa): 0.0902

Table 7.20: SCS 3 h, Type 1, time of activation 7 minutes.

Time of activation 7 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
241.5	256.5	284.0	260.7	1.36	1.44	1.60	1.47
271.0	267.5	261.0	266.5	1.52	1.50	1.47	1.50
270.5	269.5	/	270.0	1.52	1.51	/	1.52
			265.7				1.49
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.00494			standard deviation $\sigma$ (MPa): 0.0703

Table 7.21: SCS 1 h, Type 2, time of activation 0 minutes.

Time of activation 0 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
38.5	38.5	38.0	38.3	0.22	0.22	0.21	0.22
40.5	45.5	41.5	42.5	0.23	0.26	0.23	0.24
47.0	48.0	43.5	46.2	0.26	0.27	0.24	0.26
			42.3				0.24
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000464				standard deviation $\sigma$ (MPa): 0.0215			

Table 7.22: SCS 1 h, Type 2, time of activation 2 minutes.

Time of activation 2 minutes* (*cold mixture water)							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
36.5	34.0	34.0	34.8	0.21	0.19	0.19	0.20
24.5	26.5	21.0	24.0	0.14	0.15	0.12	0.13
20.5	19.5	21.5	20.5	0.12	0.11	0.12	0.12
			26.4				0.15
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00141				standard deviation $\sigma$ (MPa): 0.0375			

Table 7.23: SCS 1 h, Type 2, time of activation 4 minutes.

Time of activation 4 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
56.0	48.5	46.0	50.2	0.31	0.27	0.26	0.28
64.0	52.0	38.0	51.3	0.36	0.29	0.21	0.29
63.5	56.0	55.5	58.3	0.36	0.31	0.31	0.33
			53.3				0.30
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00217				standard deviation $\sigma$ (MPa): 0.0466			

Table 7.24: SCS 1 h, Type 2, time of activation 4 minutes (2<sup>nd</sup> test).

Time of activation 4 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
82.5	76.0	74.5	77.7	0.46	0.43	0.42	0.44
/	/	/	/	/	/	/	/
63.0	65.5	63.0	63.8	0.35	0.37	0.35	0.36
			70.8				0.40
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00207				standard deviation $\sigma$ (MPa): 0.0455			

Table 7.25: SCS 1 h, Type 2, time of activation 7 minutes.

Time of activation 7 minutes ( <i>air bubbles for “ ”</i> )							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
46.0	48.5	41.5	45.3	0.26	0.27	0.23	0.25
44.5	39.5	41.5	41.8	0.25	0.22	0.23	0.24
/	"29.5"	"29.0"	/	/	"0.17"	"0.16"	/
			43.6				0.24
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000355				standard deviation $\sigma$ (MPa): 0.0188

Table 7.26: SCS 3 h, Type 2, time of activation 0 minutes.

Time of activation 0 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
201.5	"182.0"	173.5	187.5	1.13	"1.02"	0.98	1.05
160.0	173.5	170.0	167.8	0.90	0.98	0.96	0.94
/	171.5	/	171.5	/	0.96	/	0.96
			175.6				0.99
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00612				standard deviation $\sigma$ (MPa): 0.0783

Table 7.27: SCS 3 h, Type 2, time of activation 2 minutes.

Time of activation 2 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
"211.0"	281.5	302.0	291.8	"1.19"	1.58	1.70	1.64
250.5	266.0	288.0	268.2	1.41	1.50	1.62	1.51
252.5	246.0	241.0	246.5	1.42	1.38	1.35	1.39
			268.8				1.51
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0157				standard deviation $\sigma$ (MPa): 0.125

Table 7.28: SCS 3 h, Type 2, time of activation 4 minutes.

Time of activation 4 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
286.5	"138.0"	232.5	259.5	1.61	"0.78"	1.31	1.46
237.5	295.0	271.5	268.0	1.34	1.66	1.53	1.51
246.0	229.5	/	237.8	1.38	1.29	/	1.34
			255.1				1.43
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0231				standard deviation $\sigma$ (MPa): 0.152

Table 7.29: SCS 3 h, Type 2, time of activation 7 minutes.

Time of activation 7 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
249.0	257.0	281.0	262.3	1.40	1.44	1.58	1.47
"238.0"	"202.0"	/	/	"1.34"	"1.14"	/	/
284.0	276.5	305.5	288.7	1.60	1.55	1.72	1.62
			275.5				1.55
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0129				standard deviation $\sigma$ (MPa): 0.114

Table 7.30: SCS 1 h, Type 3, time of activation 0 minutes.

Time of activation 0 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
34.5	35.5	37.5	35.8	0.19	0.20	0.21	0.20
28.0	27.5	27.0	27.5	0.16	0.15	0.15	0.15
30.0	32.0	32.0	31.3	0.17	0.18	0.18	0.18
			31.6				0.18
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000443				standard deviation $\sigma$ (MPa): 0.0211			

Table 7.31: SCS 1 h, Type 3, time of activation 2 minutes.

Time of activation 2 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
49.0	48.5	48.5	48.7	0.28	0.27	0.27	0.27
41.0	39.5	43.5	41.3	0.23	0.22	0.24	0.23
45.5	48.0	51.5	48.3	0.26	0.27	0.29	0.27
			46.1				0.26
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000511				standard deviation $\sigma$ (MPa): 0.0226			

Table 7.32: SCS 1 h, Type 3, time of activation 4 minutes.

Time of activation 4 minutes* (* very wet samples during SCS test)							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
35.5	34.5	35.5	35.2	0.20	0.19	0.20	0.20
34.5	33.5	32.5	33.5	0.19	0.19	0.18	0.19
41.0	39.0	38.0	39.3	0.23	0.22	0.21	0.22
			36.0				0.20
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000243				standard deviation $\sigma$ (MPa): 0.0156			

Table 7.33: SCS 1 h, Type 3, time of activation 4 minutes (2<sup>nd</sup> test).

Time of activation 4 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
34.5	35.0	35.5	35.0	0.19	0.20	0.20	0.20
41.0	47.5	40.0	42.8	0.23	0.27	0.22	0.24
42.0	44.0	39.5	41.8	0.24	0.25	0.22	0.24
			39.9				0.22
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000604				standard deviation $\sigma$ (MPa): 0.0246			



Table 7.34: SCS 1 h, Type 3, time of activation 7 minutes.

Time of activation 7 minutes ( <i>air bubbles for “ ”</i> )							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
47.0	42.5	48.0	45.8	0.26	0.24	0.27	0.26
52.0	48.0	50.0	50.0	0.29	0.27	0.28	0.28
"39.0"	"37.5"	42.5	42.5	"0.22"	"0.21"	0.24	0.24
			46.1				0.26
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000402				standard deviation $\sigma$ (MPa): 0.0201			

Table 7.35: SCS 1 h, Type 3, time of activation 7 minutes (2<sup>nd</sup> test).

Time of activation 7 minutes ( <i>abnormal strength values for “ ”</i> )							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
/	56.0	57.5	56.8	/	0.31	0.32	0.32
60.5	55.5	57.5	57.8	0.34	0.31	0.32	0.33
"140.5"	"130.0"	"111.5"	"127.3"	"0.79"	"0.73"	"0.63"	"0.72"
			57.3				0.32
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00012				standard deviation $\sigma$ (MPa): 0.0110			

Table 7.36: SCS 1 h, Type 3, time of activation 7 minutes (3<sup>rd</sup> test).

Time of activation 7 minutes ( <i>air bubbles for “ ”</i> )							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
"30.0"	37.0	38.5	37.8	"0.17"	0.21	0.22	0.21
58.0	53.5	55.5	55.7	0.33	0.30	0.31	0.31
57.0	57.0	50.5	54.8	0.32	0.32	0.28	0.31
			49.4				0.28
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00226				standard deviation $\sigma$ (MPa): 0.0475			

Table 7.37: SCS 3 h, Type 3, time of activation 0 minutes.

Time of activation 0 minutes* (* cold mixture water and air bubbles for “ ”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
135.0	137.5	126.0	132.8	0.76	0.77	0.71	0.75
129.5	125.5	125.0	126.7	0.73	0.71	0.70	0.71
131.0	126.0	"117.5"	128.5	0.74	0.71	"0.66"	0.72
			129.3				0.73
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000710				standard deviation $\sigma$ (MPa): 0.0266			

Table 7.38: SCS 3 h, Type 3, time of activation 0 minutes (2<sup>nd</sup> test).

Time of activation 0 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
/	/	159.0	159.0	/	/	0.89	0.89
173.5	154.5	170.5	166.2	0.98	0.87	0.96	0.93
201.5	225.5	229.5	218.8	1.13	1.27	1.29	1.23
			181.3				1.02
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0305				standard deviation $\sigma$ (MPa): 0.175			

Table 7.39: SCS 3 h, Type 3, time of activation 2 minutes.

Time of activation 2 minutes* (* wet samples during SCS test)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
146.5	142.5	146.0	145.0	0.82	0.80	0.82	0.82
139.0	127.5	/	133.3	0.78	0.72	/	0.75
175.5	177.0	178.0	176.8	0.99	0.99	1.00	0.99
			151.7				0.85
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0124				standard deviation $\sigma$ (MPa): 0.111			

Table 7.40: SCS 3 h, Type 3, time of activation 2 minutes (2<sup>nd</sup> test).

Time of activation 2 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
228.5	/	"208.0"	228.5	1.28	/	"1.17"	1.28
237.0	/	232.0	234.5	1.33	/	1.30	1.32
175.5	/	190.5	183.0	0.99	/	1.07	1.03
			215.3				1.21
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0244				standard deviation $\sigma$ (MPa): 0.156			

Table 7.41: SCS 3 h, Type 3, time of activation 4 minutes.

Time of activation 4 minutes* (* very wet samples during SCS test)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
115.5	128.5	132.5	125.5	0.65	0.72	0.74	0.71
110.5	123.5	105.0	113.0	0.62	0.69	0.59	0.64
122.0	/	127.0	124.5	0.69	/	0.71	0.70
			121.0				0.68
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00283				standard deviation $\sigma$ (MPa): 0.0532			

Table 7.42: SCS 3 h, Type 3, time of activation 4 minutes (2<sup>nd</sup> test).

Time of activation 4 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
224.0	212.5	219.5	218.7	1.26	1.19	1.23	1.23
/	217.5	213.0	215.3	/	1.22	1.20	1.21
209.5	/	/	209.5	1.18	/	/	1.18
			214.5				1.21
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000897				standard deviation $\sigma$ (MPa): 0.0300			

Table 7.43: SCS 3 h, Type 3, time of activation 7 minutes.

Time of activation 7 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
/	207.5	210.5	209.0	/	1.17	1.18	1.17
263.5	251.5	247.0	254.0	1.48	1.41	1.39	1.43
251.0	229.0	258.5	246.2	1.41	1.29	1.45	1.38
			236.4				1.33
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0146				standard deviation $\sigma$ (MPa): 0.121			

Table 7.44: SCS 1 h, Type 4, time of activation 0 minutes.

Time of activation 0 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
39.5	38.0	36.0	37.8	0.22	0.21	0.20	0.21
36.0	38.5	40.5	38.3	0.20	0.22	0.23	0.22
32.5	36.0	35.0	34.5	0.18	0.20	0.20	0.19
			36.9				0.21
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000193				standard deviation $\sigma$ (MPa): 0.0139			

Table 7.45: SCS 1 h, Type 4, time of activation 2 minutes.

Time of activation 2 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
57.5	62.0	55.5	58.3	0.32	0.35	0.31	0.33
53.5	52.0	51.0	52.2	0.30	0.29	0.29	0.29
61.5	68.5	63.5	64.5	0.35	0.39	0.36	0.36
			58.3				0.33
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00110				standard deviation $\sigma$ (MPa): 0.0332			

Table 7.46: SCS 1 h, Type 4, time of activation 4 minutes.

Time of activation 4 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
43.5	48.5	48.0	46.7	0.24	0.27	0.27	0.26
64.5	59.5	60.0	61.3	0.36	0.33	0.34	0.34
55.5	57.5	58.0	57.0	0.31	0.32	0.33	0.32
			55.0				0.31
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00148				standard deviation $\sigma$ (MPa): 0.0385			

Table 7.47: SCS 1 h, Type 4, time of activation 7 minutes.

Time of activation 7 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
58.0	58.0	58.5	58.2	0.33	0.33	0.33	0.33
52.0	46.5	52.5	50.3	0.29	0.26	0.30	0.28
59.5	54.5	57.5	57.2	0.33	0.31	0.32	0.32
			55.2				0.31
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000569				standard deviation $\sigma$ (MPa): 0.0239			

Table 7.48: SCS 3 h, Type 4, time of activation 0 minutes.

Time of activation 0 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
100.0	108.0	96.5	101.5	0.56	0.61	0.54	0.57
133.5	123.0	115.0	123.8	0.75	0.69	0.65	0.70
148.5	162.0	138.5	149.7	0.83	0.91	0.78	0.84
			125.0				0.70
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.0158	standard deviation $\sigma$ (MPa):		0.126

Table 7.49: SCS 3 h, Type 4, time of activation 2 minutes.

Time of activation 2 minutes (air bubbles for “ ”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
"163.0"	"146.0"	161.0	161.0	"0.92"	"0.82"	0.91	0.91
195.5	186.0	192.5	191.3	1.10	1.05	1.08	1.08
190.0	"144.5"	186.0	188.0	1.07	"0.81"	1.05	1.06
			180.1				1.01
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.00486	standard deviation $\sigma$ (MPa):		0.0697

Table 7.50: SCS 3 h, Type 4, time of activation 4 minutes.

Time of activation 4 minutes (air bubbles for “ ”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
185.5	202.0	198.0	195.2	1.04	1.14	1.11	1.10
210.5	205.0	"158.5"	207.8	1.18	1.15	"0.89"	1.17
223.5	214.5	"175.0"	219.0	1.26	1.21	"0.98"	1.23
			207.3				1.17
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.00473	standard deviation $\sigma$ (MPa):		0.0688

Table 7.51: SCS 3 h, Type 4, time of activation 7 minutes.

Time of activation 7 minutes (air bubbles for “ ”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
153.0	173.5	"139.5"	163.3	0.86	0.98	"0.78"	0.92
228.5	214.5	"196.0"	221.5	1.28	1.21	"1.10"	1.25
220.5	"185.0"	222.5	221.5	1.24	"1.04"	1.25	1.25
			202.1				1.14
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.0306	standard deviation $\sigma$ (MPa):		0.175

Table 7.52: SCS 1 h, Type 5, time of activation 0 minutes.

Time of activation 0 minutes* (* low room temperature and air bubbles for “”)							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
43.0	45.5	"40.5"	44.3	0.24	0.26	"0.23"	0.25
31.5	34.0	38.5	34.7	0.18	0.19	0.22	0.19
47.0	49.5	47.0	47.8	0.26	0.28	0.26	0.27
			42.3				0.24
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00138				standard deviation $\sigma$ (MPa): 0.0372			

Table 7.53: SCS 1 h, Type 5, time of activation 0 minutes (2<sup>nd</sup> test).

Time of activation 0 minutes (air bubbles for “”)							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
41.5	41.0	40.5	41.0	0.23	0.23	0.23	0.23
40.0	"38.0"	38.5	39.3	0.22	"0.21"	0.22	0.22
38.5	41.0	41.5	40.3	0.22	0.23	0.23	0.23
			40.2				0.23
variance $\sigma^2$ (MPa <sup>2</sup> ): 4.73 10 <sup>-5</sup>				standard deviation $\sigma$ (MPa): 0.00687			

Table 7.54: SCS 1 h, Type 5, time of activation 2 minutes.

Time of activation 2 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
58.5	60.0	40.0	52.8	0.33	0.34	0.22	0.30
55.0	50.0	77.0	60.7	0.31	0.28	0.43	0.34
/	/	/	/	/	/	/	/
			56.8				0.32
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00476				standard deviation $\sigma$ (MPa): 0.0670			

Table 7.55: SCS 1 h, Type 5, time of activation 4 minutes.

Time of activation 4 minutes* (* low room temperature)							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
51.5	55.5	46.5	51.2	0.29	0.31	0.26	0.29
56.0	63.0	62.0	60.3	0.31	0.35	0.35	0.34
48.5	57.0	48.0	51.2	0.27	0.32	0.27	0.29
			54.2				0.30
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00114				standard deviation $\sigma$ (MPa): 0.0338			

Table 7.56: SCS 1 h, Type 5, time of activation 7 minutes.

Time of activation 7 minutes							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
69.0	78.5	74.0	73.8	0.39	0.44	0.42	0.42
85.5	85.0	86.0	85.5	0.48	0.48	0.48	0.48
71.0	74.0	76.0	73.7	0.40	0.42	0.43	0.41
			77.7				0.44
			variance $\sigma^2$ (MPa <sup>2</sup> ):	0.00132			
					standard deviation $\sigma$ (MPa):		
					0.0364		

Table 7.57: SCS 3 h, Type 5, time of activation 0 minutes.

Time of activation 0 minutes* (* low room temperature and air bubbles for “”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
127.0	141.0	124.5	130.8	0.71	0.79	0.70	0.74
"125.5"	141.0	122.0	131.5	"0.71"	0.79	0.69	0.74
/	117.5	117.5	117.5	/	0.66	0.66	0.66
			126.6				0.71
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.00318	standard deviation $\sigma$ (MPa): 0.0564			

Table 7.58: SCS 3 h, Type 5, time of activation 0 minutes (2<sup>nd</sup> test).

Time of activation 0 minutes (air bubbles for “”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
220.5	"181.0"	254.0	237.3	1.24	"1.02"	1.43	1.33
314.0	/	/	314.0	1.77	/	/	1.77
/	/	/	/	/	/	/	/
			275.6				1.55
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0709	standard deviation $\sigma$ (MPa): 0.266			

Table 7.59: SCS 3 h, Type 5, time of activation 2 minutes.

Time of activation 2 minutes (not perfect penetration for “”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
"204.0"	243.0	311.0	277.0	"1.15"	1.37	1.75	1.56
301.0	284.5	293.0	292.8	1.69	1.60	1.65	1.65
270.5	"266.5"	275.0	272.8	1.52	"1.50"	1.55	1.53
			280.9				1.58
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0160	standard deviation $\sigma$ (MPa): 0.126			

Table 7.60: SCS 3 h, Type 5, time of activation 4 minutes.

Time of activation 4 minutes* (* low room temperature and air bubbles for “”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
"122.0"	175.5	183.0	179.3	"0.69"	0.99	1.03	1.01
150.0	152.0	143.5	148.5	0.84	0.85	0.81	0.83
189.0	189.0	223.5	200.5	1.06	1.06	1.26	1.13
			176.1				0.99
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0224	standard deviation $\sigma$ (MPa): 0.149			



Table 7.61: SCS 3 h, Type 5, time of activation 7 minutes.

Time of activation 7 minutes							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
223.5	/	268.5	246.0	1.26	/	1.51	1.38
305.0	330.5	332.0	322.5	1.71	1.86	1.87	1.81
/	/	/	/	/	/	/	/
			284.3				1.60
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0671	standard deviation $\sigma$ (MPa): 0.259			

Table 7.62: SCS 3 h, Type 5, time of activation 7 minutes (2<sup>nd</sup> test).

Time of activation 7 minutes* (* cold mixture water)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
175.5	/	166.0	170.8	0.99	/	0.93	0.96
233.5	227.0	/	230.3	1.31	1.28	/	1.29
229.5	205.0	268.5	234.3	1.29	1.15	1.51	1.32
			211.8				1.19
			variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0401	standard deviation $\sigma$ (MPa): 0.200			

Table 7.63: SCS 1 h, component A without bentonite.

Component A without bentonite							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
18.0	18.0	18.0	18.0	0.10	0.10	0.10	0.10
22.0	22.5	22.5	22.3	0.12	0.13	0.13	0.13
21.0	22.0	21.5	21.5	0.12	0.12	0.12	0.12
			20.6				0.12
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000128				standard deviation $\sigma$ (MPa): 0.0113			

Table 7.64: SCS 1 h, component A without bentonite (2<sup>nd</sup> test).

Component A without bentonite							
SCS 1 h (N)			Mean (N)	SCS 1 h (MPa)			Mean (MPa)
21.0	21.0	19.0	20.3	0.12	0.12	0.11	0.11
13.5	14.5	14.5	14.2	0.08	0.08	0.08	0.08
25.5	25.0	24.5	25.0	0.14	0.14	0.14	0.14
			19.8				0.11
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.000715				standard deviation $\sigma$ (MPa): 0.0267			

Table 7.65: SCS 3 h, component A without bentonite.

Component A without bentonite (air bubbles for “ ”)							
SCS 3 h (N)			Mean (N)	SCS 3 h (MPa)			Mean (MPa)
/	101.5	"87.5"	101.5	/	0.57	"0.49"	0.57
61.5	70.0	63.5	65.0	0.35	0.39	0.36	0.37
108.5	107.0	100.5	105.3	0.61	0.60	0.56	0.59
			90.6				0.51
variance $\sigma^2$ (MPa <sup>2</sup> ): 0.0145				standard deviation $\sigma$ (MPa): 0.120			

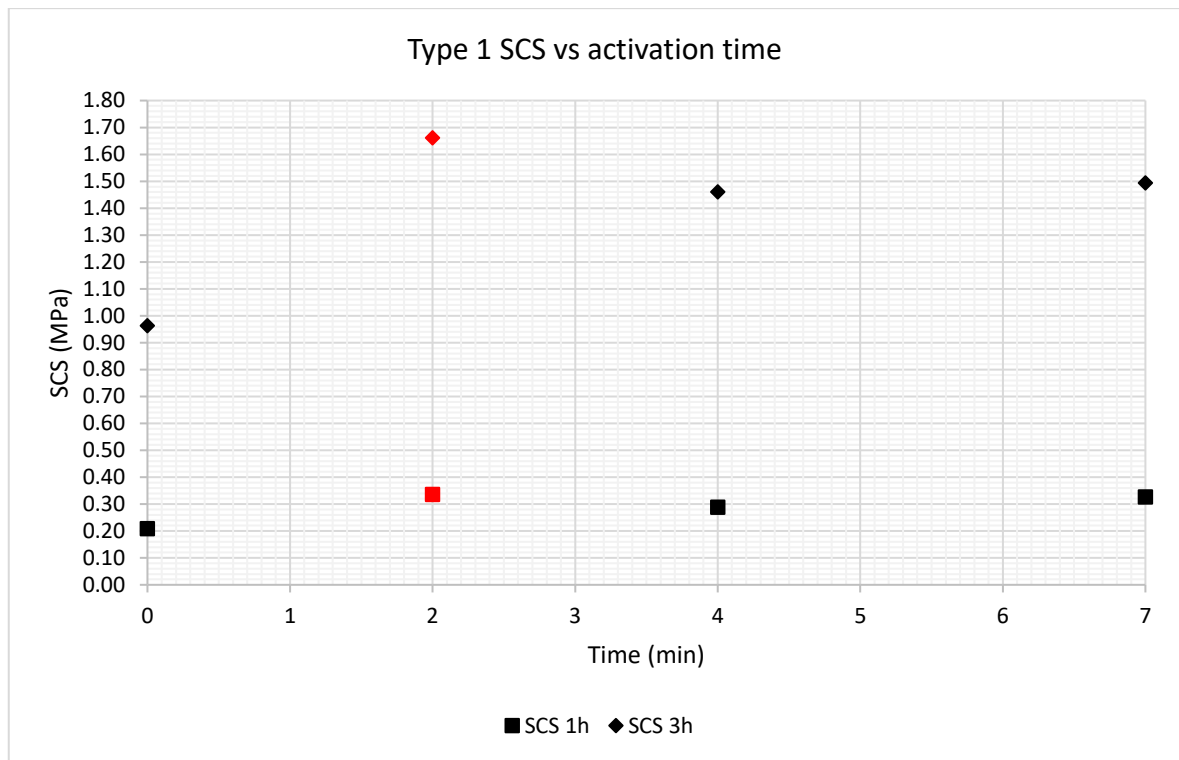


Figure 7.17: Type 1, SCS 1 h and 3 h vs time of activation. Red values: high room temperature during curing time.

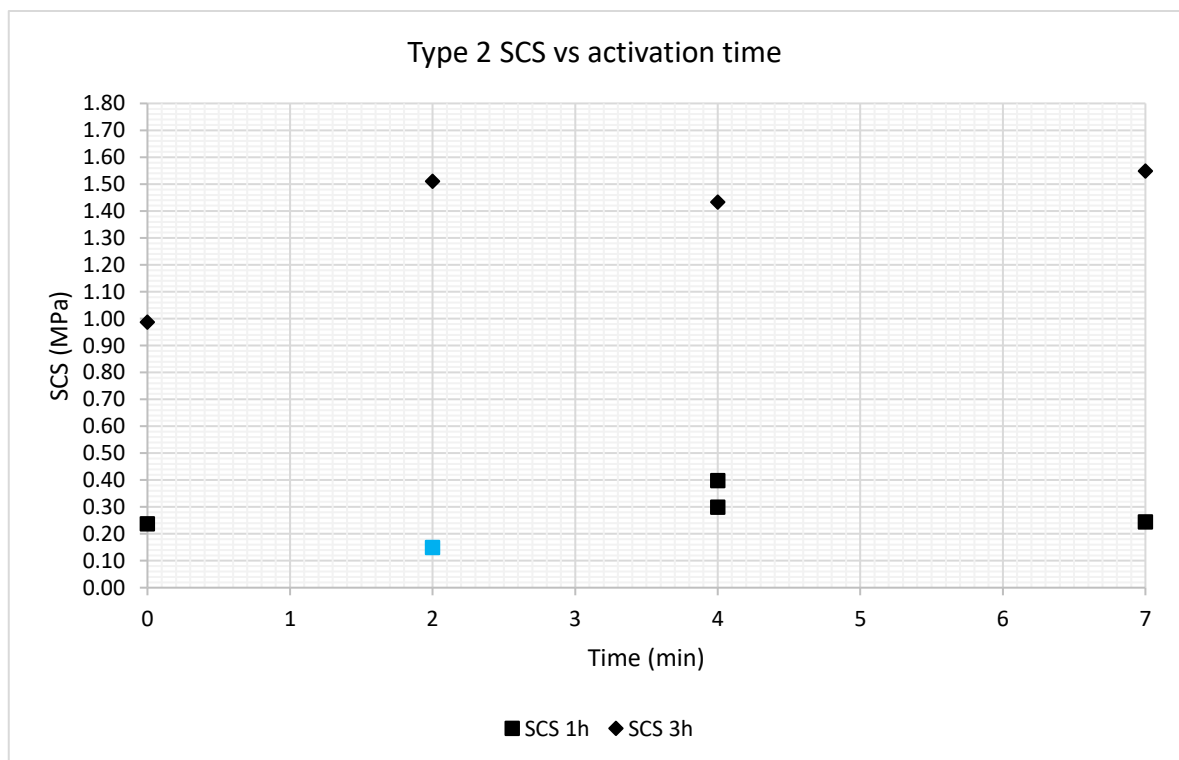


Figure 7.18: Type 2, SCS 1 h and 3 h vs time of activation. Blue value: cold mixture water.

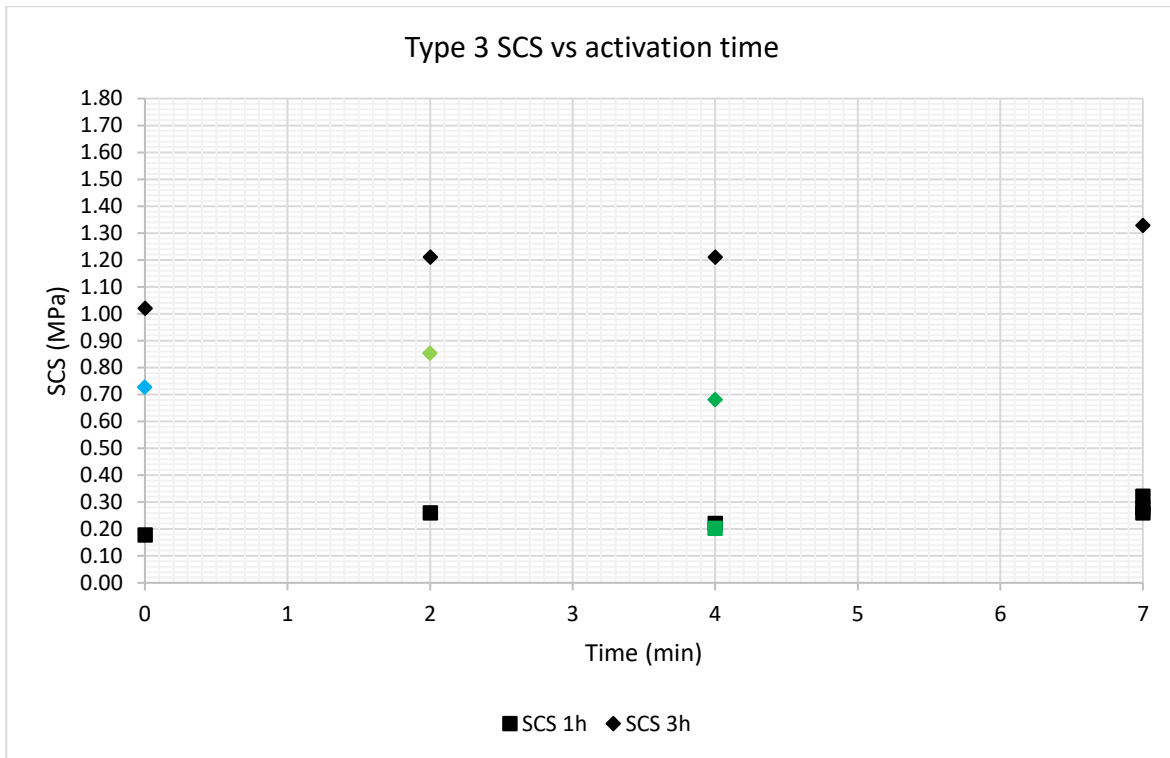


Figure 7.19: Type 3, SCS 1 h and 3 h vs time of activation. Green values: very wet samples during SCS test. Light green value: wet samples during SCS test. Blue value: cold mixture water.

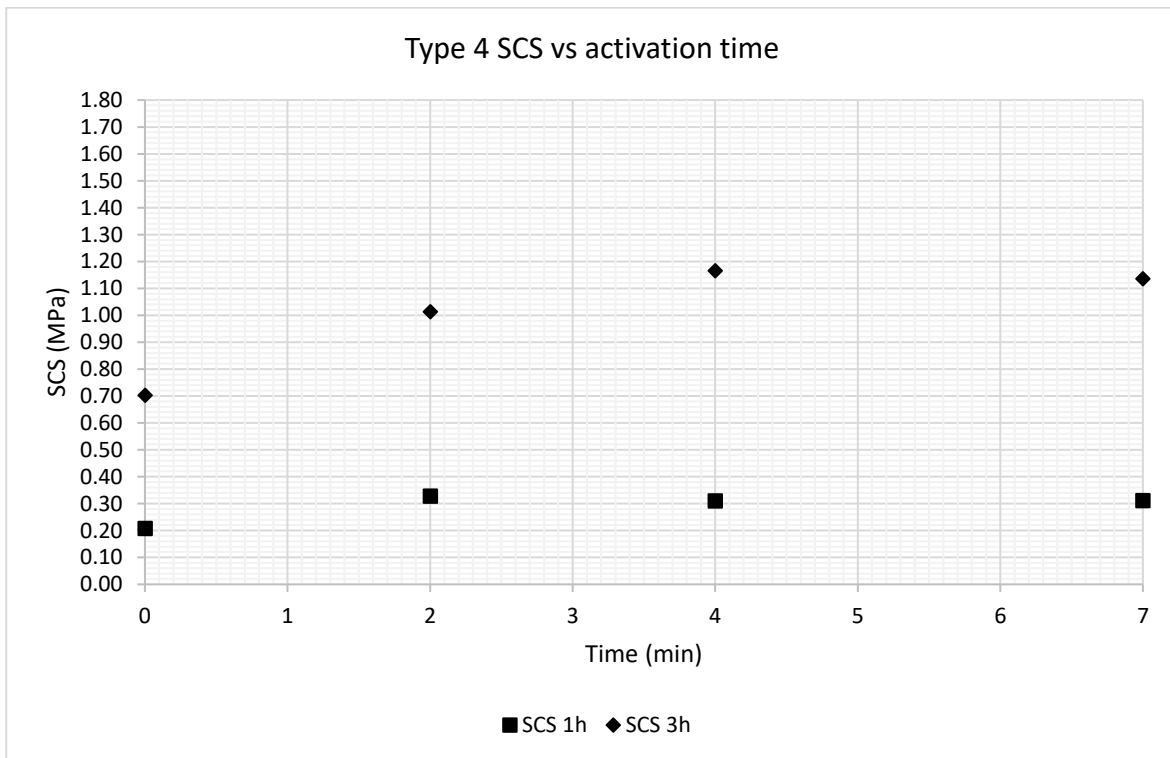


Figure 7.20: Type 4, SCS 1 h and 3 h vs time of activation.

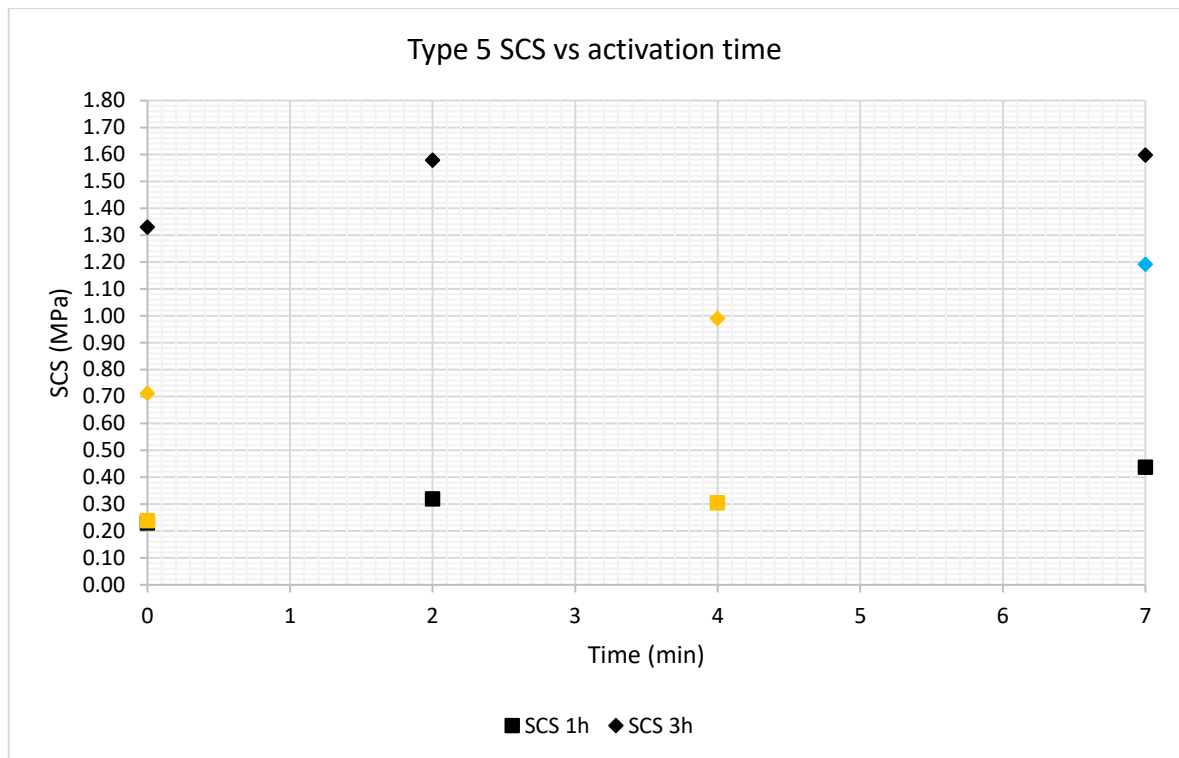


Figure 7.21: Type 5, SCS 1 h and 3 h vs time of activation. Blue value: cold mixture water. Orange values: low room temperature.

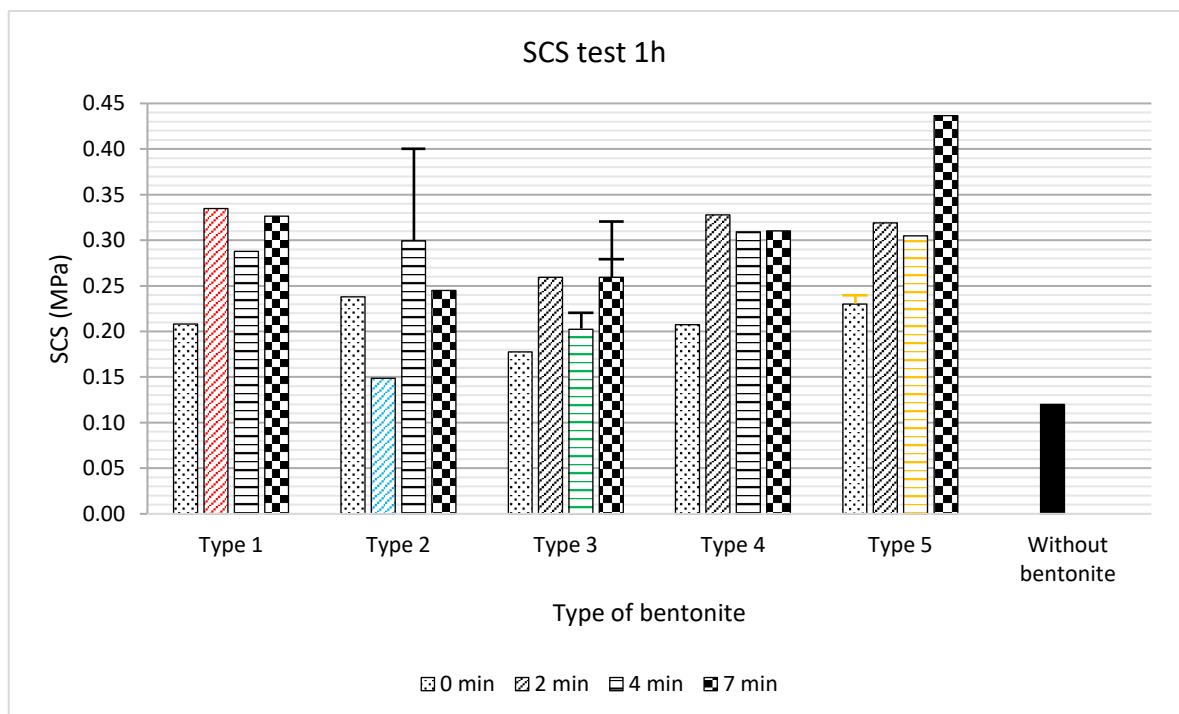


Figure 7.22: SCS 1h of all five types of bentonites. Red value: high room temperature during curing time. Blue value: cold mixture water. Green value: very wet samples during SCS test. Orange values: low room temperature.

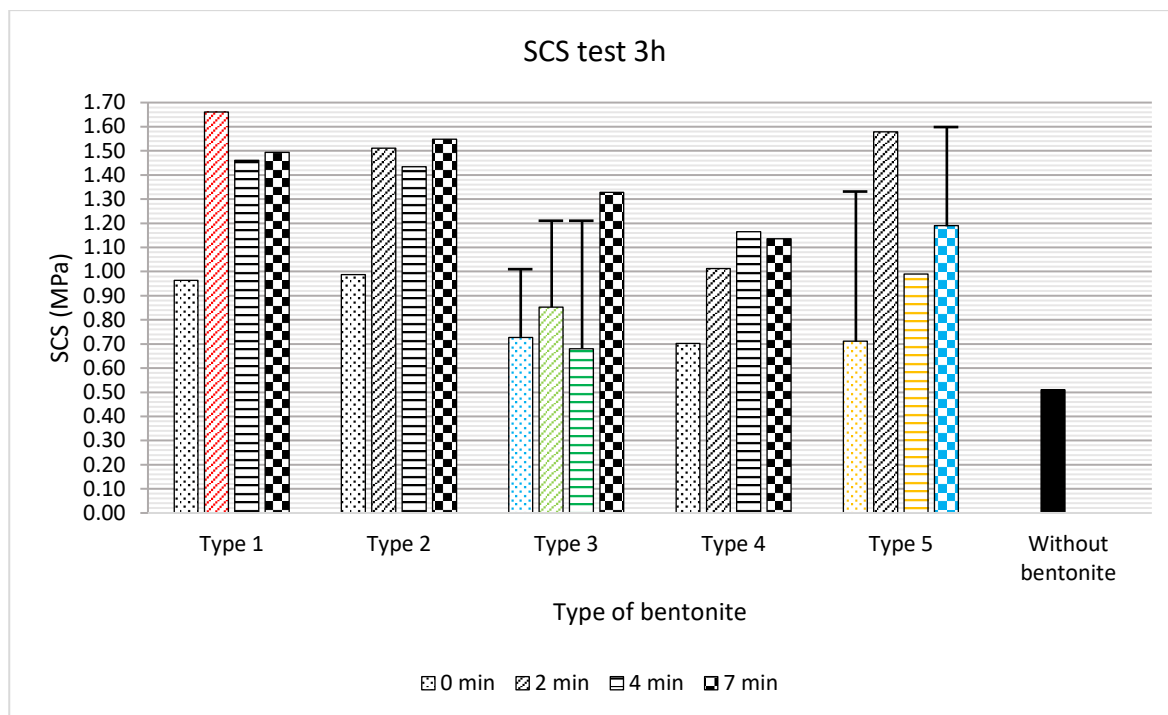


Figure 7.23: SCS 3h of all five types of bentonites. Red value: high room temperature during curing time. Blue values: cold mixture water. Light green value: wet samples during SCS test. Green value: very wet samples during SCS test. Orange values: low room temperature.

## 7.6 Result of liquid limit test

Liquid limit was obtained experimentally for two of the five tested bentonites, whilst for the other three, experimental data from Todaro et al., 2021, were employed. The liquid limit of all the five bentonites was obtained using the method explained in 6.4.4.1.

Type 2 has a liquid limit of 538.3 whilst Type 3 of 308.2 (Table 7.66 - Table 7.68 and Figure 7.24 - Figure 7.25).

Table 7.66: parameter of Type 2 obtained by Casagrande method.

n. of bumps	Tare (g)	Tare plus wet weight (g)	Wet wight (g)	Tare plus dry weight (g)	Dry weight (g)	Water content w (%)
53	2.20	9.75	7.55	3.46	1.26	499.2
37	2.14	13.84	11.70	3.97	1.83	539.3
18	2.16	12.78	10.62	3.79	1.63	551.5
10	2.16	11.56	9.40	3.57	1.41	566.7

Table 7.67: parameter of Type 3 obtained by Casagrande method.

n. of bumps	Tare (g)	Tare plus wet weight (g)	Wet wight (g)	Tare plus dry weight (g)	Dry weight (g)	Water content w (%)
52	2.36	15.17	12.81	5.69	3.33	284.7
41	2.30	17.47	15.17	6.20	3.90	289.0
23	2.27	12.95	10.68	4.83	2.56	317.2
12	2.20	15.32	13.12	5.26	3.06	328.8

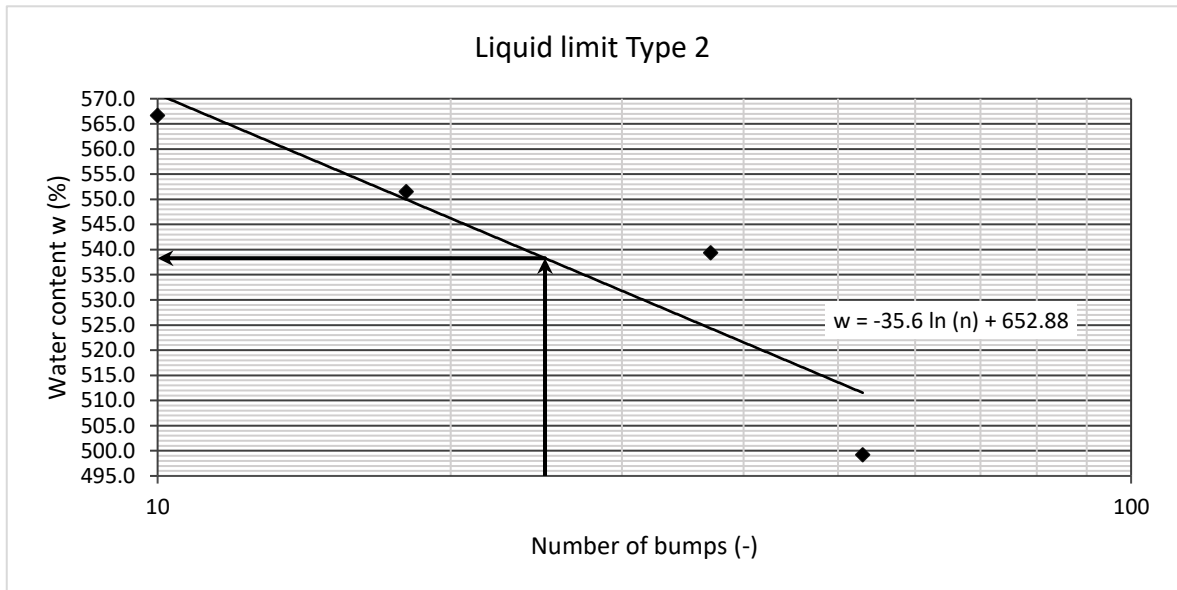


Figure 7.24: graph of liquid limit for Type 2.

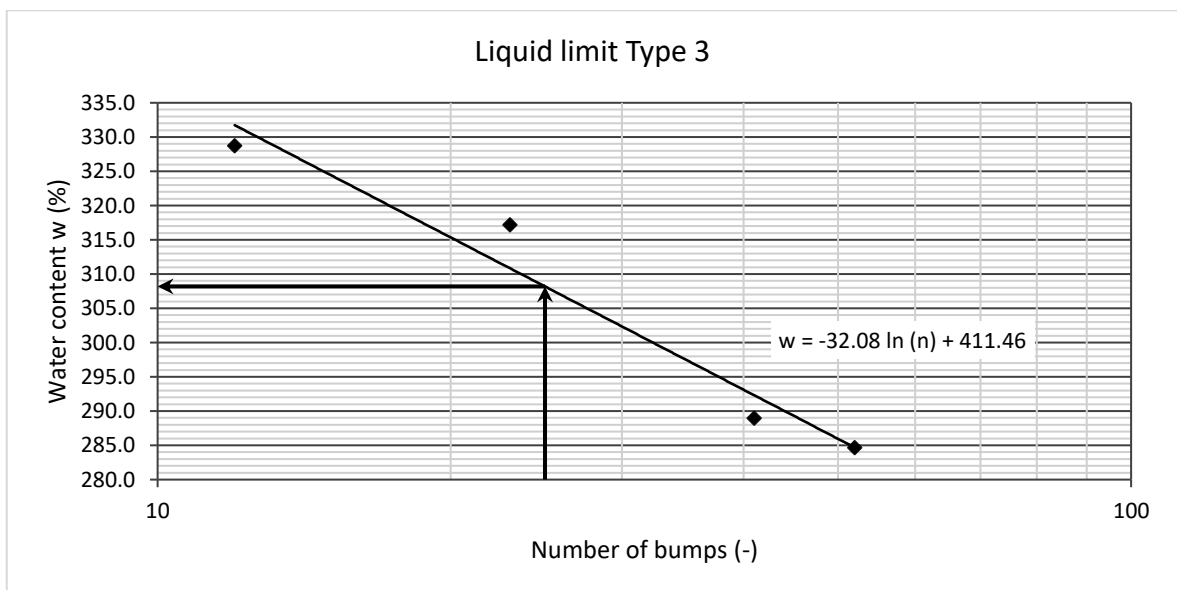


Figure 7.25: graph of liquid limit for Type 3.

Table 7.68: liquid limit of five types of bentonites.

Type of bentonite	Liquid limit (%)
Type 1	427*
Type 2	538.3
Type 3	308.2
Type 4	525*
Type 5	445*

*\*experimental data from Todaro et al. (2021)*

## 7.7 Result of swell index test

Swell index was experimental obtained for three of the five tested bentonites, whilst for the other two, experimental data from Todaro et al., 2021, were employed. The swell index of all the five bentonites was obtained using the method explained in 6.4.4.2.

The volume of the settled bentonite was calculated using the following proportion:

$$H : 250 \text{ ml} = h_{\text{mean}} : V \quad (7.1)$$

*H is the height of the graduated cylinder at 250 ml of capacity (mm);*

*h<sub>mean</sub> is the mean of the three heights (mm);*

*V is the volume of the settled bentonite (ml).*

The height of settled bentonite was calculated as mean value of three measurements took equidistantly along the perimeter of the graduated cylinder thus to take into account the uneven of settled bentonite (Table 7.69; Figure 7.26).

Table 7.69: swell indexes of five types of bentonites.

Type of bentonite	H (mm)	h1 (mm)	h2 (mm)	h3 (mm)	h <sub>mean</sub> (mm)	V (ml)	SWI (ml/2g)
Type 1	/	/	/	/	/	/	14.0*
Type 2	240	95	93	96	94.7	98.6	19.7
Type 3	240	64	57	62	61.0	63.5	12.7
Type 4	242	57	58	60	58.3	60.3	12.1 (13*)
Type 5	/	/	/	/	/	/	23.0*

*\*experimental data from Todaro et al. (2021)*



$H$  is the height of the graduated cylinder at 250 ml of capacity (mm);

$h_1$ ,  $h_2$ , and  $h_3$  are the heights of the settled bentonite (mm);

$h_{mean}$  is the mean of the three heights (mm);

$V$  is the volume of the settled bentonite (ml);

$SWI$  is the swell index (ml / 2g).

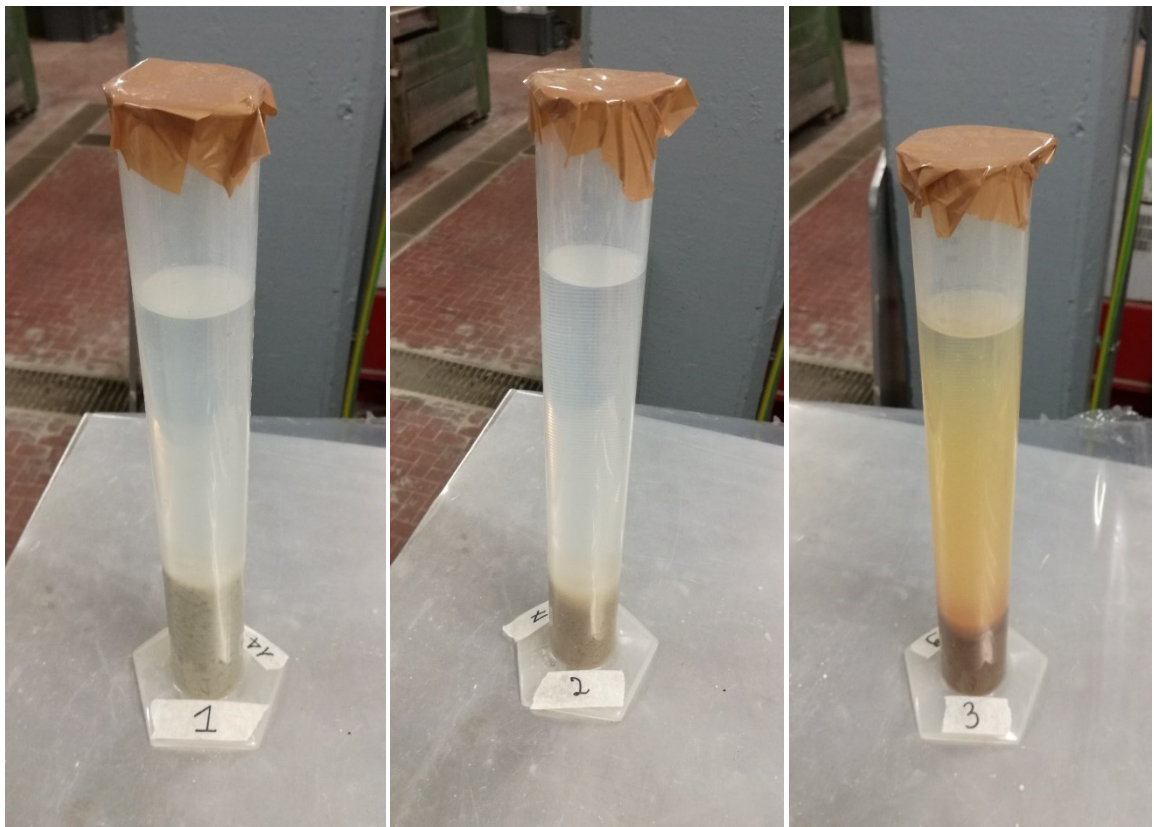


Figure 7.26: settled bentonite after the end of the test. Type 2 (left); Type 3 (centre); Type 4 (right).



## 8 ANALYSYS OF DATA

As illustrated in the previous chapter (7), type of bentonite and time of activation influence the behaviour of component A, gelled grout, and hardened grout.

To sum up the previous chapter (7), bleeding is related to time of activation and type of bentonite since greater time of activation leads to lower bleeding values, and type of bentonite influences the obtained values. Unit weight is not affected by type of bentonite neither by its time of activation because it remains constant whilst viscosity rises as time of activation increases and a relation is observed with the type of bentonite too. Gel time is not related with time of activation or type of bentonite since the difference of values is inside the interval of  $\pm 2$  seconds and gel time curves can be assumed as constants. Surface compressive strength rises as the time of activation increases and this increasing is more marked in samples cured 3 hours than samples cured 1 hour, furthermore, type of bentonite influences the reached strength values of samples.

### 8.1 Cross – analysis of data

Hereinafter a more detailed analysis is illustrated about the tested parameters.

#### 8.1.1 CROSS – ANALYSIS ON BLEEDING TEST

As regards bleeding, it has been observed that Type 2 and Type 5 have a very similar behaviour, with nearly equal values for activated component A and little difference for non-activated one. These two types of bentonites have high values of swell index (19.7 ml/2g for Type 2, 23 ml/2g for Type 5) whilst liquid limit is quite different (538.3 % for Type 2, 445 % for Type 5). We can assume, as general rule, that bentonites with high swell index parameters provide low bleeding values, furthermore, influence of liquid limit on the

behaviour should be considered too. Type 5 has the highest swell index, but its performance is nearly equal to Type 2, which have a lower swell index. Liquid limit of Type 2 is lower than Type 5 and this could balance their bleeding behaviour and explain why they have a very similar bleeding trend.

If we add the Type 4 to the analysis, we can observe that its behaviour is quite similar to Type 2 and Type 5, with slightly higher values of bleeding for activated component A and a similar value for non-activated one. Type 4 has the lowest swell index of all five bentonites (12.1 ml/2g) and the second highest liquid limit (525%). Analysing these three types of bentonites it is possible to define that bentonite with higher swelling index and higher liquid limit achieve better performance on bleeding tests. And that, if a bentonite has a low swell index, a high liquid limit can help to achieve good performance too.

According to these conditions, Type 3, which has the lowest liquid limit value (308.2%) and the second lowest swell index (12.7 ml/2g), should show a poor bleeding test, instead, its bleeding test is better than Type 1 which have both higher swell index and liquid limit (14 ml/2g, 427%). Finally, it is not possible to assess any correlation between the bleeding behaviour of component A with the swell index nor with liquid limit of bentonite.

#### 8.1.2 CROSS – ANALYSIS ON VISCOSITY TEST

In viscosity results, has been observed that Type 1, Type 3, and Type 4 are characterized by a similar flow time trend with values ranging in the interval 29.5 – 31.5 seconds for activated component A, whilst values for non-activated component A are about equal to 28.5 seconds. Type 2 and Type 5 have a similar behaviour, even though different by the previous one, where flow time values for activated component A ranging in the interval 33 – 34 seconds, whilst values for non-activated component A are about equal to 29 seconds. Analysing the parameters of the bentonite, such as the liquid limit and the swell index, has

been observed a relation with viscosity and swell index. Type 1, Type 3, and Type 4 have a similar swell index respectively of 14 ml/2g, 12.7 ml/2g, and 12.1 ml/2g whilst Type 2 and Type 5 have a higher swell index, respectively of 19.7 ml/2g and 23 ml/2g. It seems that a higher swell index of the bentonite leads to a higher viscosity of component A, even though it has no influence if bentonite is not activated.

#### 8.1.3 CROSS — ANALYSIS ON SURFACE COMPRESSIVE STRENGTH (SCS) TEST

Analysing the result of SCS test at 1 hours and 3 hours, no relation has been observed with swell index or liquid limit.

Before to analysis the data, take into account to reduce strength values for high room temperature, and to increase strength values for low room temperature, cold mixture water, and wet sample.

In 1-hour SCS test, the highest strength has been achieved by Type 5, followed by Type 4 and Type 1. Lowest values have been observed for Type 2 and Type 3.

In 3-hour SCS test, the highest strength has been achieved by Type 5, followed at close distance by Type 2 and Type 1. Lowest values have been observed for Type 4 and Type 3.

Type 5 has the highest swell index, whilst Type 3 has the lowest liquid limit and the second lowest swell index. Except for these two bentonites, it is not possible to define a relation with the swell index or the liquid limit to estimate the SCS of hardened grout. It is confirmed that hardened grouts have much higher strength if component A is activated and there is a slightly rise of the strength as the time of activation increases.

Samples of two-component grout without bentonite show very poor values of surface compressive strength. It could be speculated that bentonite works as a filler improving the mechanical strength, furthermore, its absence entails the loss of water absorption, and this

might lead to a higher content of water which might be responsible to the drop of surface compressive strength.

## 9 CONCLUSION

The bentonite is one of the ingredients used for the component A production in relation to the two-component grout technology. This work is based on a wide test campaign performed in laboratory where the role of the bentonite (and its activation) in the component A properties (unit weight, bleeding, and viscosity), its influence on the gel time and on the hardened grout (SCS) have been investigated. It has been observed that unit weight of component A and gel time of gelled grout are constant and are not in relation with time of activation or type of bentonite or properties of bentonite such as liquid limit and swell index.

Bleeding of component A is strongly affected by time of activation. Increasing the activation time, the bleeding tends to decrease. However, no unique relations have been observed related to the type of bentonite or properties of bentonite such as liquid limit or swell index. It can be stated that different bentonites exhibit different bleeding trend, but unfortunately the experimental way it seems the only procedure in order to understand the real aptitude of a certain bentonite to prevent the bleeding phenomenon.

Viscosity of component A is characterised by an upward trend as time of activation increases, furthermore, it is also a function of type of bentonite since it has been observed that bentonites with higher swell indexes lead to higher viscosity.

Surface compressive strength (SCS) of hardened grout, regardless the curing time, is correlated with time of activation. Low strength values of hardened grout have been observed when component A is non-activated, and much higher strength values if component A is activated. Strength slightly rises as time of activation increases from 2 minutes to 7 minutes. The strong innovation observed is related to the crucial role played

by bentonite in the development of strength in two-component grout at short curing time. It can be speculated that it works as a filler and might absorb water and consequently reducing the water content. This last statement should be verified by further research studies.



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