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Abstract

Cement is the main material used to ensure well integrity throughout the life of the well. It is among other things used for zonal isolation, casing support and permanent and temporary abandonment.
Improper cement design can result in leakages and reduced well integrity. Leakages from wells and reservoirs in the oil and gas industry is a major focus for both economic and environmental issues. Therefor it is important to be able to recreate the cement properties that are developed in the laboratory, in the field.

The cement is designed in the laboratory, but the challenge is to reproduce the same properties in the field. Research indicates that the mixing procedure and the mixing equipment affects both the cement slurry and the cured cement.

In this study we have investigated how total mixing time, shear rate and mixing energy added, affected the cement slurry and the cured cement. We built a downscaled 15 liters mixing unit. A NORCE cementing project of 327 - 546 liters was our full-scale reference, and we also used the API standard laboratory mixing as a reference. We used the same cement recipe for all experiments, a basic Class G with a minimum of additives. We measured UCS (unconfined compressive strength), transit time with UCA (ultrasonic cement analyses), rheology and free water.

We looked at dimensional analysis as a possible scaling method. We focused on two dimensionless groups to see if any of these can be used as a scaling reference.
We hope this will highlight the mixing process, and how it can be scaled up and down.

We did find that there is not a clear correlation between mixing energy and cement properties, and we did not find any clear correlation between the dimensionless groups and the cement properties. But we did find the cement to be quite robust. The strength and the free water measurements was similar for mixing energy ranging from 6 kJ/kg to 116kJ/kg, hence not affected by the mixing energy.

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Content

List of figures

List of tables

Abbreviations

API	American Petroleum Institute
kW	Kilo Watts
LPM	Liters Per Minute
PPA	Permanent Plug and Abandonment
PV	Plastic viscosity
RPM	Rotations Per Minute
SD	Standard Deviation
SDS	Safety Data Sheet
SME	Specific Mixing Energy
UCA	Ultrasonic Cement Analyzer
UCS	Unconfined Compressive Strength
W	Watts
YP	Yield point

Chapter 1. Introduction

1.1 Background and purpose of this thesis. Cement and well integrity

The integrity of a well, throughout its lifetime, highly depends on the cement job. Cement is the main material used to ensure well integrity, the cement is among other things used for zonal isolation and permanent and temporary abandonment. The cement´s properties, like strength, flexibility, and viscosity, depends on the cement recipe and the mixing procedure.

Zonal isolation is a hydraulic seal between the casing and the formation. The cement will bond with the casing and the formation and create a plug to prevent fluid and gas leaking from one zone to another. After each section of the well has been drilled, the drill string is removed, and a casing string a bit smaller than the section, is placed in the well. Cement slurry of the correct properties is pumped through the annular space between the casing and the formation, to the desired location (fig. 1). The well is then shut in for a time to allow the cement to cure and form a seal. The next activity will then be completion work or continued drilling to a deeper section of the well (Nelson & Guillot, 2006).

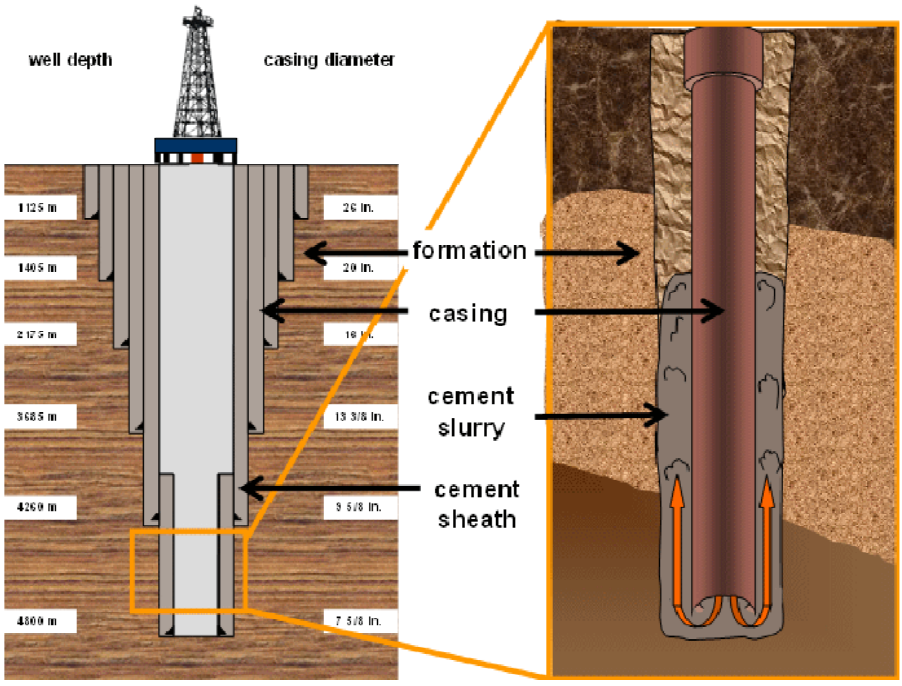


Figure 1: Illustration of cement slurry pumped to the annular space(Oil Well Sketch, 2015, by Drilling Cource).

Permanent and temporary abandonment is done when the well is no longer profitable to produce or inject from, and a cement plug is placed in the well to prevent leakages. This is done by placing cement inside the wellbore to create a plug to seal the well from the rest of the environment.

Approximately 15% of cement jobs fail, and this is a great cost in the oil and gas industry and a risk for the environment (Nelson & Guillot, 2006).

There are 3 common cement failures (Nelson & Guillot, 2006):

- Cracking, caused by fluctuation in pressure or temperature. The cement sheath expands and contract, this can cause stress gradients that can cause cracks in the cement. The stronger and more flexible the cement is, the more stress it can endure.
- Debonding, the bond between the cement and the formation or casing fails. Debonding can be caused by improper cement job, improper cement design, subsidence and vibrations as the reservoir depletes, cement shrinkage over time, vibrations due to reservoir stimulation, fluctuations in temperature and pressure.
- Shear failure, failure of the cement sheath. Shear failure can be caused by increased stress around the wellbore due to reservoir depletion or vibrations in the formation.

A schematic overview of possible leak paths is shown in figure 2 below.

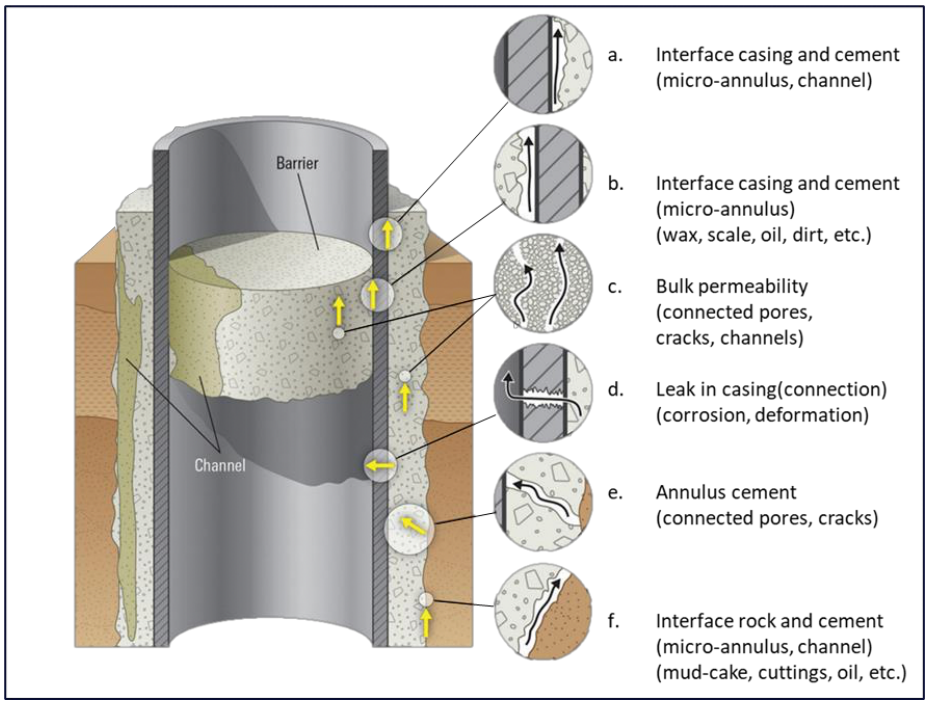


Figure 2: (Schematic of wellbore cross section including casing, annular barrier, primary cement sheath, cement plug inside casing, and possible leak paths, 2022, by Kamali)

There are 2 primary reasons cement failures: (Nelson & Guillot, 2006)

- Improper cement design or improper cement job.
- Wellbore depletion due to pressure decrease in the reservoir.

As reservoir fluids are produced, the pore pressure in the reservoir might decreases. This depletion can result in changes in the horizontal stresses and can affect surrounding formation and wellbores (Addis, 1997).

We have focused on cement design in this paper, more specific the mixing of the cement slurry.

Improper cement design can result in leakages and reduced well integrity. Leakages from wells and reservoirs in the oil and gas industry is a major focus for both economic and environmental issues. Therefor it is important to be able to recreate the cement properties that are developed in the laboratory, in the field.

Important properties of cement slurry are rheology, thickening time, free water, and fluid loss. Important properties of cured and solidified cement are unconfined compressive strength (UCS) and Young´s modulus. We will look more into these properties in chapter 2.3.

In this research we have measured rheology, free water, UCS, Young´s modulus and transit time. Transit time is the time an electrical signal travels through the cured cement. By measuring the transit time, we indirect measure the setting time, and this is closely related to the thickening time.

Cement is designed and developed in the laboratory, but the main challenge is the ability to produce similar cement properties in the field. This may be caused by the difference in mixing conditions. The mixing conditions is a key factor in the cement design and can strongly affect the properties of the cement. Both the mixing equipment and procedures in the laboratory and in the field differs quite a bit (Saleh & Teodoriu, 2021; Teodoriu et al., 2015).

In the field slurries are typically mixed in a mixing tank coupled to a pumping unit and an agitator. The slurry is mixed by circulation and agitation. In the laboratory slurries are mixed in a blender. The main difference between the field and laboratory mixing is the shear rates of the equipment, and the volume of slurry in the mixer.

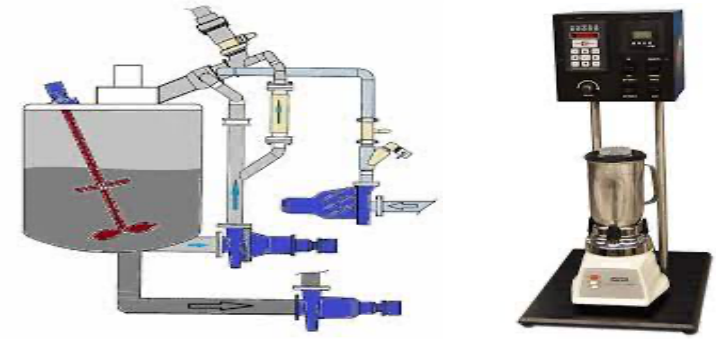


Figure 3: Schematic of a typical field mixer versus a typical blender used to mix slurries in the field.

The main concept to replicate laboratory cement in the field, is to replicate the specific mixing energy (SME). This is called the mixing energy method. The mixing energy method is built on the theory that if the effective energy per unit mass (SME) put into the mixing process is similar, the properties of the cement will be similar. Research has indicated that this is not always the case (Saleh et al., 2019; Saleh & Teodoriu, 2021; Teodoriu et al., 2015).

In this paper we explored how total mixing time, grade of shear rate and mixing energy added affects the cement slurry and the cured cement.
We looked at dimensional analyzes as a possible scaling method. Upscaling and downscaling of a process is based on similarity, both geometric similarity, for example a perfectly downscaled 1:100 copy of a mixing unit, boat or rig, and dynamic similarity by using identical dimensionless groups. This is a common approach for testing small-scales boats or riggs in an indoor pool. We wanted to look at the possibility to use the same scaling method for cement mixing.

NORCE research center built a 1000 liters field cement mixing unit, and we have been able to get logged data and cement slurry samples from their mixing sessions. We measured cement properties of the cement slurry and hardened cement samples, and we have used this as our full-scale reference.

We built a downscaled 15 liters mixing unit in the laboratory at UiS, based on a NORCE cementing project. We varied the mixing time, grade of shear rate and mixing energy and measured resulting cement properties. We also used an API standard laboratory procedure for a small-scale reference (American Petroleum Institute, 2013).

We have compared UCS (unconfined compressive strength), UCA (ultrasonic cement analyses), rheology and free water from the different mixing session, to see if there is any correlation between the cement properties and the dimensionless groups.

We focused on two dimensionless groups to see if any of these can be used as a scaling reference. We hope this will give us some focus on the mixing process, how it can be scaled

up and down, and give some guidelines to later full-scale projects where concise and reproducible properties can be achieved.

We hope to get a better understanding of how to adjust the mixing procedures to improve the correlation between slurry properties in the laboratory and in the field.

1.2 Previous research

Orban et al. (1986) brought forth one of the first theories on the relationship between cement mixing and cement slurry quality. They compared properties of cement slurries prepared under laboratory mixing conditions, with cement slurries prepared under field mixing conditions. They looked at the relationship between the specific mixing energy and cement slurry properties like rheology, free water, fluid loss, thickening time, and compressive strength. In this research they concluded that "Similarity between drastically different mixing scales and procedures was proven to be possible by summing the mechanical work provided by the mixing devices in the laboratory and in the field. Optimum cement slurry quality is generally obtained for a mixing energy close to the one corresponding to the API laboratory procedure." (Orban et al., 1986, p. 5)

This research also provided us with a formula for mixing energy:

$$\frac{E}{M} = \frac{kv^2t}{V} \qquad (1)$$

$\mathrm{E} = \text{mixing energy (kJ)}$
$\mathrm{m} = \text{mass of slurry (kg)}$
$\mathrm{k} = \text{a constant experimentally}$
$\mathrm{v} = \text{rotational speed (rad/ sec)}$
$\mathrm{t} = \text{time (seconds)}$
$\mathrm{V} = \text{slurry volume } (\mathrm{m}^3)$

The first edition of the API laboratory procedure came out in 1982 and has since been updated several times. Based on the findings of Orban and his team, American Petroleum Institute (2013) introduced a theory that stated that important properties like rheology, free water, thickening time, fluid loss and compressive strength could all be tied to the specific mixing energy, SME. This was termed the mixing energy concept, and the theory stated that when the mixing energy added a slurry is the same, then the following slurry properties will

be alike, regardless of the difference in equipment and procedure (Saleh & Teodoriu, 2021; Teodoriu et al., 2015).

Further on in 1990 Vidick (1990) considered both the physical and physicochemical aspects of the mixing process and divided the mixing process into two parts. A mechanical process where the dry particles are mixed in, deflocculated and wetted, and a physicochemical process that consists of the dissolution of the particles, the formation of supersaturated solutions and the precipitation of cement hydrates. His experiment consisted of several laboratory mixing sessions were the volume and cement recipe were constant, but with varied mixing time or rotational speed, hence changing the SME. Vidick concluded that a minimum of mixing energy and mixing time is needed to deflocculate the particles in the slurry, but when the slurry is deflocculated, thickening time, fluid loss and plastic viscosity becomes independent of the mixing energy. He also concluded that the mixing time affected the slurry´s yield value. Longer mixing time increased the yield value.

Vidick et al. (1990, 22-24 October) also did a research project where they looked at cementing through coiled tubing and the effect that has on the properties of the slurry. In that research project they discovered that the 1,6 m^3 yard recirculation mixing greatly influenced the cement slurry properties. When they compared the yard mixed cement properties with standard laboratory mixed cement properties, they found that the yield value and the fluid loss increased in the yard mixing compared to the laboratory mixing, while the thickening time decreased. When this discovery was made, Hibbert et al. (1995) joined Vidick to look more closely on the effect of yard mixing on cement slurry properties. They observed dramatic differences in the properties from the slurry prepared in the field scale mixer and the slurry prepared in the laboratory. They, as Orban and his team, concluded that the mixing energy for optimal cement slurry should be similar to the API specification 10. Hibbert estimated the SME for API specification 10 mixing to be 5,9 kJ/kg.

Hibbert and his team also concluded that mixing energy in the cement job has to be considered as important as temperature and pressure, but that “the critical parameter is not the absolute value of mixing energy, but the way in which this is applied with low and high

shear of the slurry during its recirculation through a centrifugal pump."(Hibbert et al., 1995, p. 52)

A formula for calculating SME based on recirculation and agitation mixer was given by Vidick et al. (1990, 22-24 October)

$$\frac{E}{M} = \frac{Pt}{\rho V} \tag{2}$$

In 1996 (Padgett, 1996) investigated the influence of shear rate on slurry properties, and he, as Hibbert and his team, stated that shear rate has a greater influence than total mixing energy on slurry properties. He also pointed out that there is a gap between slurry properties in the field and the slurry properties in the laboratory. Field mixing operate with a much lower shear rate than in laboratory mixing, and this might be the reason for the gap between the slurry properties.

As Teodoriu et al. (2015) stated, it is now well known in the industry that there is a gap between the slurry properties developed in the laboratory, and the slurry properties produced in the field. And there has been limited research on this gap. But several new research projects have been done the last 5-7 years.

In 2019 Saleh et al. (2019) did a study where they investigated the gap between laboratory and field mixing. They compared properties of cement mixed with the same SME but with different mixing device and procedure. Cement slurry was mixed in the laboratory according to API standards, and in a yard mixer according to field procedures. The results showed that the cement properties differed despite that they were mixed with the same SME. Both UCS and rheology measurements were higher for cement mixed in the yard compared to cement mixed in the laboratory. They also concluded that shear rate influences the strength of the cement. Cement mixed with higher shear rate developed a lower strength.

In 2021 Saleh and Teodoriu (2021) did a study on the effect of mixing procedures on the rheological properties and thickening time of cement slurries. In this study rheological

properties and thickening time was compared for slurries with similar SME but different shear rate. The results showed again that slurries mixed with similar SME does not necessarily have the similar properties. Both yield point and plastic viscosity showed increased values with increased shear rate. And this was well correlated with the thickening time measurements where slurries prepared with a lower shear rate showed a longer thickening time.

Up until now the research has been focused on a correlation between cement and cement slurry properties, and SME and shear rate of cement mixing.

This research represents a new view of cement mixing scaling. We look at dimensionless groups and geometric similarity. Earlier research has focused on API mixing versus field mixing, and this does not consider geometrical similarity.

Chapter 2. Theory

2.1 The concept of mixing energy

Mixing energy is defined as the effective energy added per unit mass (kJ/kg), into the slurry as it is mixed and conditioned.
The concept of mixing energy has been used to scale mixing procedures, for instance from laboratory to field.
The mixing energy theory claims that when the mixing energy per unit mass is equal, the cement slurry properties will be similar (Teodoriu et al., 2015).

As mentioned in the previous research section, Orban et al. (1986) provided us with formula (1) for calculating the SME, based on the laboratory mixing procedure and equipment.

$$\frac{E}{M} = \frac{kv^2t}{V} \tag{1}$$

Another formula (2) for calculating mixing energy per unit mass slurry, based on recirculation and agitation, was given by Vidick et al. (1990, 22-24 October):

$$\frac{E}{M} = \frac{Pt}{\rho V} \tag{2}$$

For the NORCE and the 15 liters mixing sessions, the power of the pumps were logged. The energy added to the slurries were calculated and divided on the mass of the slurry.

2.2 Cement mixing procedures laboratory versus field.

Laboratory mixing procedure:

Wet ingrediencies are added to the blender bowl, then the dry ingrediencies are added to the blender bowl while the mixer rotates with a rpm of 4000 for 15 seconds. Then the mixer continues mixing the slurry with a rpm of 12000 for 35 seconds. The slurry is then poured into a conditioning cup and is conditioned with an rpm of 150 for 20 minutes (American Petroleum Institute, 2013).

Field mixing procedure:

There are 3 main types of field mixing systems: (Nelson & Guillot, 2006)

- Conventional jet mixer.
- Recirculation jet mixer.
- Recirculation mixer without conventional jets.

We will look at the mixing procedure of the recirculation mixer without conventional jets, as this is what has been used at the NORCE project.

Water and additives are added to the tank and cement is added through a hopper and is mixed in with an agitator. A centrifugal pump, or a similar device, located at the bottom of the mixing tank, pumps the slurry through a recirculation line to improve the initial mixing (Nelson & Guillot, 2006).

2.3 Important properties of cement slurry and solidified cement

Rheology.

There are several reasons why it is important to optimize the rheological behavior of a cement slurry (Saleh & Teodoriu, 2021):

- to effectively remove drilling mud from the annulus. If the mud is not properly displaced, it may cause leakage pathways and de-bonding.
- to be able to pump the slurry to the desired location in the well. If the cement is to thick and not pumpable, it will not be possible to transport the cement to its planned location.
- to keep the cement particles in suspension so that the cured cement will have an even strength and elasticity. For the cement to be able to withstand stress throughout the life of the well, it needs to be strong and elastic.

Thickening time.

Thickening time is an important property as the cement needs to be transferred to its planned location before it thickens. When the slurry thickens it is harder to transport it, and it can set before it is placed in the planned location. Early set cement is a very costly complication. The set cement may have to be drilled or reamed out, and then a new cement operation is needed. It is also important that it does not take too long to set when it has arrived at its planned location.

Free water.

Free water is defined as water that is not needed by the cement for a reaction. When the slurry stops flowing, free water separates to the top of the cement column.(Saleh & Teodoriu, 2021) High free water can result in leakage pathways as a water channel can be created along the upper side of the annulus, providing a potential gas migration path.

Fluid loss.

When a cement slurry is placed across a permeable formation under pressure, water in the cement will naturally leak out of the cement into the formation. This can cause the cement slurry to dehydrate at the formation wall and this can act as a pathway for gas to migrate out of the formation. The measured fluid loss value can be an indication of the cement slurry´s resistance to gas migration. Another consequence of fluid loss is that the cement slurry becomes denser and may become unpumpable.

Unconfined compressive strength (UCS).

UCS is a measure of the cement´s strength. High UCS is important as the cement need to endure stress from the wellbore and surrounding formation.

Young´s modulus.

Young´s modulus (E) is the property that tells us how elastic the cement is, in other words how easily it can stretch and deform. Elasticity is an important property of the cement. The higher the elasticity and strength of the cement, the more stress the cement can endure before it fails.

2.4 Dimensional analysis

Upscaling and downscaling of a process is based on similarity.

- Geometric similarity, similarity in shape
- Kinetic similarity, similarity in motion
- Dynamic similarity, similarity in forces

Dimensional analysis is a useful tool when working out the criteria for dynamic similarity. It is based on Buckingham´s Π-theorem (Heller, 2011).
Buckingham´s Π-theorem states: "a physical relationship between some dimensional quantity and several dimensional governing parameters can be rewritten as a relationship between a dimensionless parameter and several dimensionless products of the powers of governing parameters; the number of dimensionless products is equal to the total number of governing parameters minus the number of governing parameters with dependent dimensions."(Barenblatt, 2003, pp. 24-25)

In other words, dimensional analysis is a tool for reducing the number and complexity of experimental variables. A physical problem with n independent parameters can be reduced to n-r independent dimensionless parameters, where r is the minimum number of reference dimension.

Dimensional analysis is a common approach for testing small-scales boats or riggs in an indoor pool. We wanted to look at the possibility to use the same scaling method for cement mixing.

We have learned from earlier research that there are three important factors affecting the cement and the cement slurry are:

- Shear rate
- Mixing energy
- Mixing time

When reasoning which Pi groups to use for our experiment, we wanted to use these factors. We ended up with two groups:

The first Π group:

$$\Pi_1 = \frac{PD^5}{\rho L Q^3} = \frac{(\frac{Nm}{s}) * m^5}{(\frac{kg}{m^3}) * (\frac{m^3}{s})^3 * m} = \frac{N * s^2}{kg * m} = \frac{kg * m * s^2}{kg * m * s^2} \quad (3)$$

$$\mathrm{N} = \frac{kg * m}{s^2}$$

P = power of circulation pump (Nm/s)

D = diameter of circulation loop (m)

L = length of circulation loop (m)

Q = flow rate(m^3/s)

ρ = density of slurry (kg/m^3)

This group is related to the shear rate and mixing energy of the mixing session.

The second Π group:

$$\Pi_2 = \frac{Qt}{V} = \frac{\frac{m^3}{s} * s}{m^3} = \frac{m^3}{m^3} \quad (4)$$

Q = Flowrate (m^3/s)

t = mixing time (s)

V = slurry volume (m^3)

This group is related to how many times the fluid is circulated through the mixing unit, hence it is related to shear rate and mixing time.

Chapter 3. Experimental setup

3.1 Mixing Equipment

In this research 3 different mixing equipment has been used.

- a 300-600 liters mixing unit designed and operated by NORCE research center. We were able to get cement slurry samples and had access to all logged data.
- API standard laboratory mixing equipment
- a 15 liters mixing unit designed and operated by us at UiS.

These mixing units will be described in detail in the next subchapters.

3.1.1 NORCE mixing equipment

The main components of the NORCE medium scale mixing unit (fig. 4) are:

- a 1000 liters tank
- an agitator
- a 15kW high pressure centrifugal pump
- a circulation loop.

The agitator was set inside the tank to mix the dry materials into the liquid inside the tank. The tank was connected to the pump through the circulation loop, and the slurry was pumped through the circulation loop to continue the mixing process of the slurry.

Figure 4: The main components of the NORCE mixing unit; A: dry material inlet, B: agitator, C: circulation pump, D: return line of the circulation loop, E: mixing tank

Agitator

To be able to mix in and deflocculate the dry cement into the water in the tank, an agitator was inside the mixing tank as seen in figure 4.

Pump

The pump used in the NORCE mixing unit was a15 kW high pressure pump (fig. 5). The power of the pump was logged throughout the operation.

Figure 5: The centrifugal pump at the NORCE mixing unit

The circulation loop (fig. 6) consists of:

- 2,0 meters 4-inch line
- 2,5 meters 3-inch line
- 0,3 meters 2-inch line

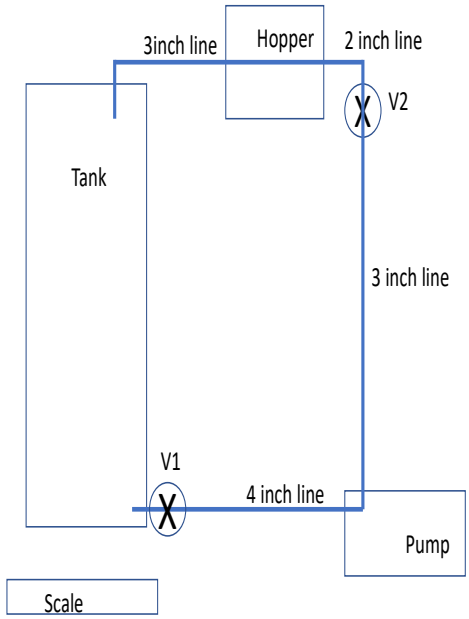


Figure 6: The circulation loop at the NORCE mixing unit.

Throughout the NORCE mixing session these measurements were logged:

- Pump pressure (bar)
- Tank temperature (deg C)
- Slurry density (sg)
- Flowrate (lpm)
- Pump speed (rpm)
- Pump power (%)

3.1.2 API mixing equipment

The API mixing equipment (fig. 7) consists of only two components:

- a Waring blender
- a consistometer

A Waring Blender was used for the mixing of the cement slurry and an Ofite Model 60 atmospheric consistometer was used to condition the cement slurry.

Figure 7: Left; Waring blender used for mixing the cement slurry in the laboratory, Right; Ofite Model 60 atmospheric consistometer used to condition the cement slurry in the laboratory.

3.1.3 UiS 15 liters mixing equipment

For geometric similarity purposes, we wanted to scale down the UiS mixing unit to 1:20 of the NORCE mixing unit. With the slurry volume of the NORCE mixing session of 327 liters, a 15 liters mixing would be in the desired range. But we had to use what was available to us, hence the 15 liters mixing unit at UiS (fig. 8) consisted of:

- a 25 liters tank (1:40 of NORCE 1000 liters tank). A 50 liters tank would give us the 1:20 scale, but the diameter of the tank would be too large and the level of the slurry in the tank would be too low. This could give us a problem of air in the circulation loop. We ended up with using a 25 liters tank.
- a 1000 W drain pump (1:15 of NORCE 15kW pump). A 750 W pump would not be able to pump the slurry due to the high density of 1,92 SG.
- 5 meters of 1 inch circulation loop (1:3 of NORCE 3 inch circulation loop).

- a spatula
- a transformer
- a power measuring unit
- a larger tank with water for cooling

The drain pump was submerged into the mixing tank, and the mixing tank was submerged into a 100-liter tank which was continuously filled with cold water for cooling the slurry. A hole was cut in the 100 liters tank just below the edge of the 25 liters tank so that the cooling water would not enter the slurry. A 0,25-inch hose of 5 meters was connected to the pump and returned to the 30-liter bucket. A transformer was used to reduce the volt/ energy supply to the pump.

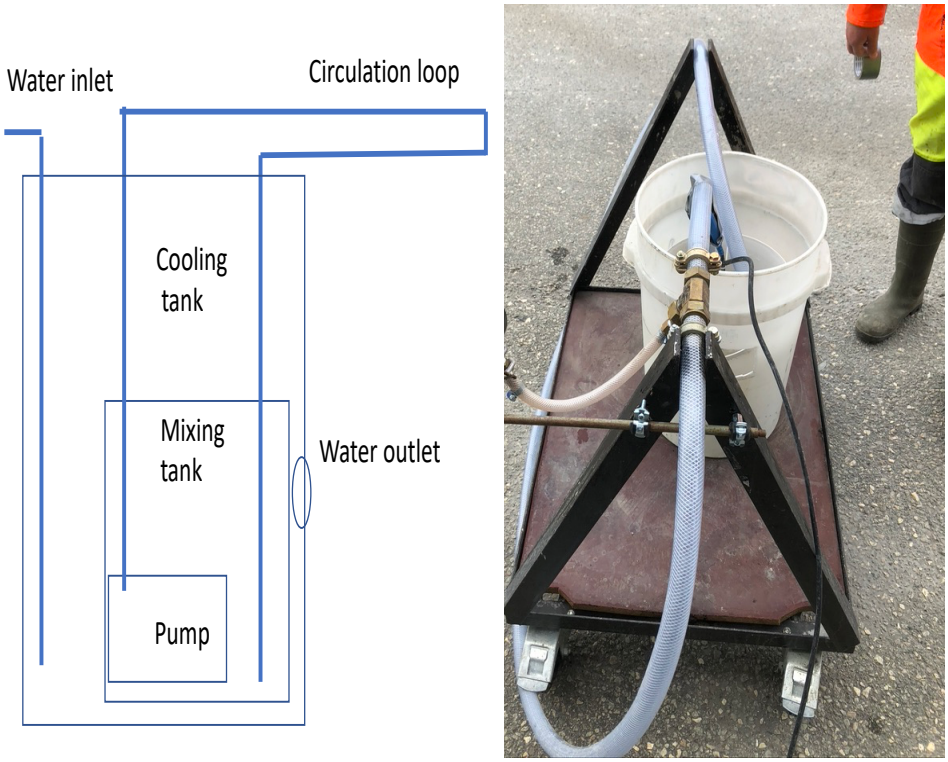


Figure 8: The 15 liters mixing unit.

Figure 9: The 15 liters mixing unit. A: Cooling tank, B: Pump, C: Mixing tank, D: Circulation loop.

Biltema drain pump DP 1001

We wanted to get a pump that we could regulate, to be able to regulate the flow rate. But it was too expensive. Instead, a Biltema 1000 W drain pump with no regulation options, was

used to circulate the slurry. The pump was equipped with a thermic overheating protection at 35 degrees Celsius.

Transformer

A transformer, shown in Figure 9 B, was used to regulate the volt output of the power to the pump, and that way regulate the flowrate.

It was not possible to get a Coriolis meter, or something similar, to measure the flowrate, so to estimate the flowrate, we had to measure it manually. We took the time filling a 2000 ml measuring cylinder shown in figure 10. We then measured the volume of slurry and calculated the flowrate. We measured flow for 160, 180, 200, 220 and 240 volts. The results can be seen in table 1.

Figure 10: 2000ml measuring cylinder.

Table 1: Measurements and calculations for the flowrate of the 15 liters UiS mixing unit.

Volt	Time, sec	Volume ml	Flowrate l/min	Flowrate m^3/sec
160	4,62	800	10,39	0,0001732
180	2,53	900	21,34	0,0003557
200	2,16	1140	31,67	0,0005278
220	1,94	1240	38,35	0,0006392
240	1,63	1600	58,89	0,0009816

Power measuring unit

Throughout the 15 liters mixing session the watt used by the pump was measured with a power measuring unit and logged. The measured watt was used to calculate the energy added to the slurry.

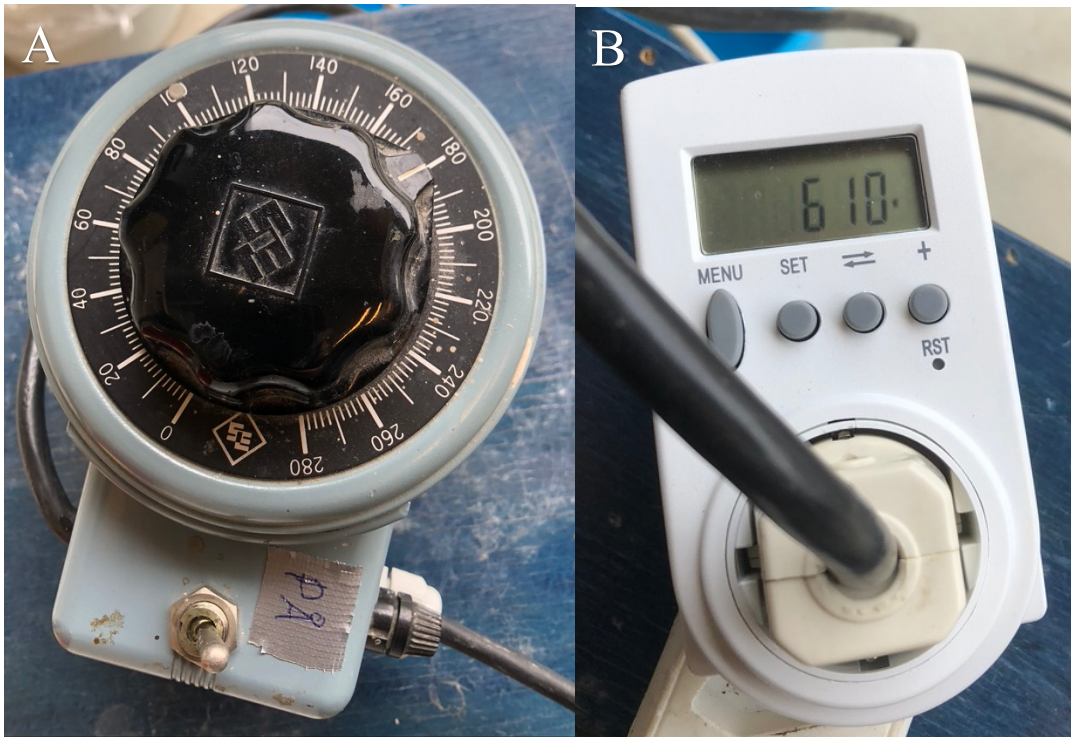


Figure 11: A: The transformer used to regulate the flowrate, B: The power measuring unit to measure the energy used by the pump.

3.2 Mixing procedures

3.2.1 NORCE mixing procedure

We have samples from 3 NORCE mixing secssions where the equipment is the same, but the procedures slightly differ. There is an overview of the NORCE mixing sessions in table 2.

Water and additives were added to the tank and mixed through circulation and agitator, then Dyckerhoff class G + 35% SSA-1 cement were added in from the top and mixed in with an agitator and a circulation pump.

1st NORCE mixing session:

This session was for a volume of 327 liters.

Motor power while the cement was added:

- Increasing steadily from 72% to 100% for 8 minutes
- Continued at 100% for 29 minutes

The cement was added with a circulation of 650 lpm for 37 minutes.

2nd NORCE mixing procedure:

This session was also for a volume of 327 liters.

Motor power while the cement was added:

- Increasing steadily from 67% to 100% for 8 minutes
- Continued at 100% for 15 minutes
- Conditioned the slurry at 12% for 40 minutes

The cement was added with a circulation of 650 lpm for 23 minutes, and then conditioned with a circulation of 270 lpm for 40 minutes.

3rd NORCE mixing procedure:

This session was for a volume of 546 liters.

Motor power while the cement was added:

- Increasing steadily from 65% to 100% for 14 minutes
- Continued at 100% for 20 minutes
- Conditioned the slurry at 15% for 44 minutes

The cement was added with a circulation of 650 lpm for 34 minutes, and then conditioned with a circulation of 270 lpm for 44 minutes.

Table 2: NORCE mixing sessions overview.

	Volume (l)	Time adding dry cement, with flowrate 650 lpm	Time conditioning with flowrate 270 lpm
1st NORCE mixing session	327	37	0
2nd NORCE mixing session	327	23	40
3rd NORCE mixing session	546	34	44

3.2.2 Standard API procedure

Water and liquid additives were added to the Warren blender. The dry ingredients were added for 15 seconds while the mixer ran at 4000rpm. The blender did then continue at 12000 rpm for 35 seconds. The slurry was then be poured into an Ofite Model 60

atmospheric consistometer at room temperature and pressure for 20 minutes with an rpm of 150 (American Petroleum Institute, 2013).

3.2.3 UiS 15 liters mixing procedure

We have samples from 3 UiS 15 liters mixing sessions where the equipment is the same, but the procedures slightly differ. There is an overview of the 15 liters mixing sessions in table 3.

When we built the 15 liters mixing unit at UiS, we wanted to do test rounds with it for both flowrates before we used it for our planned mixing sessions. And in these test rounds, we wanted to check the effect of the additives. We had one test round at 60 lpm flowrate where we left out the suspension agent, this is the 1st UiS 15 liters mixing session. The 2nd UiS 15 liters mixing session was a test round for 20 lpm flowrate where we left out the retarder agent.

Both test rounds were successful, and we decided to use the data from these mixing sessions. Unfortunately, we did not have time to do a mixing session with the 60 lpm flowrate with the original recipe.

Water and additives were added to the tank and mixed through circulation and agitator, then Dyckerhoff class G + 35% SSA-1 cement were added in from the top and mixed in with a pump.

<u>1st UiS 15 liters mixing session procedure:</u>

This mixing session was a test run and was done without the suspension agent to measure the effect of the agent. The transformer was set to 240 volts, which corresponds to a flowrate of approximately 60 lpm.

The cement was added in for 25 minutes, then first test was taken. We then continued mixing at 100% power for 15 minutes, and the second samples were taken. We continued mixing still at 100% power for 15 more minutes, then third and last samples were taken.

2nd UiS 15 liters mixing procedure:

This mixing session was a test run and was done without the retarder agent to measure the effect of the agent. The transformer was set to 180 volts, which corresponds to a flowrate of approximately 20 lpm.

The cement was added in for 23 minutes. The pump stopped 5 times while we were adding in the cement, and the time the pump was stopped was subtracted from the total time. After 23 minutes the pump shut down and did not start up again. Our first and only sample was taken then. The slurry was very thick/ viscous, and this is probably caused by the absent of retarder in the slurry.

3rd UiS 15 liters mixing procedure:

This mixing session was done with the original cement recipe. The transformer was set to 180 volts, this corresponds to a flowrate of approximately 20 lpm.

The cement was added in for 20 minutes, then the first sample was taken. The pump stopped twice while we were adding the cement, and the time the pump was stopped was subtracted from the total time. We then continued mixing at 100% power for 7 minutes, but the pump kept shutting off. After 7 minutes of pumping the pump stopped for a last time and did not restart, and the second and last sample was taken.

Table 3: 15 liters mixing session overview

	Flowrate (lpm)	Time first sample (min)	Time second sample (min)	Time third sample (min)
1st 15 liters mixing session	60	25	40	55
2nd 15 liters mixing session	20	23	-	-
3rd 15 liters mixing session	20	20	27	-

3.3 Measurement equipment and procedures

3.3.1 Rheology measurements

For the rheology measurements of the NORCE mixing sessions, the measurements were done by the laboratory technicians at the NORCE research center.

For the rheology measurements of the API and 15 liters mixing sessions, an Ofite Model 900 Viscometer was used. The slurry was poured into the viscometer cup within 1 minute after the mixing procedure was done. An automatic cement rheology analyzer feature on the Viscometer, which follows the procedure recommended by API 10 B-2, was used. With the motor running at 3 rpm the measurement was read off after 10 seconds, then the rotor was increased to 6 rpm for next reading. This was continued through 3, 6, 30, 60, 100, 200 and 300. First ascending, the descending. The average of the ascending and descending reading is used for further analysis.

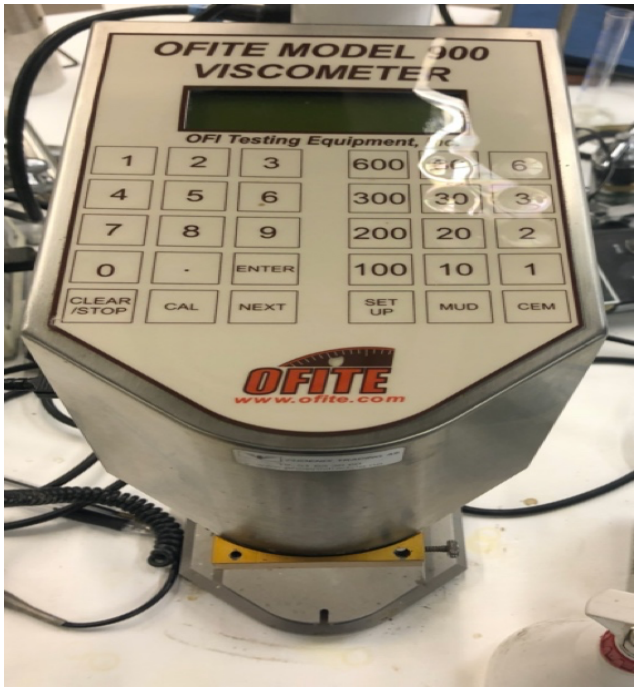


Figure 12: Ofite Model 900 viscometer.

3.3.2 Transit time measurements

The transit time of the cement was measured with a Halliburton Services Ultrasonic Cement Analyzer (UCA). The UCA measures the transit time of an electric impulse through the slurry. The transit time is an indirect measurement of the setting time. The shorter the transit time, the harder the sample is. The transit time is measured from the slurry is mixed and until it has solidified.

The UCA molds were filled and covered within 5 min after mixing. The molds were placed in the UCA machine at different times due to logistic issues. The samples from the NORCE mixing sessions were filled at the NORCE research center, but as the UCA machines were at UiS, it took between 1-2 hours before the molds were placed into the UCA machines. For the 15 liters mixing session, the samples were placed directly in the UCA machine.

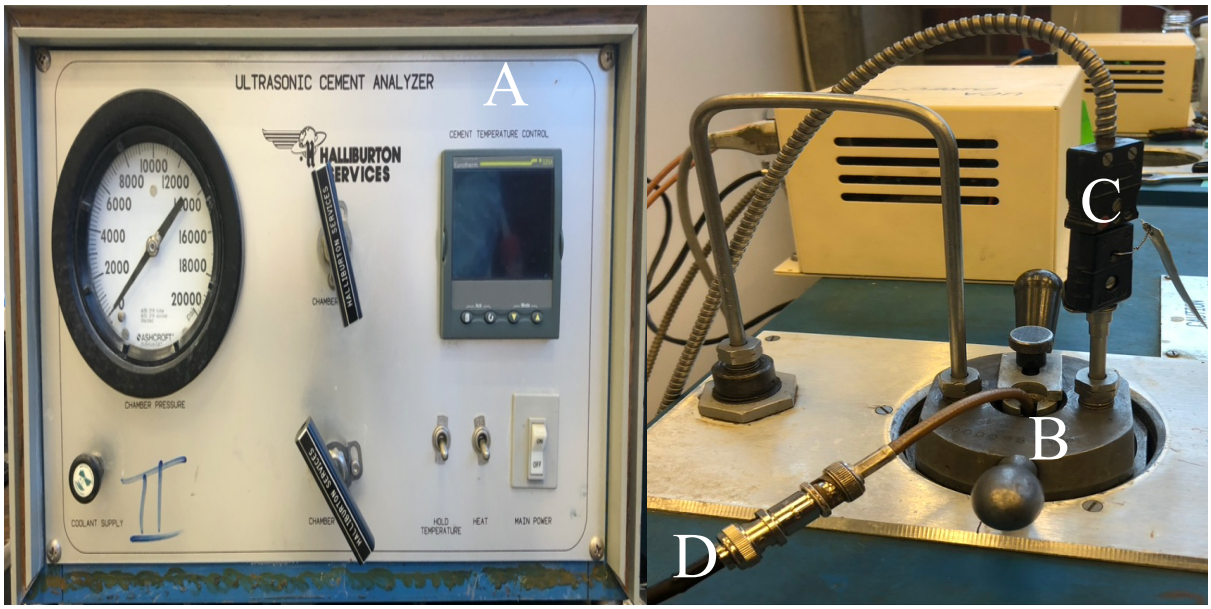
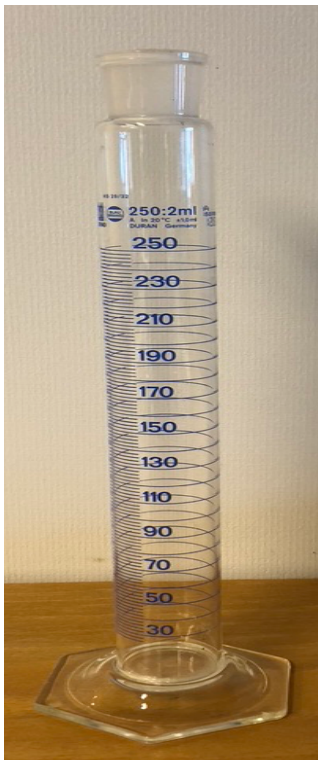


Figure 13: Halliburton Services Ultrasonic Cement Analyzer was used to measure the transit time through the slurry. A; The manifold of the UCA machine, B; UCA mold,C; temperature sensor, D; electric signal sensor.

This experiment was a cooperation with NORCE research center, and NORCE requested UCA measurements under 70 degrees Celsius and 50 bar/ 725 psi. Hence the measurements were done under these circumstances.

3.3.3 Free water measurements



To measure free water, a 250 ml graduated cylinder with 250 mm in length and 40 mm in diameter, were used.

Slurry was poured into the cylinder and then the cylinder was sealed with plastic film. After 2 hours in ambient temperature, the volume of free water (clear or colored) was measured. The volume fraction of free fluid was calculated.

Figure 14: 250 ml measuring cylinder used to measure free water.

3.3.4 Compressive strength and Young´s modulus measurements

For the NORCE and API mixing sessions, four 2x5 inch reusable metal molds were filled within 5 minutes after mixing. Water was added on top of the cement slurry to prevent dehydration and microcracks, and then the containers were covered with plastic film. For the 15 liters mixing session we needed 9 samples curing at the same time, and the workshop were able to 3D print 5 2x6 reusable plastic molds for us.

We did have some issues with the 3D printed molds as some of them developed cracks and small deformations as the cement cured. This was probably due to the expansion of the cement as it cured. We were not able to reuse some of these, hence the number of samples reduces for each 15 liters mixing session.

Total amount of cured samples can be seen in table 4.

Table 4: Overview of cured samples.

Mix	Number of samples
1st NORCE mix	4
2nd NORCE mix	4
3rd NORCE mix	4
API mix	4
1st 15 liters mix, 60 lpm	9
2nd 15 liters mix, 20 lpm	8
3rd 15 liters mix, 20 lpm	4
Total	37

Figure 15: Metal molds (in the front) and 3D printet plastic molds (in the back) used to cure samples for UCS and Young´s modulus measurements.

After curing for 7 days in room temperature and atmospheric pressure, the samples were trimmed with a Baldor Grinder machine to make sure the surfaces were smooth and the radius to length ratio were 2-2,5 times the diameter.

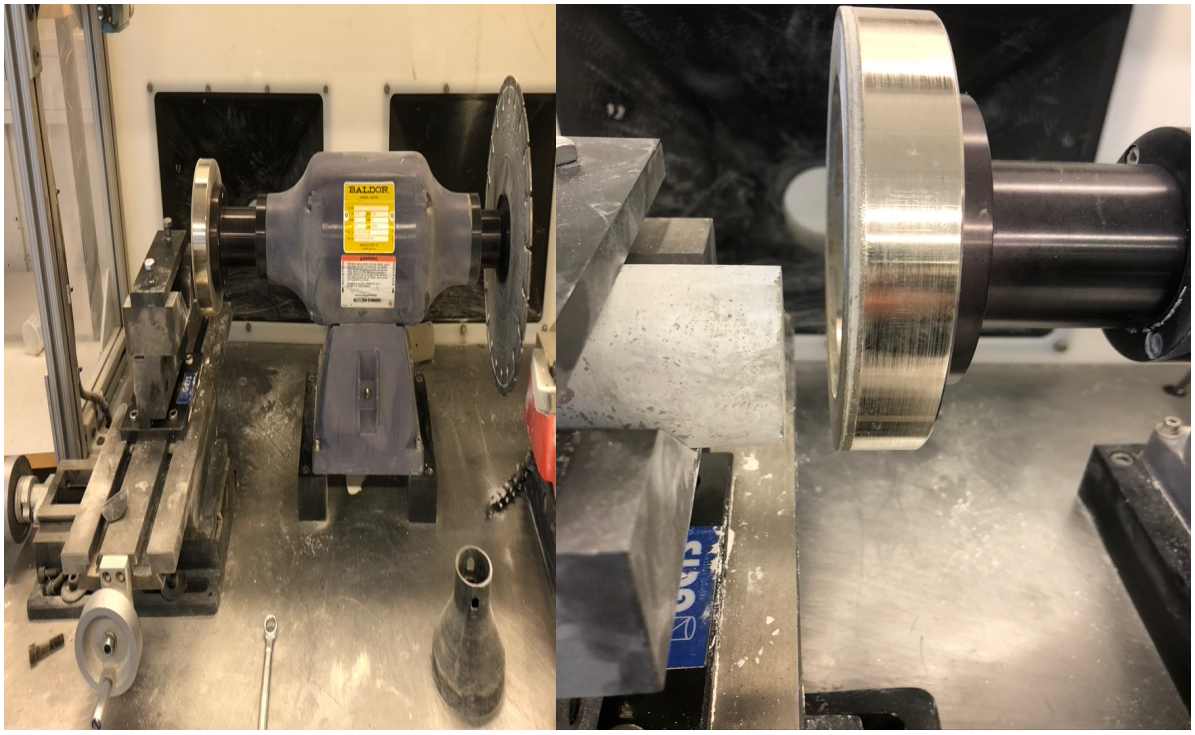


Figure 16: Baldor Grinder machine used to trim the cured and solidified samples.

To measure the unconfined compressive strength (UCS) of the cured cement sample, a MTS Criterion M45 machine was used together with TW Elite Software.

According to American Petroleum Institute, "For load control, the force may be applied in a rate such that a constant stress rate in the range of 3.5 MPa/min to 14 MPa/min is produced in the specimen." (American Petroleum Institute, 2017, p. 22). Stress is load divided by area. Our samples had a diameter of approximately 50,9mm, and that gave us an area of 0,00203378 m^2 (eq. 5).

$$\text{Area} = \frac{\pi * d\text{^}2}{4} = \frac{3{,}14}{4} * 50{,}9*50{,}9 = 2033{,}78\ \text{mm}^2 = 0{,}00203378\ \text{m}^2 \qquad (5)$$

As we see from the calculations below, both alternatives are outside the API recommendation. We chose to compress our samples with the 30kN/min force rate.

As a student user of the machine, the only available compression program was monotonically compression with a force rate of 7kN/min or 30kN/min.

1 Pascal = a pressure of 1 newton per square meter = $\frac{Force}{Area}$ (6)

$$\frac{30000\ N}{0{,}00203378\ m^2} = 14{,}770\ \text{MPa}$$

$$\frac{7000\ N}{0{,}00203378 m^2} = 3{,}435\ \text{MPa}$$

After the 2nd NORCE mixing session we got access to an axial extensometer, and we used this to measure the young´s modulus. According to API TR 10TR7 "Specimens are typically loaded cyclically 2 to 4 times within its elastic limit to determine their elastic constants, such as Young´s modulus (E) and Poisson´s ratio (ν). The first cycle often closes micro defects and produces different results that subsequent cycles. It is not recommended to use data for the first cycle for any calculations." (American Petroleum Institute, 2017, p. 23)

A cyclic loading program with 2 cycles were made available to me, where the second cycle were used to calculate young´s modulus. The cyclic loading program loaded the sample up to a force of 25 kN as this was 50 % of the expected UCS, and then unloaded the sample back to 1 kN. This was done 2 times with a force rate of 30 kN/min. After the cyclic loading program, we removed the extensometers and measured the UCS with the same monotonically compression program as before.

Some of the samples had a lower UCS and broke during the cyclic loading program. We then reduced the max load of the program to 15 kN.

We did have some problems with oscillations when using force rate for the cyclic loading program, so it was decided to use displacement rate. A new a cyclic loading program with a 0,25mm/ min displacement rate was made available for me. The monotonically compression program were kept the same for the UCS measurements to be consistent.

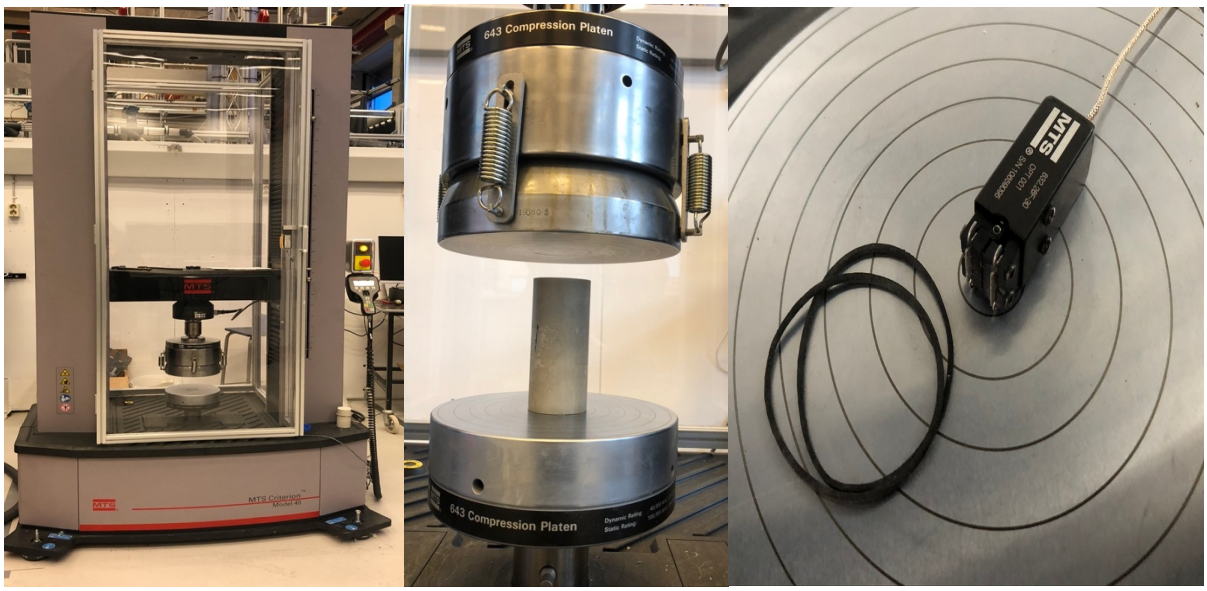


Figure 17: A; MTS Criterion M45 was used for measuring UCS, B; the crosshead providing the force of the UCS machine, C; the axial extensometer measuring the deformation of the samples.

An axial extensometer was used to measure the deformation of the cured cement samples. The extensometer has two knife blades that are attached to the sample by two rubber bands. The movement of the two blades is then measured.

Young´s modulus, E, or the modulus of elasticity, is a mechanical property that measures the tensile or compressive stiffness of a solid material when a force is applied lengthwise. Young´s modulus is the linear relationship between stress (MPa) and strain (%) (Nelson & Guillot, 2006).
Stress is the normal force on the surface of the sample per unit area of the surface of the sample. Strain is the resulting deformation as the force is applied to the surface of the sample.

$$\mathrm{E} = \frac{\sigma}{\varepsilon} \tag{7}$$

$$\sigma = \text{stress} = \frac{force}{area} \tag{8}$$

$$\varepsilon = \text{strain} = \frac{\Delta L}{\mathrm{L_0}} \tag{9}$$

ΔL = deformation

L_0 = original length of sample

Strain can be measured by the crosshead of the UCS machine. As the specimen deform, the crosshead moves. But there are some uncertainties with regards to the deformation of the crosshead itself.

Strain can also be measured by using an axial extensometer. The extensometer has two knife blades that are attached to the specimen by two rubber bands. The movement of the two blades is then measured, hence only the deformation of the specimen is measured.

To use an extensometer, we needed to use a cyclic loading program to be able to remove the extensometers before the specimen were crushed. This was to make sure the extensometer was not broken through the process. According to API technical report 10TR7 (American Petroleum Institute, 2017), it is not recommended to use data from the first loading for any calculations when using a cyclic loading. The first cycle closes micro cracks and defects and produces different results that the following cycles. Only the second loading were used for Young´s modulus calculations.

We do not have young´s modulus measurements for 1st and 2nd NORCE mixing sessions because we did not have the axial extensometer available to us.

When we compared Young´s modulus calculated with the crosshead displacement and Young´s modulus calculated with the extensometer displacement, we saw a great difference. The results are presented in table 5 and table 6.

Table 5: UCS, Young´s modulus(E) and UCS/E for the third NORCE mixing session.

3rd Norce mixing session	UCS, MPa	E, GPa, with extensometer	UCS/E	E, GPa, with crosshead
CP2	13,93	9,91	1,41	2,93
CP3	20,18	13,32	1,52	2,72
CP4	21,63	11,49	1,88	2,88
Mean	18,58	11,57	1,60	2,84
SD	4,09	1,71	0,25	0,11

Table 6: UCS,E and UCS/E for the 15 liters 60 lpm mixing session.

UiS 15 liters mixing session	UCS, MPa	E, GPa, with extensometer	UCS/E	E, GPa, with crosshead
CP1	23,04	8,33	2,77	2,44
CP2		8,09		2,41
CP3	22,47	8,86	2,54	2,47
Mean	22,76	8,42	2,65	2,44
SD	0,40	0,40	0,16	0,03

We cannot exclude the possibility of the specimen to be affected by the cyclic loading, hence alter the UCS of the specimen, but as discussed earlier, it has been crucial to get good measurements for Young´s modulus calculations.

The mechanical quality of the cement includes both strength and flexibility. A cement with high strength and high flexibility can handle more external stress. But often as the strength of the cement increases, the flexibility of the cement decreases. Therefore, the ratio of UCS to Young´s modulus (UCS/E) has also been calculated.

3.5 Cement recipe

We have used the same recipe throughout all experiments, except for the test rounds of the 15 liters mixing unit. The cement recipe is shown in table 7.

As we mentioned in subchapter 3.2.3, our two first UiS mixing sessions were test rounds. We did one test round at 60 lpm flowrate where we left out the suspension agent, and one test round for 20 lpm flowrate where we left out the retarder agent. We decided to use the data from these test rounds. Unfortunately, we did not have time to do a mixing session with the 60 lpm flowrate with the original recipe.

Table 7: Cement recipe from NORCE research center.

Order	Material	Amount for 9 sakcs of cement	Unit
1	Fresh water	176,2	Liter
2	SA-1015	0,067	Kg
3	NF-6	0,34	Liter
4	HR-5L	2,34	Liter
5	Dyckerhoff gHT Blend	450	Kg
	Slurry volume	327	Liter

Dyckerhoff is a class G cement from Halliburton. In this cement there has been added 35 % Silica flour, SSA-1. A laboratory report of the cement and a safety data sheet, SDS, of the SSA-1, can be found in attachment D.

NF-6 is a defoaming agent consisting of mostly vegetable oil, and a small percentage of aluminum stearate. HR-5L is a retarder agent. SA-1015 is suspension agent. The SDS´s for all the additives can also be found in attachment D.

Chapter 4: Results

4.1 Specific mixing energy calculations

A summary of the SME calculations for all the mixing sessions can be seen in figure 18. We will go through the SME calculations in detail in the next subchapters.

The 2nd and 3rd NORCE mixing sessions and the UiS 15 liters, 20 lpm mixing sessions has similar SME of 28-40 kJ/kg. According to the mixing energy theorem, we should expect to see similar properties from these mixing sessions. We will discuss this in the subchapter where we present the cement properties.

Both the NORCE mixing sessions and the UiS 15 liters sessions have much higher SME than the standard API SME of 5,9 kJ/kg, the highest being 116 kJ/kg. We have not seen this high SME in earlier research. In the research of Saleh et al. (2019), they operate with an SME of 15,9 kJ/kg and 11,8 kJ/ kg for the large scale mixer.

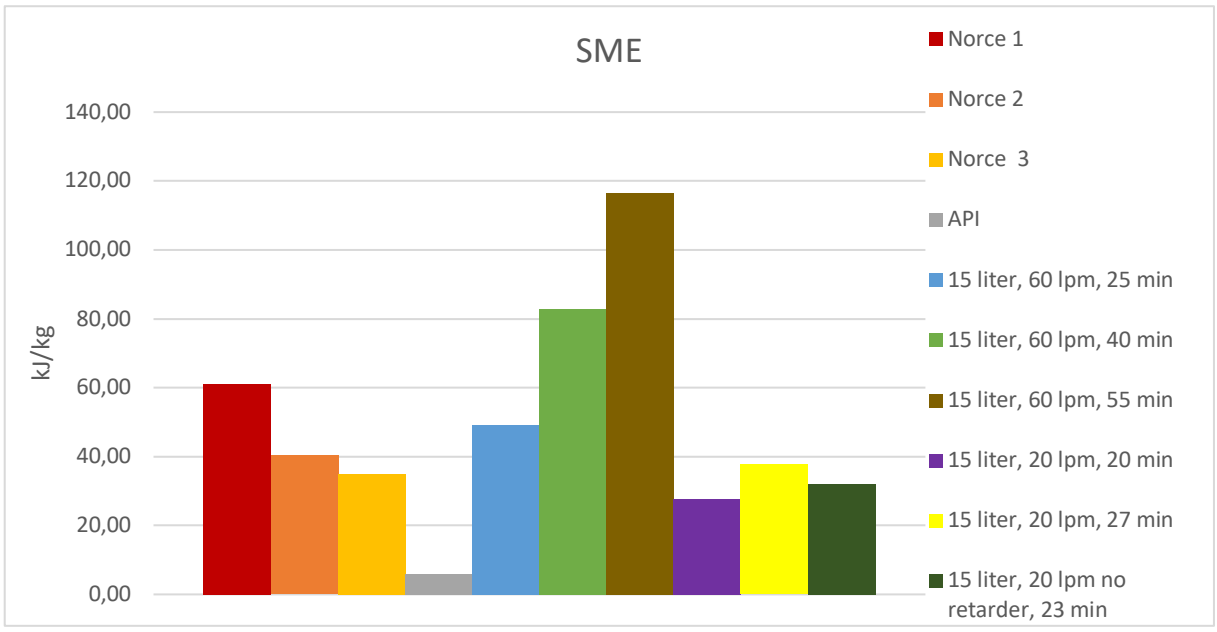


Figure 18: SME results for all the mixing sessions.

4.1.1 Specific mixing energy calculations for the NORCE mixing sessions

To be able to calculate the SME we needed to calculate the energy (E) added to the slurry and divide it on the mass (M). For the NORCE mixing sessions, the power of the pump was logged every second. The energy added to the slurry was calculated using the pump power measurements and the trapezoidal rule (eq. 10). The trapezoidal rule is a technique for approximating the definite integral.

$$A_{t1\text{-}t2} = (t_2 - t_1) * 0{,}5(P_1 + P_2) \quad (10)$$

P = pump power
t = time

We then summarized A for the entire time interval of the mixing, and multiplied by power of the pump, which in this case was 15 kW. We then got the total energy added to the mixing session. The total energy added, and the resulting SME is shown in table 8.

The mass of the 1st and 2nd NORCE mixing sessions was:

$$\rho = 1{,}92\ sg = 1920\ \text{kg/m}^3$$
$$V = 327\ liter\ \text{V} = 0{,}327\ \text{m}^3$$
$$M = \rho V = 1920\ \text{x}\ 0{,}327 = 627{,}84\ \text{kg}$$

The mass of the 3rd NORCE mixing session was:

$$\rho = 1{,}92\ sg = 1920\ \text{kg/m}^3$$
$$V = 546\ liter\ \text{V} = 0{,}547\ \text{m}^3$$
$$M = \rho V = 1920\ \text{x}\ 0{,}547 = 1048{,}32\ \text{kg}$$

Table 8: An overview of pump power, time, volume, mass, added energy and resulting SME for the NORCE mixing sessions.

	Norce 1	Norce 2	Norce 3
P (Nm/s)	18277	6601	7727
t (s)	2100	3840	4740
V (m3)	0,33	0,33	0,55
M (kg)	627,84	627,84	1048,32
E (kJ)	38381	25346	36625
E/M (kJ/kg)	61,13	40,37	34,94

In the 1st NORCE mixing session, the samples were taken as soon as the high energy mixing was done. The 2nd and 3rd NORCE mixing sessions were divided up in two phases, a high energy mixing phase with high pump power and flowrate, and a low energy conditioning phase with low pump power and flowrate. This can be seen in the change of slope for both 2nd and 3rd NORCE data. Due to the larger volume of slurry for the 3rd NORCE mixing session, the slope is lower for the mixing phase.

A comparison of the SME over time for NORCE mixing sessions can be seen in figure 19.

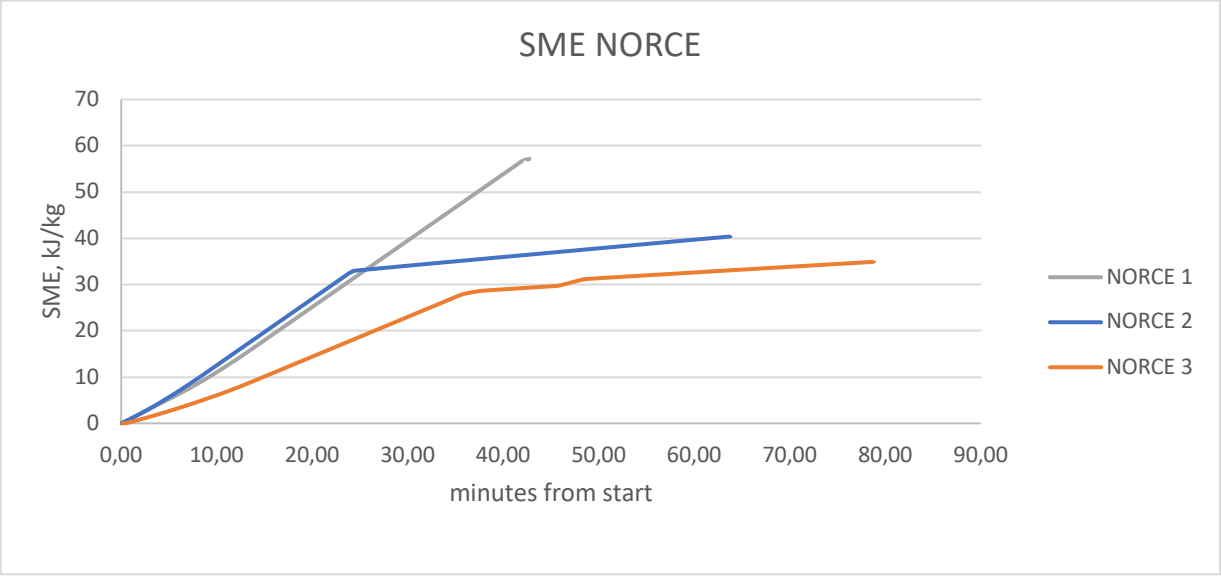


Figure 19: SME over time results for the NORCE mixing sessions.

As we can see in figure 20, the conditioning phase is a low energy phase, and it does not contribute much to the SME pattern that we see. We will use the combined SME for the 2nd and 3rd NORCE mixing sessions for further analyses.

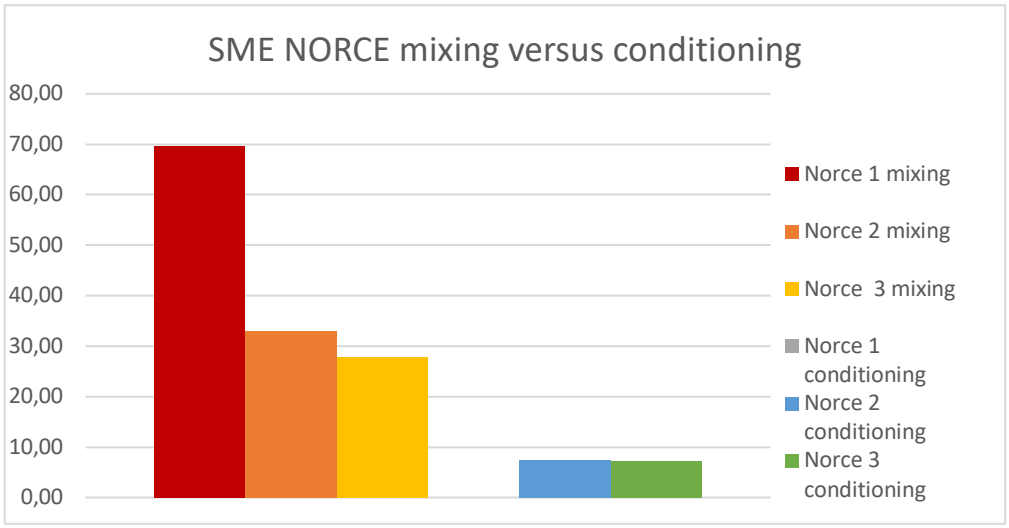


Figure 20: Comparing SME calculations from the mixing phase and the conditioning phase.

4.1.2 Specific mixing energy for the API mixing sessions

To calculate the specific mixing energy, SME, equation 1 (introduced in chapter 1.2) has been used. SME for the API mixing procedure has been calculated to be 5,9 kJ/kg in earlier research, and k was experimentally found to be equal to 6,1 * 10^{-11} m^2/s^3 (Vidick, 1990). This SME is from the mixing in the warren blender only. Due to the low rpm of the conditioning, the energy from the conditioning is so small that it is neglectable.

$$\frac{E}{M} = \frac{kt\rho v^2}{V\rho} = \frac{ktv^2}{V} \quad (1)$$

The calculation of the SME for the API mixing session can be divided into 3 parts, and is shown in detail below:

- 1st part, cement is mixed in with 4000 rpm for 15 seconds

- 2nd part, cement is deflocculated with 12000 rpm for 35 seconds
- 3rd part, cement is conditioned with 150 rpm for 20 min.

V = 600 ml = 0,0006 m^3

v_1 = 4000 rpm = 418,88 rad/sec

t_1 = 15 seconds

E/M_1 = 0,268 kJ/kg

v_2 = 12000 rpm = 1256,64 rad/sec

t_2 = 35 seconds

E/M_2 = 5,62 kJ/kg

v_3 = 150 rpm = 15,71 rad/sec

t_3 = 20 minutes = 1200 seconds

E/M_3 = 0,0301 kJ/kg

4.1.3 Specific mixing energy calculations for the 15 liters mixing sessions

The same method as for NORCE has been used for the 15 liters mixing sessions. The only difference is that for the 15 liters mixing session the power of the pump was logged every 1-2 minutes. This gives us a much less accurate measurement of the energy added to the slurry. The measurements from the 15 liters mixing sessions can be found in attachment C. The total energy added, and the resulting SME is shown in table 9.

The mass of the 15 liters mixing sessions was:

$\rho = 1{,}92\ sg = 1920\ \mathrm{kg/m^3}$

$V = 15\ liter\ \mathrm{V} = 0{,}015\ \mathrm{m^3}$

$M = \rho V = 1920\ \mathrm{x}\ 0{,}015 = 28{,}80\ \mathrm{kg}$

Table 9: An overview of pump power, time, volume, mass, added energy and resulting SME for the UiS 15 liters mixing sessions.

	15 l, 60 lpm, 25 min	15 l, 60 lpm, 40 min	15 l, 60 lpm, 55 min	15 l, 20 lpm, 23 min	15 l, 20 lpm, 20 min	15 l,20 lpm, 27 min
P (Nm/s)	942	994	1017	671	664	660
t (s)	1500	2400	3300	1380	1200	1650
V (m3)	0,02	0,02	0,02	0,02	0,015	0,015
M (kg)	28,8	28,80	28,8	28,8	28,8	28,8
E (kJ)	1412,750	2384,750	3356,750	925,810	797,175	1089,075
E/M (kJ/kg)	49,05	82,80	116,55	32,15	27,68	37,82

An overview of the SME over time for 15 liters mixing sessions can be seen in figure 21.

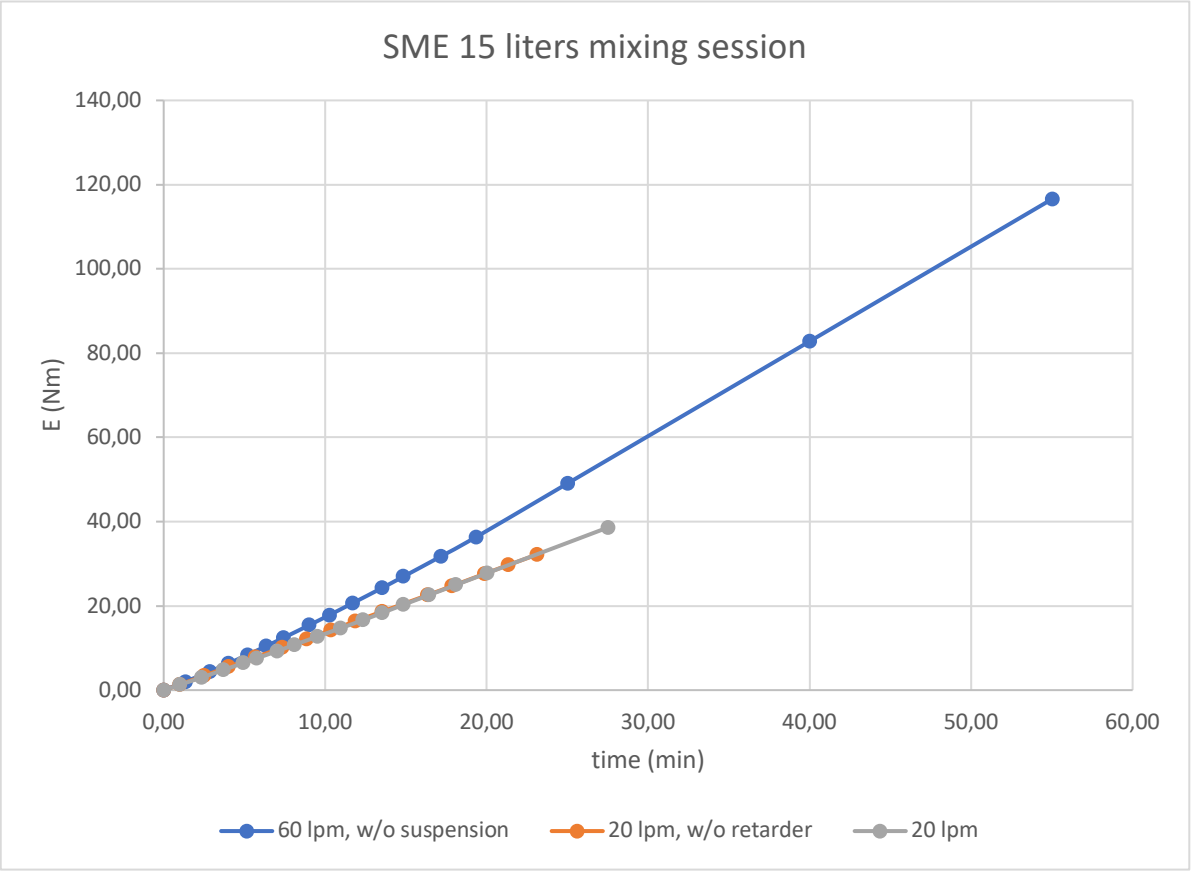


Figure 21: SME results for the 15 liters sessions.

4.2 Dimensionless group Π_1 calculations

As mentioned earlier in subchapter 2.4, the first Π group that we used was:

$$\Pi_1 = \frac{P*D\text{^}5}{\rho*Q^3*L} \quad (3)$$

$P = \text{power of circulation pump (Nm/s)}$

$D = \text{diameter of circulation loop (m)}$

$L = \text{length of circulation loop (m)}$

$Q = \text{flow rate}(m^3/s)$

$\rho = \text{density of slurry (kg/m\^3)}$

The dimensions and resulting Π_1 value can be found in table 10 and figure 22.

Table 10: An overview of dimensions and resulting Π_1 value for all the mixing sessions.

	Norce 1	Norce 2	Norce 3	15 l, 60 lpm, 25 min	15 l, 60 lpm, 40 min	15 l, 60 lpm, 55 min	15 l, 20 lpm, 23 min	15 l, 20 lpm, 20 min	15 l,20 lpm, 27 min
D (m)	0,076	0,076	0,076	0,025	0,025	0,025	0,025	0,025	0,025
Q (m3/s)	0,01083	0,01083	0,01083	0,00098	0,00098	0,00098	0,00033	0,00033	0,00033
P (Nm/s)	18277	6601	7727	942	994	1017	671	664	660
rho (kg/m3)	1920	1920	1920	1920	1920	1920	1920	1920	1920
L (m)	4,80	4,80	4,80	5,00	5,00	5,00	5,00	5,00	5,00
Π_1	4,01	1,45	1,69	1,01	1,07	1,09	18,43	18,25	18,13

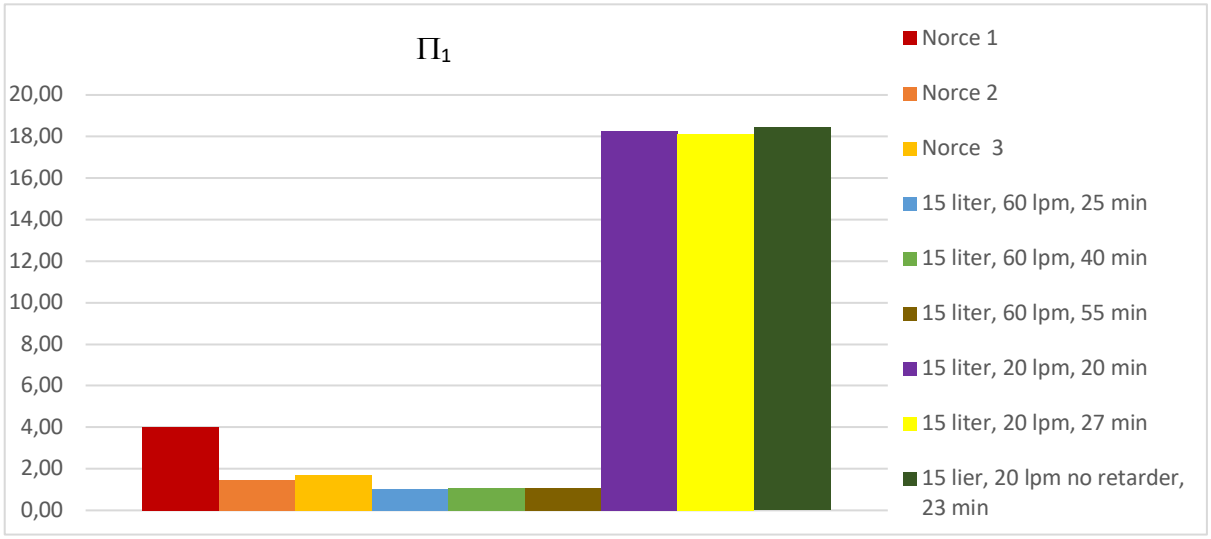


Figure 22: Π_1 results for all the mixing sessions.

There are no Π calculations for the API mixing session because we do not have the data to calculate it.

As we see in figure 22, the Π_1 is much higher for 20 lpm mixing sessions. This is due to the low flowrate and the low pump power. As the flowrate is raised to the power of 3, it does influence the Π_1 value greatly. The higher the flowrate, the lower Π_1 value. The low pump power will also affect the Π_1 value, the lower the pump power, the lower the Π_1 value.

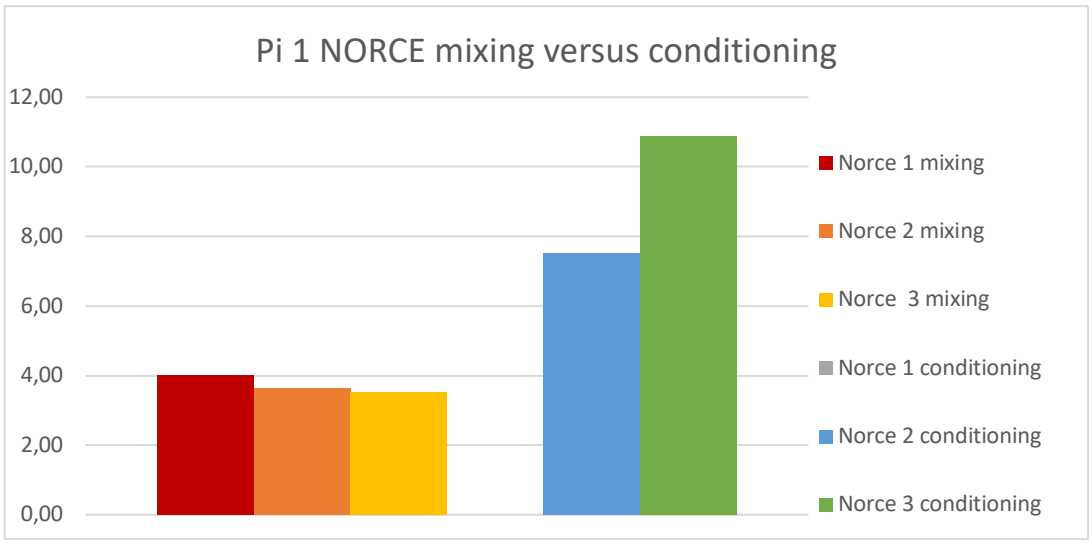


Figure 23: Comparing Π_1 results from the mixing phase and the conditioning phase.

An interesting observation we can see in figure 23, where we compare the Π_1 value for the mixing phase and the conditioning phase, is that for the Π_1 value of the NORCE mixing sessions it is the conditioning phase that influences the Π_1 value the most. This is due to a lower flowrate in the condition phase.

4.3 Dimensionless group Π_2 calculations

The second Π group that we used is:

$$\Pi_2 = \frac{Q * t}{V} \tag{4}$$

$\mathrm{Q} = \mathrm{Flowrate}\ (\mathrm{m}^3/\mathrm{s})$
$\mathrm{T} = \mathrm{mixing\ time}\ (\mathrm{s})$
$\mathrm{V} = \mathrm{slurry\ volume}\ (\mathrm{m}^3)$

Dimensionless:

$$\Pi_2 = \frac{\frac{m^3}{s} * s}{m^3}$$

The dimensions and resulting Π_2 value can be found in table 11

Table 11: An overview of dimensions and resulting Π_2 value for all the mixing sessions

	Norce 1	Norce 2	Norce 3	15 l, 60 lpm, 25 min	15 l, 60 lpm, 40 min	15 l, 60 lpm, 55 min	15 l, 20 lpm, 23 min	15 l, 20 lpm, 20 min	15 l,20 lpm, 27 min
Q (m3/s)	0,01083	0,01083	0,01083	0,00098	0,00098	0,00098	0,00033	0,00033	0,00033
t (s)	2100	3840	4740	1500	2400	3300	1380	1200	1650
V (m3)	0,33	0,33	0,55	0,015	0,015	0,015	0,015	0,015	0,015
Π_2	69,57	127,22	94,05	98,16	157,06	215,95	30,67	26,67	36,67

As we see in figure 24, the Π_2 is lower for 20 lpm mixing sessions. This is due to the low flowrate and the short mixing time.

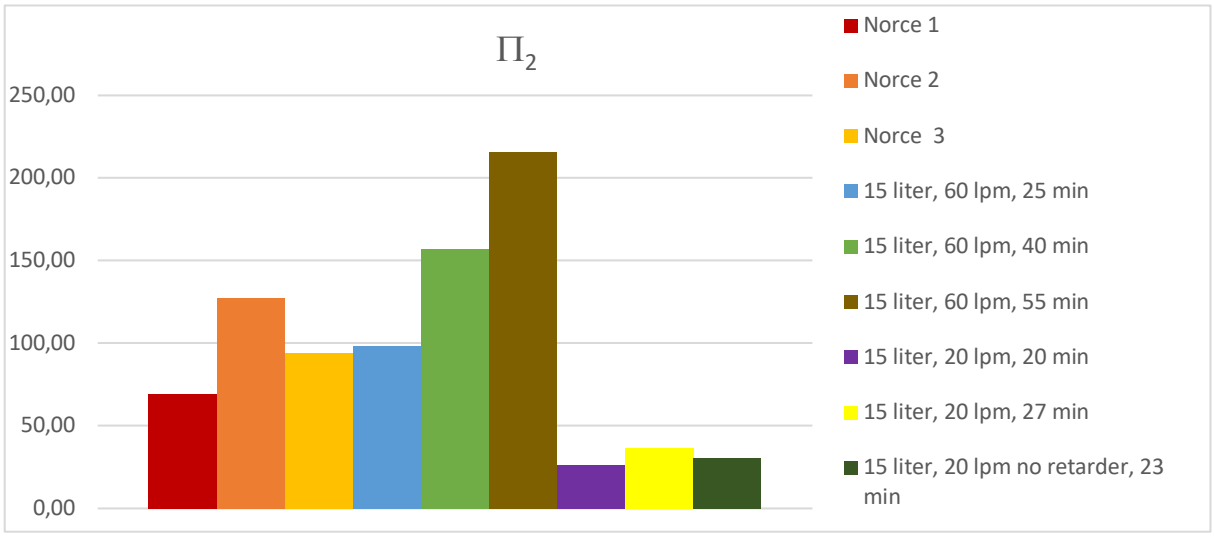


Figure 24: Π_2 results for all the mixing sessions.

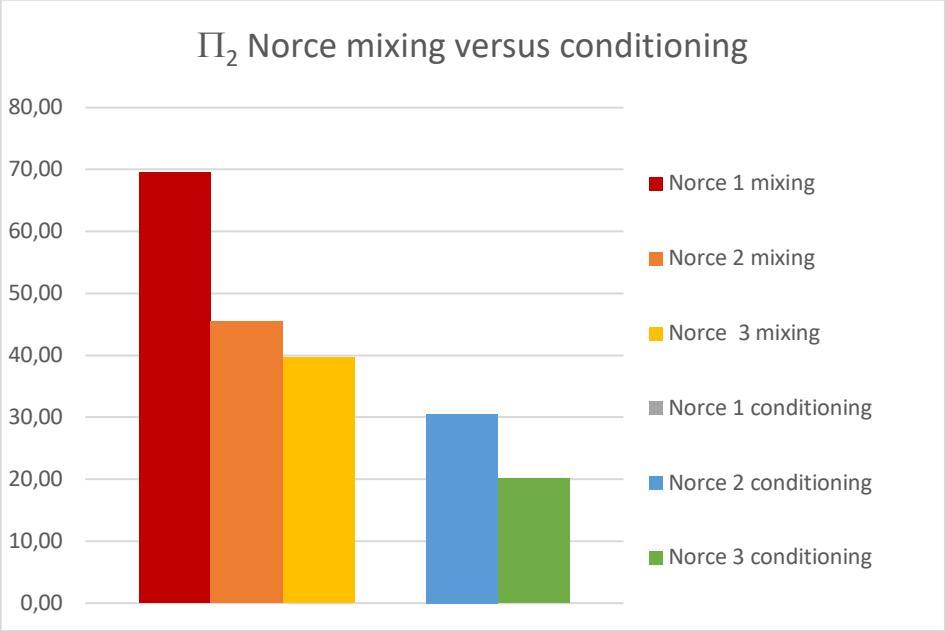


Figure 25: Comparing Π_2 results from the mixing phase and the conditioning phase of the NORCE mixing sessions.

When we compare the Π_2 value for the mixing phase and the conditioning phase of the NORCE mixing sessions in figure 25, we observe that the conditioning phase that influences the Π_1 value quite a bit.

4.4 Measurements

4.4.1 Transit time measurements

As stated earlier, the UCA transit time is an indirect measurement of the setting time of the cement slurry, and the time the sample takes for the transit time to reach approximately 9-9,5 is the setting time.

2nd and 3rd NORCE mixing sessions, and UiS 15 liters 20 lpm mixing sessions due have similar SME but does not have similar setting time measurements (fig. 26). This suggests that, like Saleh and Teodoriu concluded, the mixing energy theory is not consistent, and slurries mixed with similar SME does not necessarily have the similar properties.

Also, the samples from the NORCE mixing sessions show very similar setting time, despite that they do not have similar SME. But they do have similar shear rates. This can indicate that the setting time is influenced by shear rate, and not SME This is consistent with Hibbert and his team´s statements that "the critical parameter is not the absolute value of mixing energy, but the way in which this is applied with low and high shear of the slurry during its recirculation through a centrifugal pump."(Hibbert et al., 1995, p. 52)

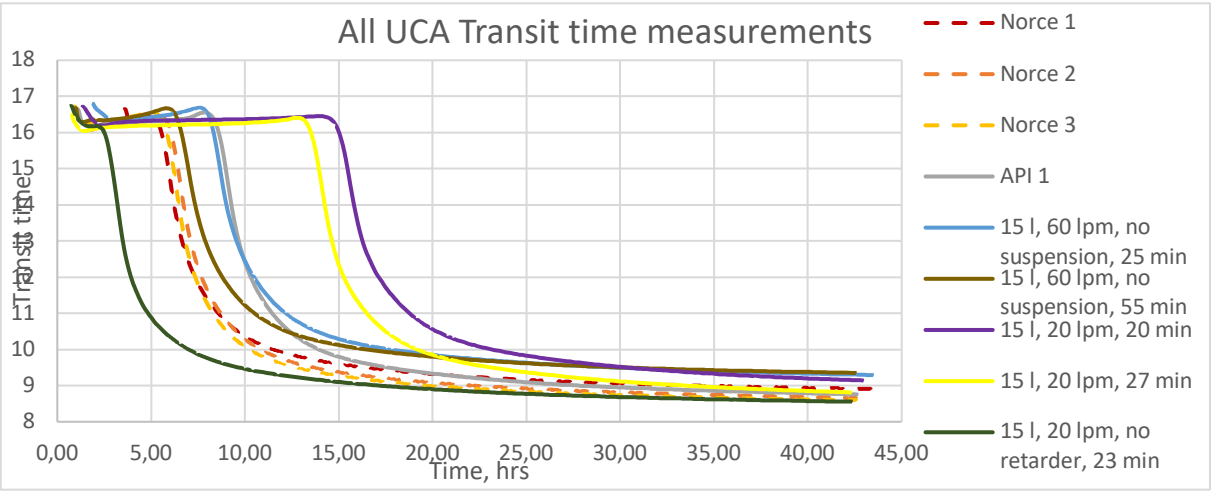


Figure 26: UCA transit time for all mixing sessions.

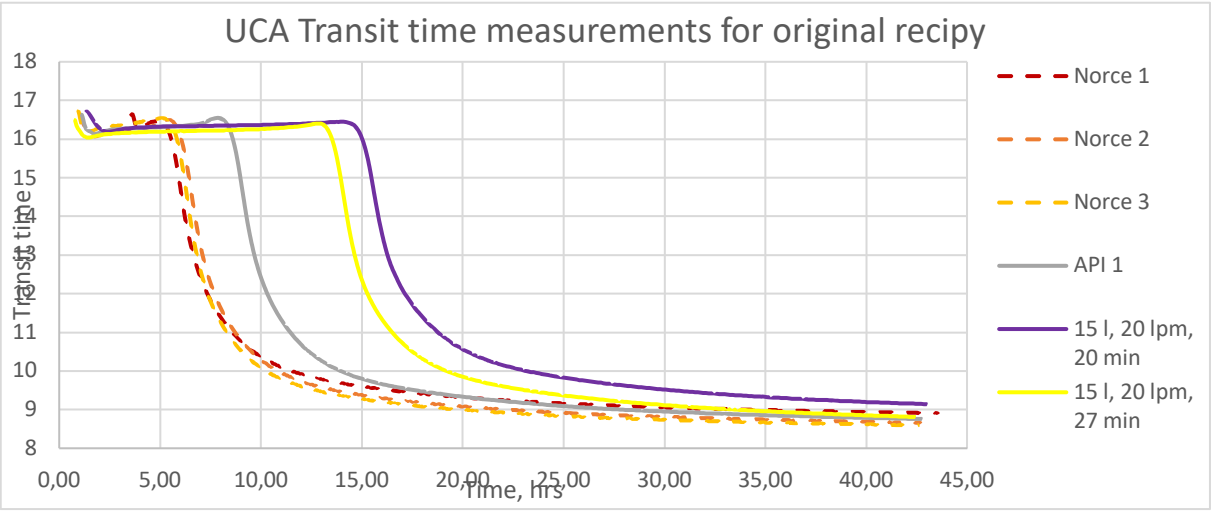


Figure 27: UCA transit time for all mixing sessions with original cement recipe.

When we compare the mixing sessions with the same cement recipe in figure 27, there may be a correlation between the Π groups and setting time. High Π_1 value correlates with longer setting time, and low Π_1 correlates with shorter setting time. For Π_2 it is opposite, high Π_2 value correlates with shorter setting time.

When we compare the two mixing sessions where we took samples at different time intervals in figure 28, we can see that in both cases the samples with longer mixing time has a shorter setting time.

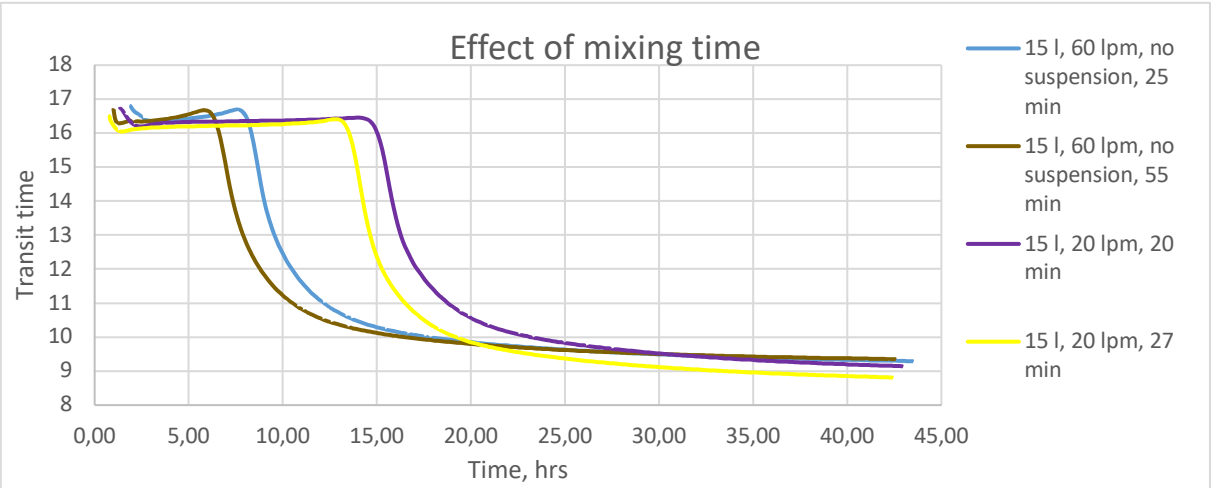


Figure 28: The effect of mixing time on the setting time.

We can see a clear effect of the retarder when we compare the 15 liters, 20lpm mixing sessions with and without retarder in figure 29. The sample without retarder agent hardens much faster than the sample with the retarder agent.

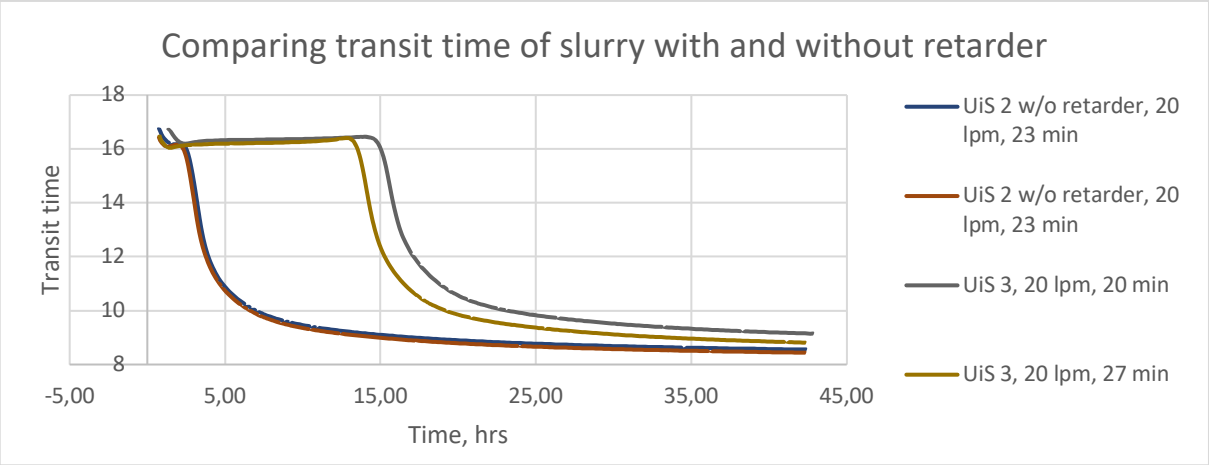


Figure 29: The effect of the retarder agent.

4.4.2 Strength and flexibility measurements

In figure 30, the UCS, or max stress, seems to be similar for all the mixing sessions, with exception of the 3rd NORCE mixing session. For the 1st and 2nd NORCE mixing sessions, samples were taken straight after mixing and conditioning. For the 3rd NORCE mixing sessions, the slurry was still in the tank for 16 minutes before the samples were taken. This might have caused some particles in the slurry to drop out and weakened the resulting cement strength. The standard deviation (SD) of the samples from the 3rd NORCE mxing session is also quite big, which means there were a large variation in the strength of the samples.

We observe that the UCS is similar for all the mixing session (except 3rd NORCE mixing session), and this can imply that varying mixing procedures, SME and Π groups, result in similar strength. This suggests that the cement is robust and that it is possible to vary the mixing procedure and still get cement with similar strength.

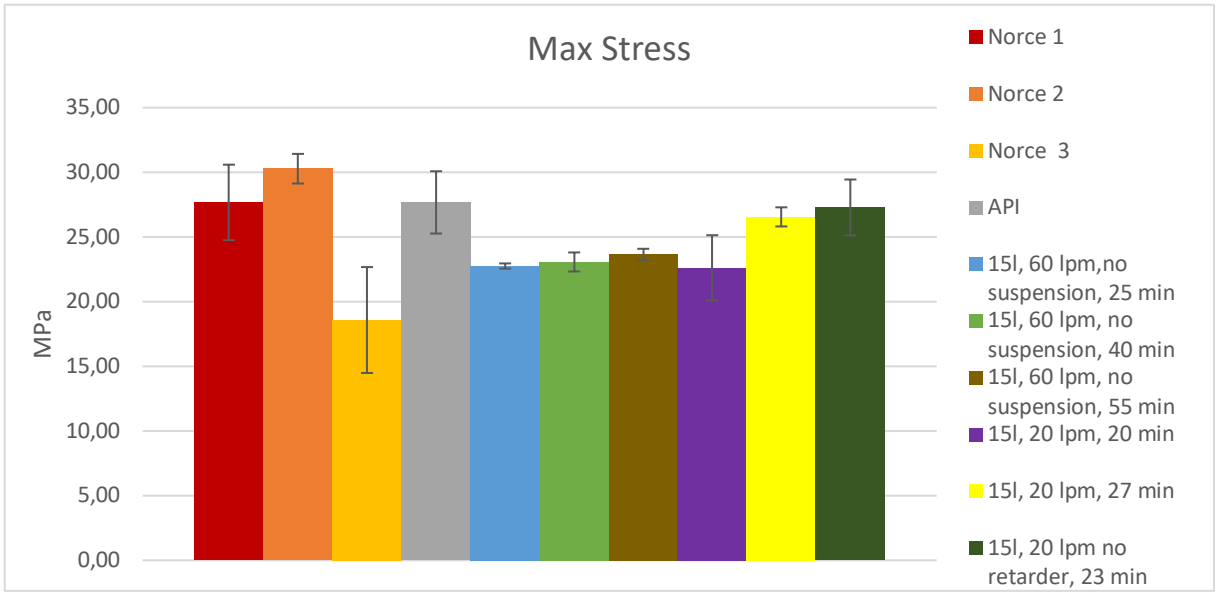


Figure 30: Max stress measurements of all mixing sessions.

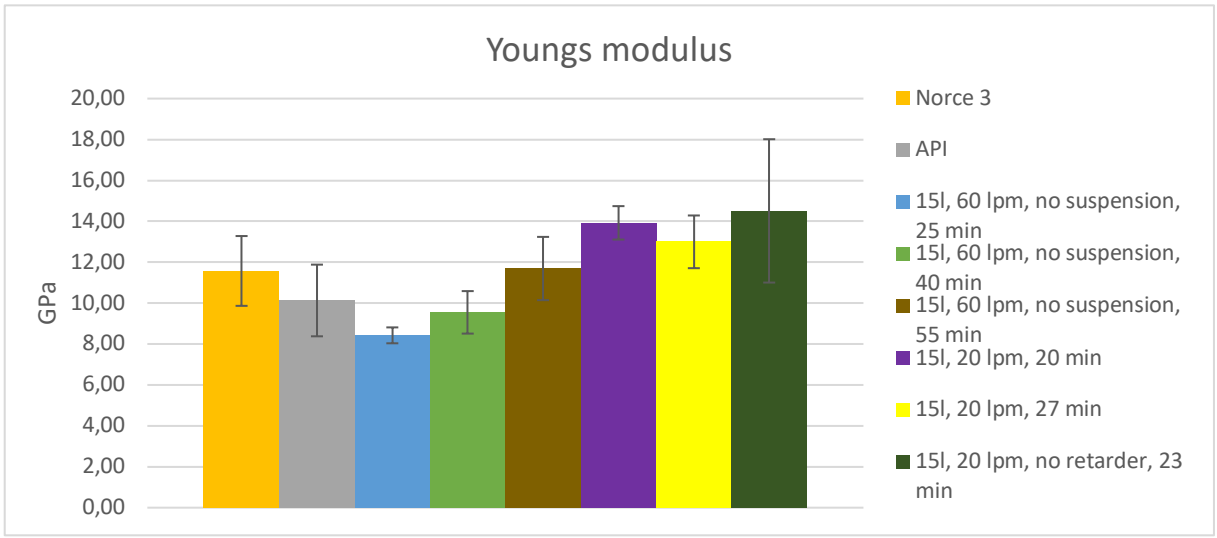


Figure 31: Young´s modulus measurements of all mixing sessions.

In the Young´s modulus measurements (fig. 31) we found no correlation with SME nor Π_2. There are no Young´s modulus measurements for 1st and 2nd NORCE mixing sessions as there was no extensometer available.

There may be a correlation between the Young´s modulus measurements and Π_1. The 15 liters, 20 lpm mixing sessions have the highest Young´s modulus measurements and the highest Π_1 values.

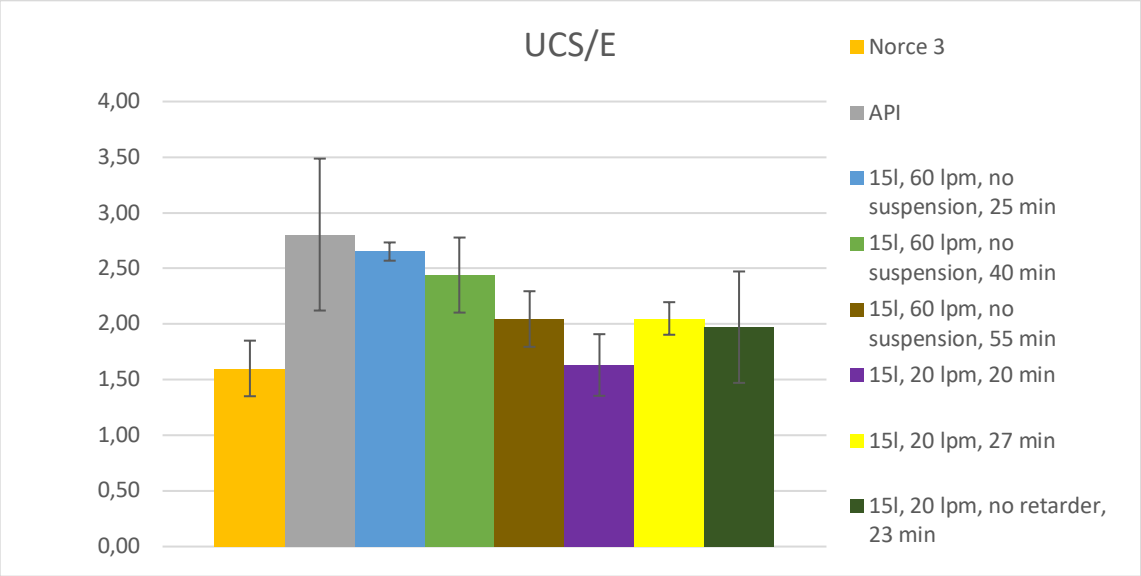


Figure 32: UCS/E calculations of all mixing sessions.

In these UCS/E measurements we found no correlation with SME, Π_1 nor Π_2.

But there is one interesting observation. The best quality cement, with regards to high strength and high flexibility, is the cement mixed with the API standard mixing procedure. This may indicate the same as Orban and his team, that the mixing energy for optimal cement slurry should be similar to the API specification 10.

4.4.3 Rheology measurements

The rheology measurements from the 2nd and 3rd NORCE mixing sessions were done by the laboratory technicians at NORCE. The rheology measurements from the API and UiS 15 liters mixing unit were done by us at UiS by using the automatic program for cement analyzes with the Ofite model 900 Viscometer.

The Herschel-Bulkley (H-B) model was calculated in excel using the problem solver tool. The H-B values were calculated using the shear rate and random H-B parameters, and the problem solver tool in Excel was used to adjust the H-B parameters to get the closest match to the shear rate readings.

We do see a clear trend for the 3 samples without suspension, the slurry is much less viscous. This does show that the suspension additive does give the slurry a clear increased viscosity.

For the two data sets that were taken at different timespans, UiS 15 liters 60 lpm and UiS 15 liters 20 lpm, we see that viscosity over time does not change much. The SME does increase but the measurements stay similar.

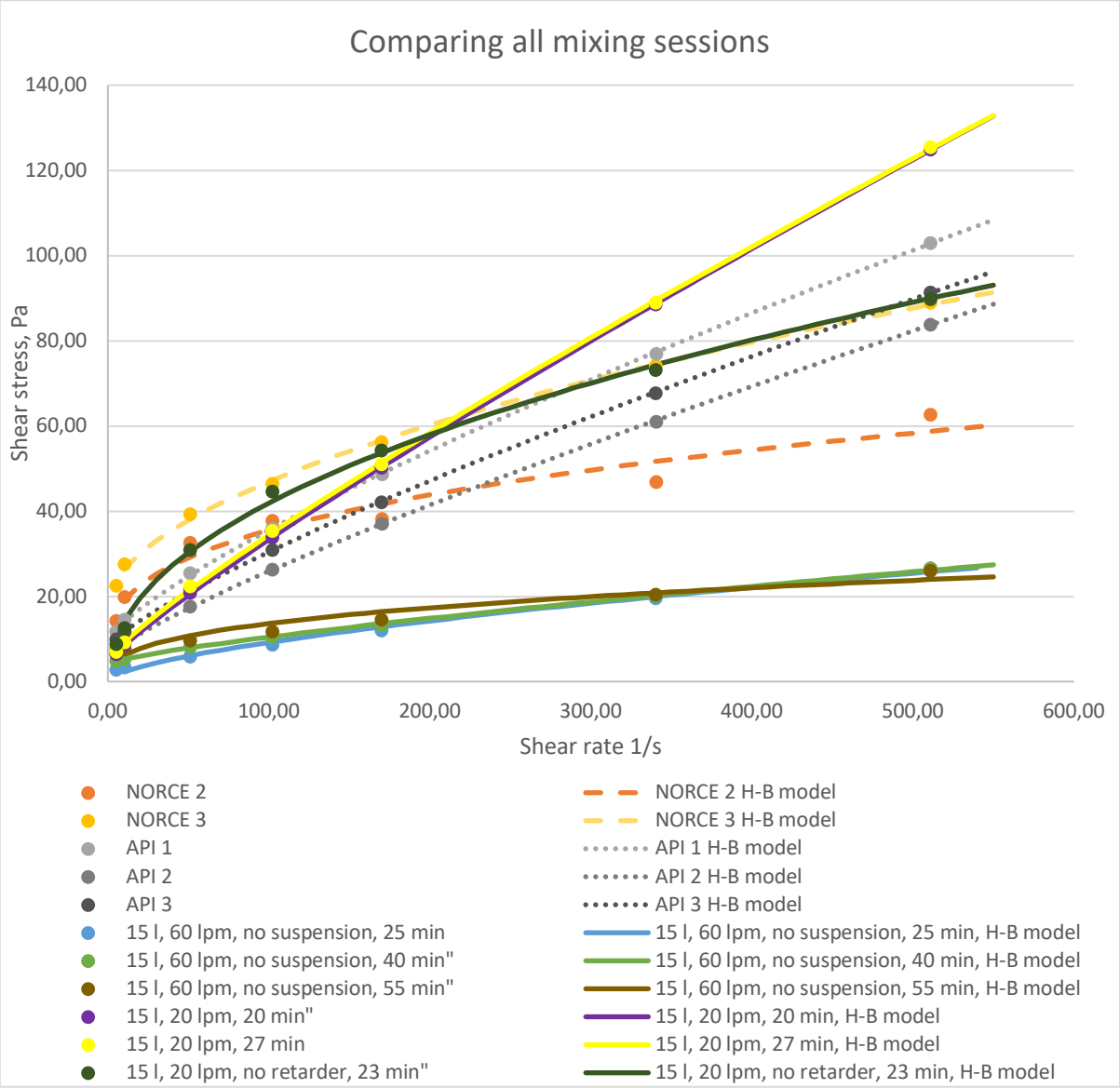


Figure 33: Rheology measurements for all mixing sessions.

In figure 34, where we compare the rheology measurements of the NORCE mixing sessions, we see that there is a gap between the two viscosity measurements. This might be due to the difference in volume. The 2nd NORCE mixing session was for 327 liters slurry and the 3rd NORCE mixing session was for 546 liters slurry. The mixing procedure is similar, hence the shear rate is the same, but since the volume differs, both the SME and Π_2 is higher for the 2nd NORCE mix.

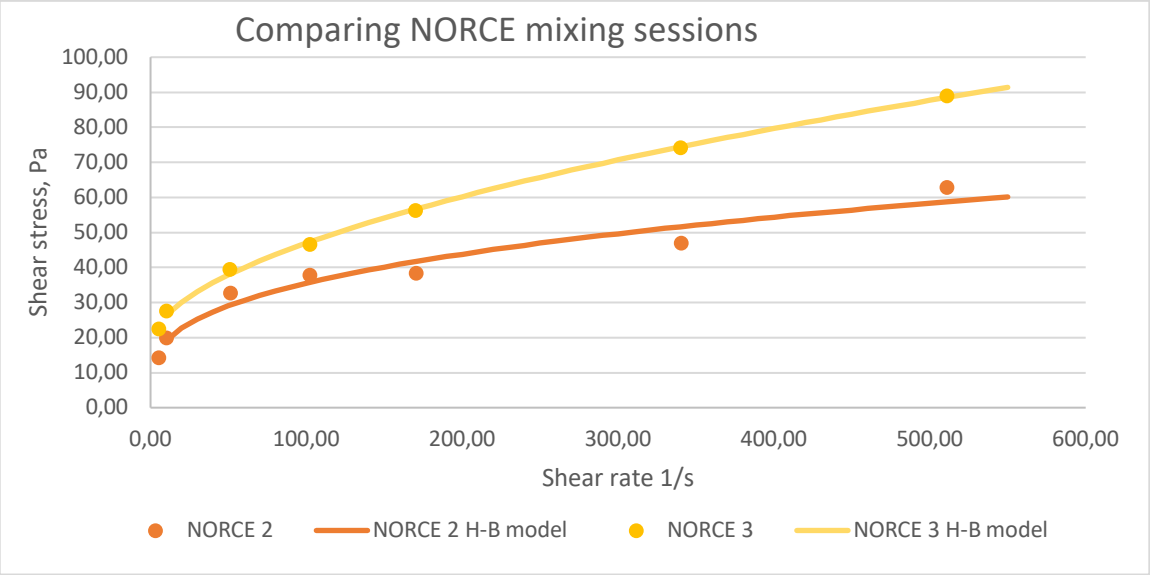


Figure 34: Rheology measurements for the NORCE mixing sessions.

From figure 35 we see no clear correlation between SME nor the Π groups.

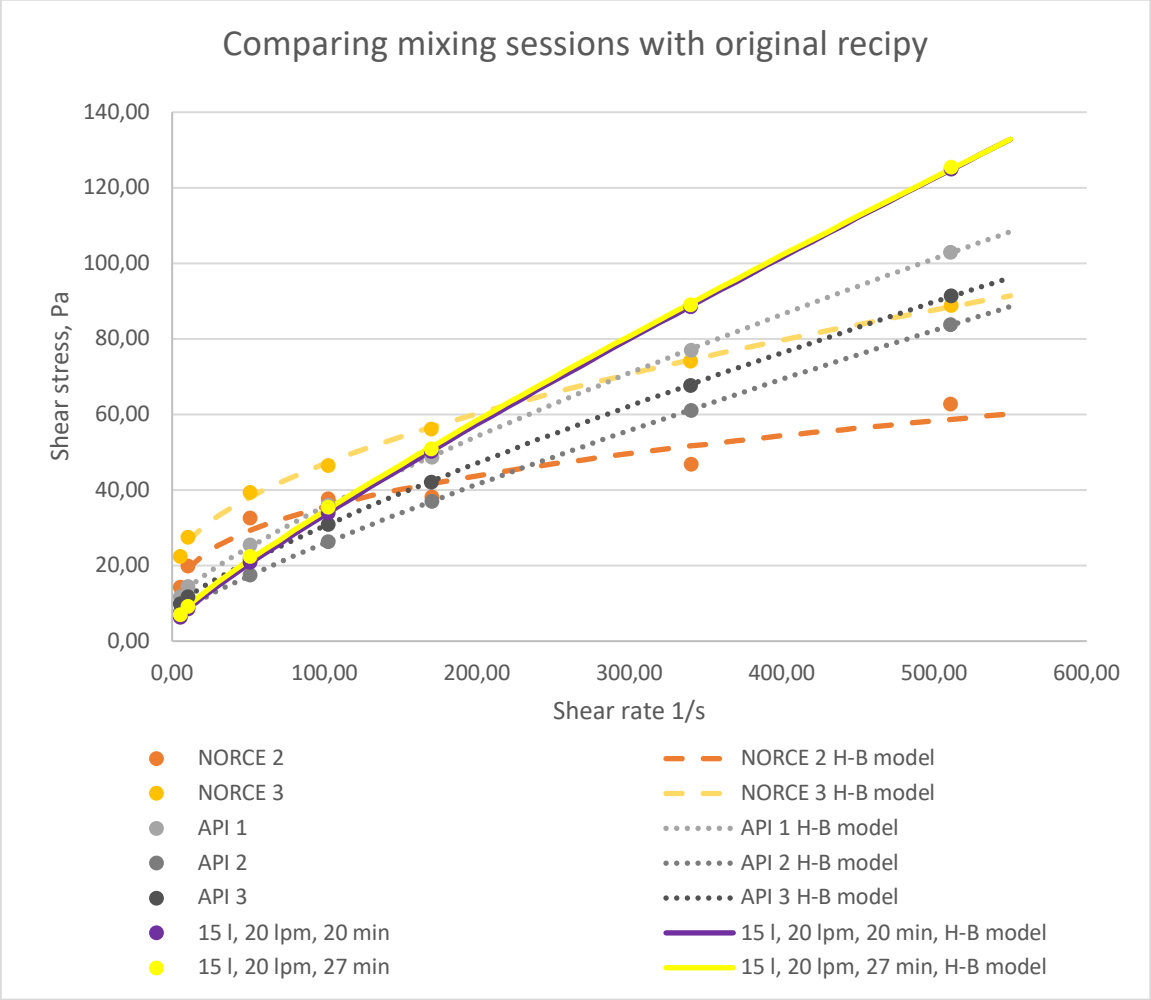


Figure 35: Rheology measurements for mixing sessions with original cement recipe.

From figure 36 we can see that there is also a difference in the viscosity measurements for the three different API mixing sessions. The reason may be the inaccurate measuring of the additives. As the additives are of very small volumes and weight, the measuring error is higher than during the larger mixes.

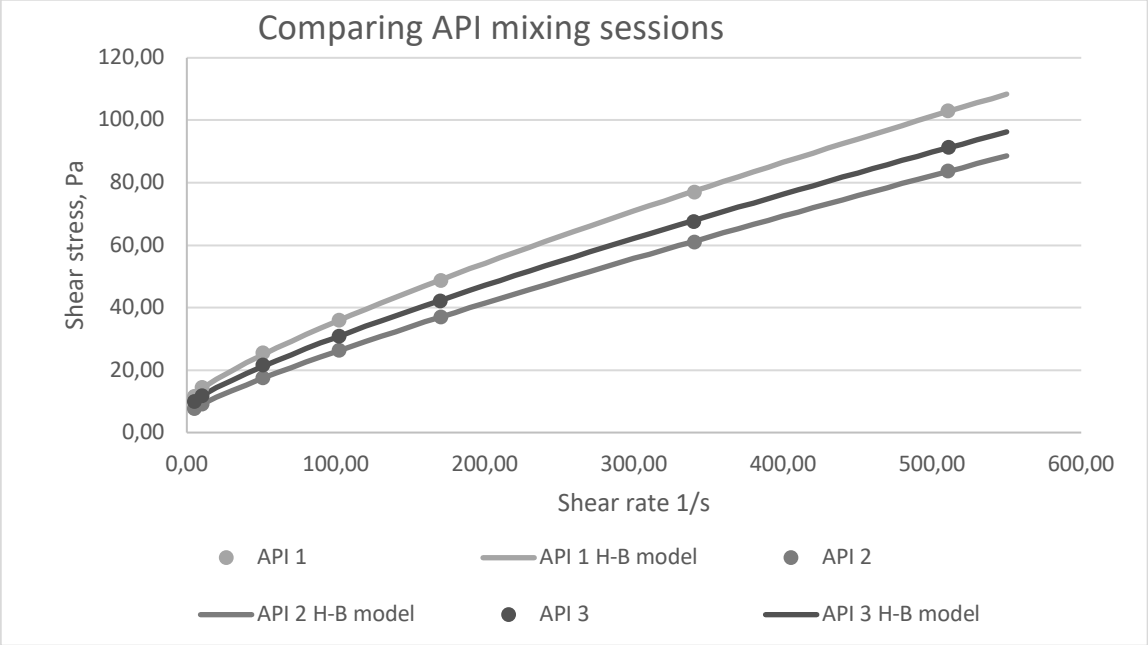


Figure 36:Rheology measurements for the API mixing session.

From figure 37 we see that without retarder the slurry is more shear thinning than the slurry of the original recipe.

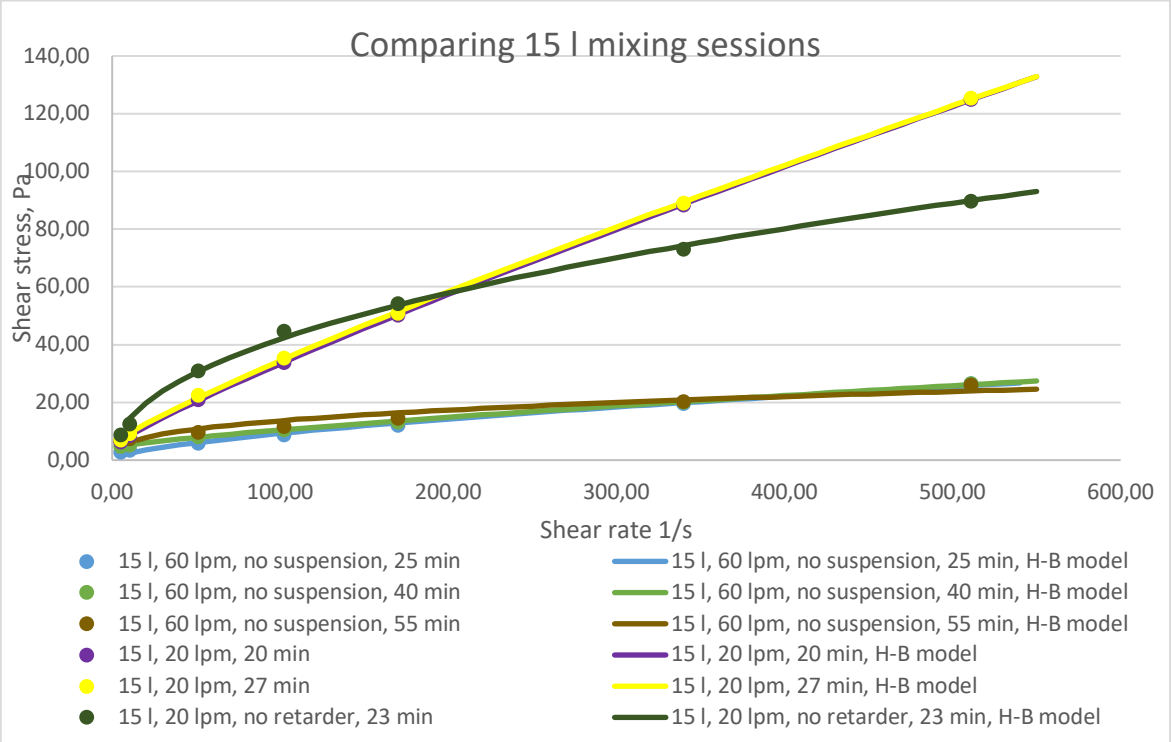


Figure 37: Rheology measurements for the UiS 15 liters mixing sessions.

4.4.4 Free water measurements

Free water seems to be due to the lack of additives in the slurry. This can suggest that free water can be controlled by additives, and that the difference in mixing procedures does not influence the free water.

When we look at the 15 liters, 60 lpm mixing sessions, (where there is no suspension agent) we can see that free water is time dependent. The longer we mix, the less free water.

We also do see much more free water in the slurry when there is no retarder agent added. This may be due to the low mixing energy. Or it might indicate that the retarder agent is very influential on free water.

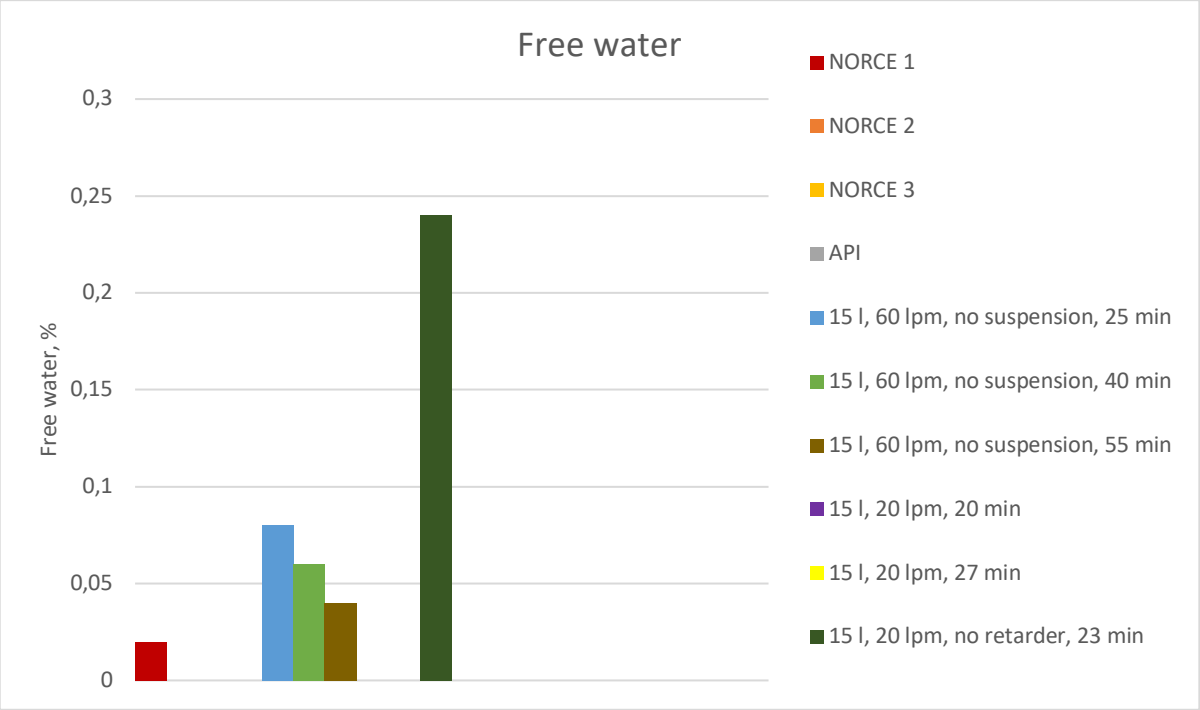


Figure 38: Free water measurements for all mixing sessions.

Chapter 5: Conclusion

For the mixing sessions with the similar SME, we did not see any similarity in neither cement slurry properties nor hardened cement properties. This suggests that, like Saleh and Teodoriu concluded, the mixing energy theory is not consistent, and slurries mixed with similar SME does not necessarily have the similar properties.

Two of our full-scale mixing sessions at NORCE were divided into two phases, a high energy with high flowrate mixing phase, and a low energy with low flowrate conditioning phase. When we compared the SME and the Π groups for these phases, we see that the conditioning phase does not have too much influence on the SME value. But the conditioning phase does have a great influence on the Π_1 value. The conditioning phase does also influence the Π_2 value but not to the same extend.

The Norce mixing sessions very different SME values but did have similar shear rate. The Norce mixing sessions also had a similar setting time. This can indicate that the setting time is influenced by shear rate, and not SME. This is consistent with Hibbert and his team´s statements that "the critical parameter is not the absolute value of mixing energy, but the way in which this is applied with low and high shear of the slurry during its recirculation through a centrifugal pump."(Hibbert et al., 1995, p. 52)

The strength of the cement, the UCS, is similar for most of the mixing session and this can imply that varying mixing procedures, SME and Π groups, result in similar strength. This suggests that the cement is robust and that it is possible to vary the mixing procedure and still get cement with similar strength.

There was no correlation between UCS/E and SME, Π_1 nor Π_2. But we did see that the best quality cement, with regards to high strength and high flexibility (UCS/E), is the cement mixed with the API standard mixing procedure. This may indicate the same as Orban and his team, that the mixing energy for optimal cement slurry should be similar to the API specification 10.

Both additives did show a great efficiency. We also saw that free water seemed to be due to the lack of additives in the slurry, especially the retarder agent. This can suggest that free water can be controlled by additives, especially the retarder agent, and that the difference in mixing procedures does not influence the free water.

Chapter 6: Lessons Learned and Further research

The main issues we had with the 15 liters mixing unit setup was:

- Pump shut down during the mixing due to overheating or blocked impeller
- Difficult to get the last 30% of the dry ingrediencies mixed in the slurry.
- Dry ingrediencies and thick slurry stuck on the wall of the tank, and on the pump.

An improvement of the experiment could be to use a stronger pump. The pump stopped several times while mixing the slurry, this might have been due to the overheating of the pump. The pump had an overheating protection and would stop if the temperature of the pump or slurry would exceed 35 degrees Celsius. Another reason for the pump to stop can be that the impeller was blocked by cement lumps in the slurry.

It would be of great benefit to place the pump outside the tank. This would enlarge the space in the tank to add the ingrediencies, there would be less surfaces that the cement and slurry could stick to, and it would give space for an agitator that would help mix in the dry ingrediencies.

The setup of the 15 liters mixing unit would also improve if we used a tank with a funnel bottom, and a circulation loop that would go from the bottom of the tank to the pump, and then back to the tank. This would ease the workload of the pump to get the slurry into the impeller before pumping it through the circulation loop. This would also give more space in the mixing tank adding the dry ingrediencies. An alternative setup of the mixing unit is shown in figure 39.

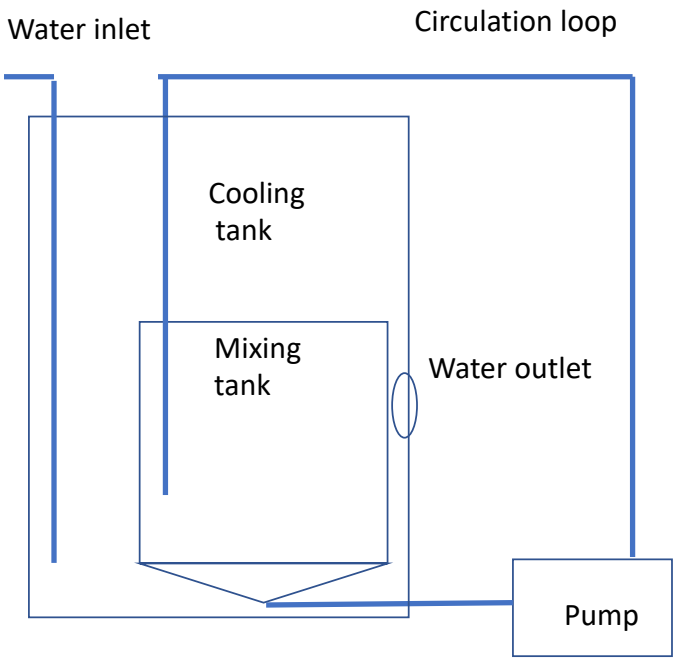


Figure 39: An alternative setup of the UiS 15 liters mixing unit.

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Appendix A: UCS and Young´s modulus calculations

1st NORCE mixing session

Norce 1 Specimen	Max Stress (Mpa)	E (Gpa) based on crosshead measurements
CP1	26,33	2,2415
CP2	30,37	2,13
CP3	29,78	2,09
CP4	24,21	2,10
Mean	27,67	2,14
SD	2,92	0,07

2nd NORCE mixing session

Norce 2 Specimen	Max Stress (MPa)	E (Gpa) based on crosshead measurements
CP1	**31,09**	**2,6105**
CP2	no data due to oscillation	2,47
CP3	29,47	2,63
CP4	no data due to oscillation	2,51
Mean	30,28	2,55
SD	1,15	0,08

API mixing session

API Specimen	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E
CP 1	Broken before test		

CP2	25,39	10,03	2,53
CP3	27,44	11,93	2,30
CP4	30,19	8,43	3,58
Mean	27,67	10,13	2,80
SD	2,41	1,75	0,68

3rd NORCE mixing session

Norce 3 Specimen	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E	E (Gpa) based on crosshead measurements
CP1	13,93	9,91	1,41	2,93
CP2	20,18	13,32	1,52	2,72
CP3	21,63	11,49	1,88	2,88
Mean	18,58	11,57	1,60	2,84
SD	4,09	1,71	0,25	0,11

15 liters, 60 lpm, no suspension agent, 25 minutes

15 l, 60 lpm, 25 min	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E	E (Gpa) based on crosshead measurements
CP1	23,04	8,33	2,77	2,44
CP2	Broken after load-deload measurements	8,09		2,41
CP3	22,47	8,86	2,54	2,47
Mean	22,76	8,42	2,65	2,44
SD	0,40	0,40	0,16	0,03
SD	0,20	0,39	0,08	0,03

15 liters, 60 lpm, no suspension agent, 40 minutes

15 l,60 lpm, 40 min	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E
CP1	23,71	8,57	2,77
CP2	22,27	10,64	2,09
CP3	23,23	9,44	2,46
Mean	23,07	9,55	2,44
SD	0,74	1,04	0,34

15 liters, 60 lpm, no suspension agent, 55 minutes

15 l, 60 lpm, 55 min	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E
CP1	23,88	12,91	1,85
CP2	23,16	9,95	2,33
CP3	23,93	12,21	1,96
Mean	23,66	11,69	2,05
SD	0,43	1,55	0,25

15 liters, 20 lpm, 20 minutes

15 l, 20 lpm, 20 min	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E
CP1	24,40	13,35	1,83
CP2	20,83	14,50	1,44
Mean	22,61	13,93	1,63
SD	2,52	0,82	0,28

15 liters, 20 lpm, 27 minutes

15 l, 20 lpm, 27 min	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E
CP1	26,03	12,09	2,15
CP2	27,08	13,91	1,95
Mean	26,55	13,00	2,05
SD	0,74	1,29	0,15

15 liters, 20 lpm, no retarder agent, 23 minutes

UiS 20 lpm, 23 min	Max Stress (MPa)	E (Gpa) based on extensometer	UCS/E
CP1	23,80	11,84	2,01
CP2	30,33	10,95	2,77
CP3	26,46	18,34	1,44
CP4	27,24	13,16	2,07
CP5	27,56	19,39	1,42
CP6	28,34	13,38	2,12
Mean	27,29	14,51	1,97
SD	2,16	3,50	0,50

Appendix B: Fann 35 measurements

There were no rheology measurements for the first NORCE mixing session as the mixing was at the NORCE research center, and the Fann 35 was UiS. We set up a Fann 35 at the NORCE research center for the two next mixing sessions at NORCE.

2nd NORCE mixing session

RPM	Ramp up reading	Ramp down reading	Average reading **θ**	Shear rate (1/s)	Shear stress (**θ** * 0,51)
3	28	28	28	5,10	14,28
6	39	39	39	10,20	19,89
30	64	64	64	170,00	32,64
60	74	74	74	340,00	37,74
100	75	75	75	170,00	38,25
200	92	92	92	340,00	46,92
300	123	123	123	511,00	62,73

3rd NORCE mixing session

RPM	Ramp up reading	Ramp down reading	Average reading **θ**	Shear rate (1/s)	Shear stress (**θ** * 0,51)
3	**44**	**44**	44	5,10	22,44
6	**54**	**54**	54	10,20	27,54
30	**77**	**77**	77	170,00	39,27
60	**91**	**91**	91	340,00	46,41
100	**110**	**110**	110	170,00	56,10
200	**145**	**145**	145	340,00	73,95
300	**174**	**174**	174	511,00	88,74

1st API mixing session

RPM	Ramp up reading	Ramp down reading	Average reading ($\boldsymbol{\theta}$)	Shear rate (1/s)	Shear stress ($\boldsymbol{\theta}$*0,51)
3	19,9	26,1	23	5,10	11,73
6	27,4	29,6	28,5	10,20	14,54
30	50	50	50	170,00	25,50
60	71	70	70,5	340,00	35,96
100	97	94	95,5	170,00	48,71
200	153	149	151	340,00	77,01
300	202	202	202	511,00	103,02

2nd API mixing session

RPM	Ramp up reading	Ramp down reading	Average reading ($\boldsymbol{\theta}$)	Shear rate (1/s)	Shear stress ($\boldsymbol{\theta}$*0,51)
3	14,8	15,3	15,05	5,10	7,68
6	17,5	18,3	17,9	10,20	9,13
30	34	35	34,5	170,00	17,60
60	51	52	51,5	340,00	26,27
100	72	73	72,5	170,00	36,98
200	119	120	119,5	340,00	60,95
300	164	164	164	511,00	83,64

3rd API mixing session

RPM	Ramp up reading	Ramp down reading	Average reading ($\boldsymbol{\theta}$)	Shear rate (1/s)	Shear stress ($\boldsymbol{\theta}$*0,51)
3	18,8	20,3	19,55	5,10	9,97
6	23	23,3	23,15	10,20	11,81

30	43	42	42,5	170,00	21,68
60	61	60	60,5	340,00	30,86
100	84	81	82,5	170,00	42,08
200	134	131	132,5	340,00	67,58
300	179	179	179	511,00	91,29

15 liters, 60 lpm, 25 minutes

RPM (N)	Ramp up reading	Ramp down reading	Average reading θ	Shear rate (1/s)	Shear stress (θ * 0,51)
3	**5,1**	**6,1**	5,6	5,10	2,86
6	**6**	**7,3**	6,65	10,20	3,39
30	**11**	**12**	11,5	170,00	5,87
60	**16**	**18**	17	340,00	8,67
100	**23**	**24**	23,5	170,00	11,99
200	**38**	**39**	38,5	340,00	19,64
300	**52**	**52**	52	511,00	26,52

15 liters, 60 lpm, 40 minutes

RPM (N)	Ramp up reading	Ramp down reading	Average reading θ	Shear rate (1/s)	Shear stress (θ * 0,51)
3	**8,6**	**10,5**	9,55	5,10	4,87
6	**9,7**	**11,1**	10,4	10,20	5,30
30	**15**	**17**	16	170,00	8,16

60	**20**	**21**	20,5	340,00	10,46
100	**25**	**27**	26	170,00	13,26
200	**39**	**40**	39,5	340,00	20,15
300	**52**	**52**	52	511,00	26,52

15 liters, 60 lpm, 55 minutes

RPM (N)	Ramp up reading	Ramp down reading	Average reading $\boldsymbol{\theta}$	Shear rate (1/s)	Shear stress ($\boldsymbol{\theta}$ * 0,51)
3	**12,3**	**14,6**	13,45	5,10	6,86
6	**14,1**	**15,4**	14,75	10,20	7,52
30	**18**	**20**	19	170,00	9,69
60	**22**	**24**	23	340,00	11,73
100	**27**	**30**	28,5	170,00	14,54
200	**39**	**41**	40	340,00	20,40
300	**51**	**51**	51	511,00	26,01

15 liters, 20 lpm, 20 minutes

RPM (N)	Ramp up reading	Ramp down reading	Average reading $\boldsymbol{\theta}$	Shear rate (1/s)	Shear stress ($\boldsymbol{\theta}$ * 0,51)
3	**12,8**	**12,4**	12,6	5,10	6,43
6	**17,4**	**16,4**	16,9	10,20	8,62
30	**42**	**40**	41	170,00	20,91
60	**68**	**65**	66,5	340,00	33,92
100	**100**	**97**	98,5	170,00	50,24
200	**175**	**172**	173,5	340,00	88,49
300	**245**	**245**	245	511,00	124,95

15 liters, 20 lpm, 27 minutes

RPM (N)	Ramp up reading	Ramp down reading	Average reading θ	Shear rate (1/s)	Shear stress (θ * 0,51)
3	**13,9**	**13,6**	13,75	5,10	7,01
6	**18,1**	**17,9**	18	10,20	9,18
30	**45**	**43**	44	170,00	22,44
60	**70**	**69**	69,5	340,00	35,45
100	**101**	**99**	100	170,00	51,00
200	**176**	**173**	174,5	340,00	89,00
300	**246**	**246**	246	511,00	125,46

15 liters, 20 lpm, no retarder agent, 23 minutes

RPM (N)	Ramp up reading	Ramp down reading	Average reading θ	Shear rate (1/s)	Shear stress (θ * 0,51)
3	**14**	**20,4**	17,2	5,10	8,77
6	**20,3**	**29**	24,65	10,20	12,57
30	**60**	**61**	60,5	170,00	30,86
60	**89**	**86**	87,5	340,00	44,63
100	**107**	**106**	106,5	170,00	54,32
200	**144**	**143**	143,5	340,00	73,19
300	**176**	**176**	176	511,00	89,76

Appendix C: Free water measurements

Free water	ml water loss	% water loss
Norce 1	5	0,02
Norce 2	0	0
Norce 3	0	0
API	0	0
15 l, 60 lpm, no suspension, 25 min	20	0,08
15 l, 60 lpm, no suspension, 40 min	15	0,06
15 l, 60 lpm, no suspension, 55 min	10	0,04
15 l, 20 lpm, 20 min	0	0
15 l, 20 lpm, 27 min	0	0
15 l, 20 lpm, no retarder, 23 min	60	0,24

Appendix D: Logged measurements from the 15 liters mixing sessions

15 liters,60 lpm, without suspension agent						
spoons of dry material	t (sec)	t (min)	Watt	E (Nm)	Total E (Nm)	SME (kJ/kg)
0	0	0,00	675		0	0,00
10	80	1,33	720	57600	57600	2,00
20	170	2,83	760	68400	126000	4,38
30	240	4,00	800	56000	182000	6,32
40	310	5,17	830	58100	240100	8,34
50	380	6,33	860	60200	300300	10,43
60	445	7,42	880	57200	357500	12,41
70	540	9,00	905	85975	443475	15,40
80	615	10,25	925	69375	512850	17,81
90	700	11,67	940	79900	592750	20,58
100	810	13,50	950	104500	697250	24,21
110	890	14,83	980	78400	775650	26,93
120	1030	17,17	990	138600	914250	31,74
130	1160	19,33	1010	131300	1045550	36,30
130	1500	25,00	1080	367200	1412750	49,05
130	2400	40,00	1080	972000	2384750	82,80
130	3300	55,00	1080	972000	3356750	116,55

15 liters, 20 lpm, without retarder agent						
spoons of dry material	t (sec)	t (min)	Watt	E (Nm)	Total E (Nm)	SME
0	0	0,00	610		0	0,00
10	60	1,00	640	38400	38400	1,33

20	150	2,50	670	60300	98700	3,43
30	240	4,00	665	59850	158550	5,51
40	340	5,67	660	66000	224550	7,80
50	440	7,33	660	66000	290550	10,09
60	530	8,83	665	59850	350400	12,17
70	620	10,33	660	59400	409800	14,23
80	710	11,83	663	59670	469470	16,30
90	810	13,50	665	66500	535970	18,61
100	980	16,33	667	113390	649360	22,55
110	1070	17,83	685	61650	711010	24,69
120	1190	19,83	690	82800	793810	27,56
130	1280	21,33	680	61200	855010	29,69
140	1385	23,08	670	70350	925360	32,13

15 liters, 20 lpm

spoons of dry material	t (sec)	t (min)	Watt	E (Nm)	Total E (Nm)	SME
0	0	0,00	580	0	0	0,00
10	60	1,00	605	36300	36300	1,26
20	140	2,33	620	49600	85900	2,98
30	220	3,67	635	50800	136700	4,75
40	295	4,92	640	48000	184700	6,41
50	345	5,75	645	32250	216950	7,53
60	420	7,00	650	48750	265700	9,23
70	485	8,08	665	43225	308925	10,73
80	570	9,50	665	56525	365450	12,69
90	655	10,92	660	56100	421550	14,64
100	740	12,33	670	56950	478500	16,61
110	810	13,50	690	48300	526800	18,29
120	890	14,83	705	56400	583200	20,25

130	985	16,42	695	66025	649225	22,54
140	1085	18,08	695	69500	718725	24,96
150	1200	20,00	695	79925	798650	27,73
150	1650	27,50	695	312750	1111400	38,59