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Statistical Analysis of Rheological characterization of Water-based Drilling Fluids

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Abstract

Of fundamental importance in drilling technology is the drilling fluid. It has to counter varying wellbore conditions, and still be able to fulfil its objectives like suspension and transport of solids, pressure control and wellbore stabilization. Viscosity, density and shear behaviour impact the ability to handle these tasks. Rheological properties are being affected when subjected to variations in parameters like temperature, shear stress and pressure. For deeper and longer wells, surrounding conditions is also getting more extreme, which increases the requirements for specialized drilling fluids. In order to design these appropriately, behavioural knowledge is important. Influence of temperature and degradation are two subjects being investigated in more detail in this report. Such information is obtained through manual or automated tests. Reliability for obtained results is essential whether it is by automated or manual means.

This study was a follow up to Priyadharshini Saptharishi's Master thesis entitled "*Rheological Characterization of Water-based Drilling Fluids- a Comparative Analysis of Manual and Automated measurements*" (2016). Questions was raised related to uncertainties and reliability of the experiments involved. This project was trying to verify repeatability, and provide estimates for the uncertainties by the use of statistical methods. Rheological behaviour related to temperature and time dependency was also investigated.

In this report, results from Fann viscometer tests carried out for a selection of fluids containing poly-anionic cellulose (PAC), Xanthan Gum (XG) and barite will be presented. Compositions were chosen on a comparable basis to Saptharishi (2016). By using an increased number of measurements, repeatability could be assessed, and used as a foundation to compare the previously obtained results from Saptharishi (2016).

The results revealed a strict inverse relation between temperature and viscosity as expected. Degradation over time was significant, but varied amongst the compositions. Mixtures of pure PAC, and with additions of barite, had a decrease in viscosity over a time window of 14 days, in the order of 30-75% – the lower concentration of PAC being the most stable of these. Samples containing XG had a rapid decrease in viscosity during the first 48 hours, before they did show the most stable behaviour during the rest of the time window. The degradation of

fluids makes it important to add stabilizers in order to slow down this rate, and maintain the desired properties.

The repeatability found in measurements performed during this project appeared to be of a precise nature, with variations from arithmetic mean values in the order of 0.5 to 2.3%. Comparing Saptharishi's (2016) results with 95 % confidence intervals created on the basis of results from this project, none of the compositions did fit. For three compositions, deviations from mean values was in the order of 8-10 %, while the lower concentration of PAC deviated with 28%. On this basis, it was concluded that the experiments could not be assessed as reliable. It was reasonable to ascribe procedural inequalities as of one of the main reasons for this, which emphasizes the need for detailed standardization in procedures, in order to obtain comparable and reliable results from experiments.

Sammendrag

Borevæske er av fundamental viktighet innen boreteknologi. Den har som funksjon å takle varierende nedihulls forhold, mens den oppfyller de krav som stilles til oppgaver som transport av borekaks, trykk-kontroll og stabilisering av borehull. Viskositet, tetthet og skjærspenninger påvirker evnen til å gjennomføre disse oppgavene. Rheologiske egenskaper blir påvirket når de blir utsatt for parametere som temperatur, skjærkraft og trykk. For dypere og lengre brønner blir også de omgivende forhold mer ekstreme, som påvirker de krav som blir satt for spesialiserte borevæsker. For å kunne designe disse på en tilfredsstillende måte er det viktig å ha kunnskap om væskens oppførsel. Påvirkning av temperatur og forringelse er to faktorer som vil bli undersøkt nærmere i denne rapporten. Informasjon om slike påvirkninger blir tilegnet gjennom manuelle og automatiserte tester. Påliteligheten for tilegnede resultater er ekstremt viktig, uansett om de er oppnådd ved hjelp av manuelle eller automatiserte hjelpemidler.

Denne studien var en oppfølger til Priyadharshini Saptharishi’s master-oppgave ved tittel “*Rheological Characterization of Water-based Drilling Fluids- a Comparative Analysis of Manual and Automated measurements*” (2016). Spørsmål ble stilt hvorvidt de eksperimentielle resultatene var pålitelig og inneholdt usikkerhet. Gjennom denne oppgaven skulle det verifiseres hvorvidt de foregående resultater var repeterbare samt skaffe estimater for usikkerheten ved hjelp av statistiske metoder. Rheologisk oppførsel relatert til temperatur og tidsavhengighet skulle også undersøkes.

I denne rapporten blir det presentert resultater fra Fann viskometer, gjennomført for et utvalg av fluider med innhold av poly-anionisk cellulose (PAC), Xanthan Gum (XG) og baritt. Sammensetningene ble valgt ut fra et sammenligningsgrunnlag til Saptharishi (2016). Gjennom å bruke et større antall målinger, kunne repeterbarheten anslåes og bli brukt som et grunnlag for å sammenligne resultater fra Saptharishi (2016).

Resultatene viste et tett relatert forhold mellom temperatur og viskositet, som forventet. Forringelsen over tid var signifikant, men varierte mellom de ulike komposisjonene. Miksturene av ren PAC, og med tilsatt baritt, hadde en reduksjonen i viskositet over et

tidsvindu på 14 dager, i størrelsesorden 30-75%. Den laveste konsentrasjonen var den mest stabile av disse. Prøvene som inneholdt XG hadde en rask reduksjon i viskositet over de første 48 timene, før de viste stabil oppførsel gjennom resten av tidsvinduet. Forringelsen av fluidene medfører at det er viktig å tilsette stabilisatorer for å bremse opp denne effekten for å bevare de ønskede egenskapene.

Repeterbarheten som ble funnet i forsøkene gjennomført for dette prosjektet, synes å være av presis natur, med variasjoner fra gjennomsnittsverdiene i størrelsesorden 0.5 til 2.3 %. Sammenligning av Saptahrishi's (2016) resultater, med 95 % konfidens intervaller laget på bakgrunn av dette prosjektet, viste at ingen av fluid-komposisjonenes resultater passet inn. For tre komposisjoner var avviket sammenlignet med gjennomsnittsverdier oppnådd i dette prosjektet i størrelsesorden 8-10%, mens den laveste konsentrasjonen av PAChadde et avvik på 28%. Med bakgrunn i dette ble det konkludert at Saptharishis (2016) eksperimenter ikke kunne bli anslått å være pålitelige. Det er rimelig å tilskrive forskjeller ved prosedyrene som hovedårsak til dette, som igjen understreker behovet for detaljert standardisering i prosedyrer for å kunne oppnå sammenlignbare og pålitelige resultater fra eksperimenter.

Preface

This Master's thesis, entitled "***Statistical Analysis of Rheological characterization of Water-based Drilling Fluids***", is submitted as the last subject in part of fulfilling the requirements for the Master of Science (M.Sc.) degree in Petroleum Engineering at the Norwegian University of Science and Technology.

The pathway from the beginning of this course at NTNU, till this moment, has been a challenging one. I want to express appreciation to my family for great support all along.

The project work has been performed at the Department of Petroleum Engineering and Applied Geophysics, NTNU, with Professor John-Morten Godhavn as supervisor and in collaboration with Statoil ASA, Rotvoll, Trondheim with Dr. Aminul Islam as co-supervisor.

I want to thank supervisor Professor John-Morten Godhavn, and Dr. Aminul Islam for guidance in planning and execution of the laboratory work. I also want to thank Principal Researcher at Statoil, Zalpato Ibragimova, for helping with explanations related to experimental work, and Mr. Roger Overå, Chief lab-technician at IPT NTNU with assistance during the laboratory work.

Trondheim, March 2017

Ole Julius Rye

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Nomenclature

Symbol

σ^2	Variance
Ea	Activation energy
R^*	Reference value
R	Universal gas constant
s^2	Variance estimate
s	Standard deviation estimate
t	Critical value in t-distribution
T	Absolute temperature
μ^*	True population value
η	Viscosity

Abbreviations

API	American Petroleum Institute
AV	Apparent viscosity
CMC	Carboxymethyl cellulose
DP	Differential pressure
IPT	Institute of Petroleum Technology
HT	High temperature
NTNU	Norwegian University of Science and Technology
OJR	Ole Julius Rye
PAC	Poly-anionic cellulose

PS	Priyadharshini Saptharishi
PV	Plastic viscosity
ROP	Rate of penetration
RPM	Revolutions per minute
SF	Speed factor
YP	Yield point
XG	Xanthan Gum

1 Introduction

In this chapter, a brief background of the project will be presented as well as problem description, assigned tasks and hypothesis. The structure of the report will also be outlined.

1.1 Background

Drilling fluid is one of the fundamental ingredients in order to drill a well. The properties of drilling fluids may be affected by duration and rate of shear, temperature and pressure. In order to characterize fluids – the term rheology is applied – which is the study of flow and deformation of materials. The most important rheological parameters within the oil and gas industry relates to viscosity, density, temperature and shear behaviour. These are closely related to the performance of the drilling fluid in order to fulfil its objectives. Solids need to be suspended in the drilling fluid in order to be transported to the surface or maintain density, wellbore need to be kept stable, and pore pressure has to be countered. Drilling technology evolves, and challenging conditions for deeper and longer wells requires specialized drilling fluids. To design appropriate mixtures, knowledge of behaviour is essential. Behaviour is measured manually or by automated tests which is becoming more common. Regardless of the method in use, it has to be accurate and reliable. Temperature is one of the parameters affecting viscosity the most, and precise knowledge about the temperature dependant behaviour is essential in creating fluids able to counter wellbore conditions. As many polymers are biodegradable, rheological characterization could be altered over time. In order to maintain the rheological profile which was created initially, knowledge about such time-dependency will be of great interest. The latter will be investigated thoroughly during this report.

1.2 The Nature of Measurements

A measurement is an attempt of assigning a value to a given property (Mandel, 1964). It may be the true value of the property, but in most cases it is not. Fluctuations will occur if one investigates at a level detailed enough, because of the surrounding conditions. If all these detailed changes are random, one will obtain a value closer to the true value when increasing the amount of measurements (Mandel, 1964). In order to gain thorough information from rheological measurements, they have to be reliable. To make reliable results, a repeatable procedure needs to be applied, and parameters able to affect the outcome have to be diminished. Even if it would be impossible to recreate exactly the same conditions between laboratories or on-site installations, arrangements of standard procedures makes it possible to create approximations that satisfies accuracy and precision of the outcome. The quantity exactness could be adjusted to the fit the need and requirements wherever it is to be applied.

1.3 Problem Description

This study is a follow up to Priyadharshini Saptharishi's Master's thesis entitled "Rheological characterization of water based drilling fluids - A Comparative analysis of Manual and automated measurements". The thesis involved examining rheological implications of PAC fluids upon addition of selected additives and investigation of their dynamic rheological properties like viscoelasticity, shear thinning, thixotropy and yield stress. In addition, the project also comprised of performing flow loop tests at the test rig facility of Statoil Bergen Sandsli using Dual differential pressure (DP) principle and comparing the automated measurements to manual measurements. Rheological modelling was performed to determine best fit model to experimental data. Fann viscometer, Offite 900 viscometer, Anton Paar rheometer and mud balance were used for manual measurements.

Questions have been raised about the uncertainties and reliability of the experiments. This follow up project shall try to verify repeatability of the results and provide estimates of the uncertainty by using statistical methods. The laboratory work makes a considerable part of this project, in order to collect a satisfying number of measurements. Saptharishi (2016) presented a detailed overview of rheology fundamentals, which will not be repeated in this report. If the reader finds a need for reviewing rheology basics, Saptharishi's (2016) thesis can be used as supportive information.

1.4 Project scope and Tasks

1. Literature review of rheology, in particular dependency on time and temperature. Consider references in Saptharishi's thesis.

2. Develop test matrix and a selection of fluids. (Selection of fluids and test matrix were altered after some initial findings, which will be explained in Chapter 3.) Propose statistical methods suited to determine uncertainty.
3. Run experiments.
4. Analysis with statistics, compare to Saptharishi's results, and results from automated tests in Bergen.

1.5 Project Hypothesis

Measurements of different character will always be related to their reliability. With a constant evolvement in technological applications – more detailed information is getting available – and requisitions for accuracy and precision are getting stricter. Saptharishi's (2016) conducted experiments at the IPT lab was of limited number, which made it reasonable to question the reliability. The work in this project involves an increased number of tests, in order to verify whether the results can be reliable. By creating a confidence interval for an increased number of tests, previous results can be compared to such an interval, and thereby assessed a quantity for reliability. For additionally experiments performed during this work, which is not comparable with others, reliability can be assessed by the use of standard deviation and proportions of variation from the obtained mean values.

It is expected that increasing temperatures will lower the viscosity, as Saptharishi observed. Regarding time dependency, it was indicated in her work that time elevated the rheological properties, based on a difference in results between manual and automated tests which could be explained by this. When expanding the measurements to a wide time interval, such behaviour should be confirmed or rejected during these experiments. As Saptharishi did perform measurements once for each mixture, it is likely that these would differ in magnitude compared with a more numerous set of tests. To be characterized as reliable, the results should be found within a reasonable interval obtained by the new experiments. Size of such an interval will depend on values collected in this project. Gel strength was observed only in samples containing Xanthan Gum, and was showing decreasing tendency. It is expected that a further study could contradict this, as gelling is expected to increase in magnitude with time.

1.6 Report Structure

Chapter 1 is outlining the background for this project, along with problem description, objectives and structure.

In Chapter 2, a brief theoretical background is presented, related to the main topics which will be treated during this report.

Chapter 3 contains description of method. Information of instrumentation and procedures related to the practical laboratory work are presented. A brief outline of statistical methods are also included.

Chapter 4 is the main part where the results are presented. Statistical analysis and comparison with results from Saptharishi (2016) will be found here. Time and temperature influence of rheological properties are the main parameters.

Chapter 5 summarizes results and contain discussion of these.

Chapter 6 contain conclusions drawn from the performed work.

Chapter 7 deals with potential improvements and suggestion for a way forward.

Some additional information, graphical extras and complete tabulation of measurements are found in appendices.

2 Background Theory

2.1 Drilling Fluid applications

During the process of drilling a well, drilling fluid has two basic functions; the fluid column has to overcome the pore pressure – in order to prevent formations fluids from entering the surface – and cuttings need to be removed from the wellbore (Skalle, 2014). There is a wide range of actual conditions in different wellbores which may desire different designs for drilling fluid to fulfil its task. Two main properties are related to the basic functions: viscosity and density. Special additives are used to influence these properties as desired. Weighting agents increases the density of fluid systems, where barite is most common. As viscosifiers, polymers and clay are the main types (Ramsey, 2017).

2.2 Polymer fundamentals

Polymers are organic molecules built from monomers, and can be built into long chains. The molecules can be of linear shape or crosslinked – when linear chains connect to each other through cross linking agents. Polymers can be divided into three main groups based on their ionic character: non-ionic, anionic and cationic (American Association of Drilling Engineers, 1999). They are respectively with no charge, negative net charge and positively charged. As mentioned previously, polymers are applied as viscosifier, but also in order to keep control of fluid loss, encapsulation of the wellbore, stabilizer in high temperature conditions and as flocculants or deflocculants (Azar & Samuel, 2007). While oil and water behaves like a Newtonian fluid – where shear stress is proportional to shear rate – polymeric solutions tend to be better fitted into the Power-law or Herschel Bulkley model (Schlumberger, 2016). The appearance of the polymer will depend on its charge and on the salinity of the solution. An-ionic molecules are stretched by repulsive forces, and these forces are lowered in saline solutions (Skalle, 2014). This is illustrated in Figure 1: Appearance of an-ionic polymer molecule affected by salinity. A compressed shape will have a smaller interacting surface thus creating lower viscosity than a stretched form.

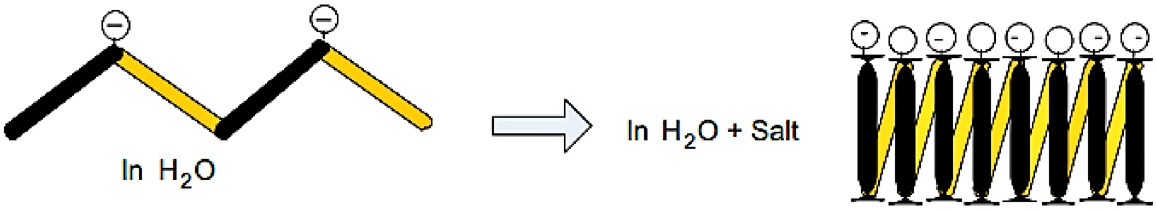

Figure 1: Appearance of an-ionic polymer molecule affected by salinity (Skalle, 2014)

2.3 Polymer additives

Two polymer additives are used in the experiments, poly-anionic cellulose (PAC) and Xanthan Gum (XG). Their properties are presented below.

2.3.1 Poly-anionic Cellulose (PAC)

PAC (Figure 2: Structural formula for PAC is a water-soluble anionic polymer of sodium salt, similar to carboxymethyl cellulose (CMC), but with a certain level of substitution degree (Sidley Chemical, 2017). PAC is produced under rigid conditions in order to create this uniformity, and thus classified as partly synthetic. Solubility is given with a bit varying numbers, but it ranges from 2.5% to 5%. Solutions of 4% PAC is stated to have a viscosity in between 50-200 cp at 25° C. The effect of temperature will influence the viscosity temporarily, but under normal conditions these effects are reversible (Sigma-Aldrich, 2017). Several manufacturers claim that heating over time will degrade the product and permanently reduce viscosity.

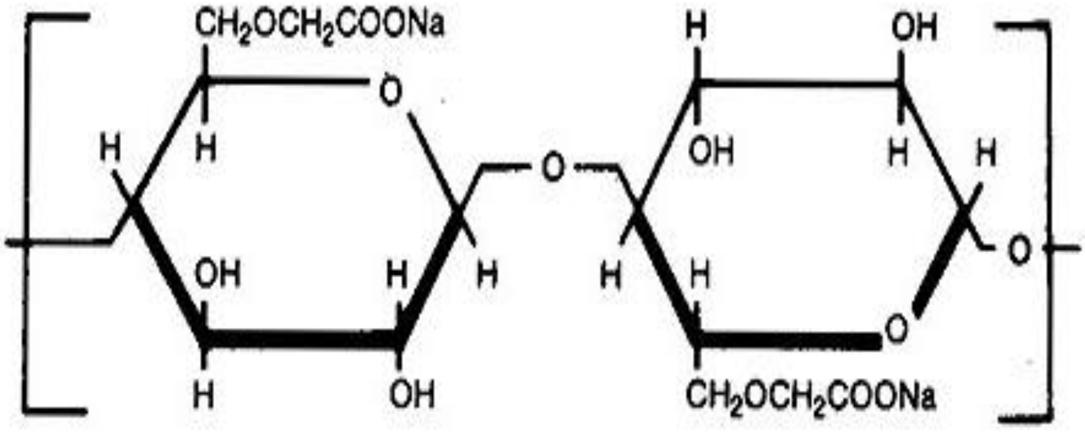

Figure 2: Structural formula for PAC (Sidley Chemical, 2017)

PAC is mainly used as a viscosifier and for filtration control, but also has additional benefits. These includes i.e. fluid loss control, thin mud cakes, low solids-shear thinning fluid, borehole stability and it is also environmental acceptable. The latter is due to its properties of being naturally biodegradable (Hercules, Aqualon Division, 2017).

2.3.2 Xanthan Gum (XG)

Xanthan Gum (XG) is a natural polymer generated by a bacteria called Xhanthomas Campestrius (Force Chem Technologies, 2016). It is widely used due to its ability to suspend solids under low shear conditions. Shear thinning effect minimizes pump friction pressure loss and increases ROP. It is a high-molecular weight polysaccharide consisting of three different monosaccharides; mannose, glucose, glucuronic acid. The polar side chains provides for extensive hydrogen bonding with water and interchain reactions giving a helical structure. It is biodegradable and environmental friendly, with high tolerance to temperature, changes in pH and mineral contaminants (Prince Energy, 2016). Cross linking between chains maintains the suspending ability under static conditions.

2.5 Polymer dispersion

When preparing polymer solution, addition of powder has to be precise in order to avoid creation of agglomerates. The particles first have to be dispersed in water, and should be individually wet before they dissolve. When the solution is ready, it should be clear and transparent (This also depends on the concentration). Mud hoppers are most commonly used for dispersion of chemicals (ASME Shale Shaker Committee, 2011), as illustrated in Figure 3: Mud hopper schematic. The Venturi effect – which is shown in Figure 3: Mud hopper schematic – is an effect where fluid pressure decrease as the fluid flows through a section, which is constrained and increases the fluid velocity. The differential pressure causes a drag on the particles to be dispersed and they get evenly distributed into the flow.

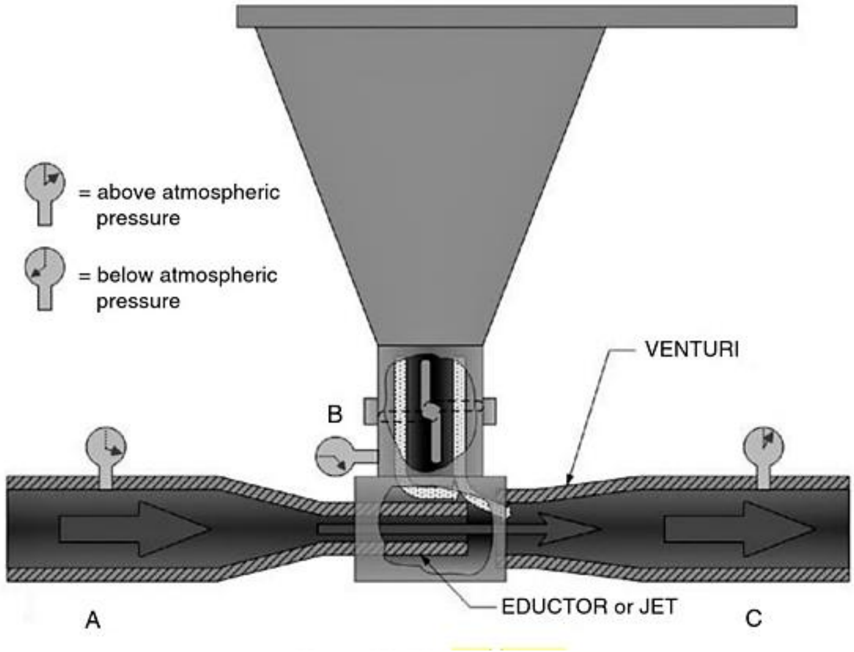

Figure 3: Mud hopper schematic

Degree of disaggregation of polymers dispersed in a solvent depends mainly of the following three factors: shear exerted on the solution, solvation power and the chemical composition of the polymer (Aqualon, 2010). Different states of disaggregation is illustrated in Figure 4: Degree of disaggregation vs viscosity.

- 1 is a state where the particles remains as powder without being dissolved.
- 2 is a state where the polymers are swollen to maximum without being completely dissolved.
- 3 represents the maximum degree of disaggregation, which has a stable viscosity profile.

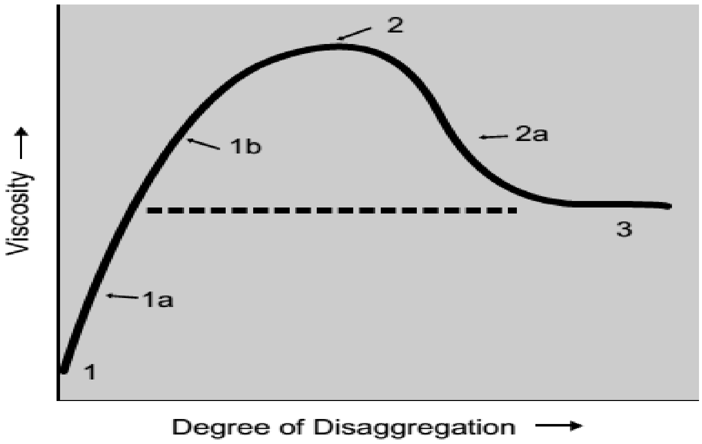

Figure 4: Degree of disaggregation vs viscosity

In correlation with preparation and testing of drilling fluids, a process of high temperate aging is often performed (SPE International, 2017). A sample of the fluid is set to static or dynamic conditions over a time interval of 16 hours or more, with a pre-set high temperature (HT) to simulate real well conditions. The process is conducted to assess conditions for what a fluid might be subject to, in order to measure the properties after such impact. These subsequent tests are typically of the same nature as for non-aged samples.

2.6 Temperature influence

On a general basis, temperature changes always affects viscosity, it is only a matter of how much the material is affected (Mezger, 2011). The relationship is inversely proportional, but the relative decreased viscosity with increased temperature differs among substances. As energy get supplied to a system, the molecular energy increases, resulting in farther distance between molecules and giving lower viscosity. John Lennard-Jones proposed such a model in 1924 (Laboratory of Atomistic and Molecular Mechanics, 2016). The mathematical correlation will not be presented here, but the corresponding graphical explanation is illustrated in Figure 5: Intermolecular Lennard-Jones potential with r being the intermolecular distance. Take the lower dashed line as initial low temperature with corresponding intermolecular distance r_1. Consider the graph as an energy well. When temperature is increased, energy is transmitted to the molecules, and the level rises to the upper dashed line. The slopes on left and right side of r_0 is respectively repulsive and attractive energy. An uneven correlation of these intermolecular forces results in increased equilibrium distance r_2, proportional to temperature. This is simply referred to as thermal expansion.

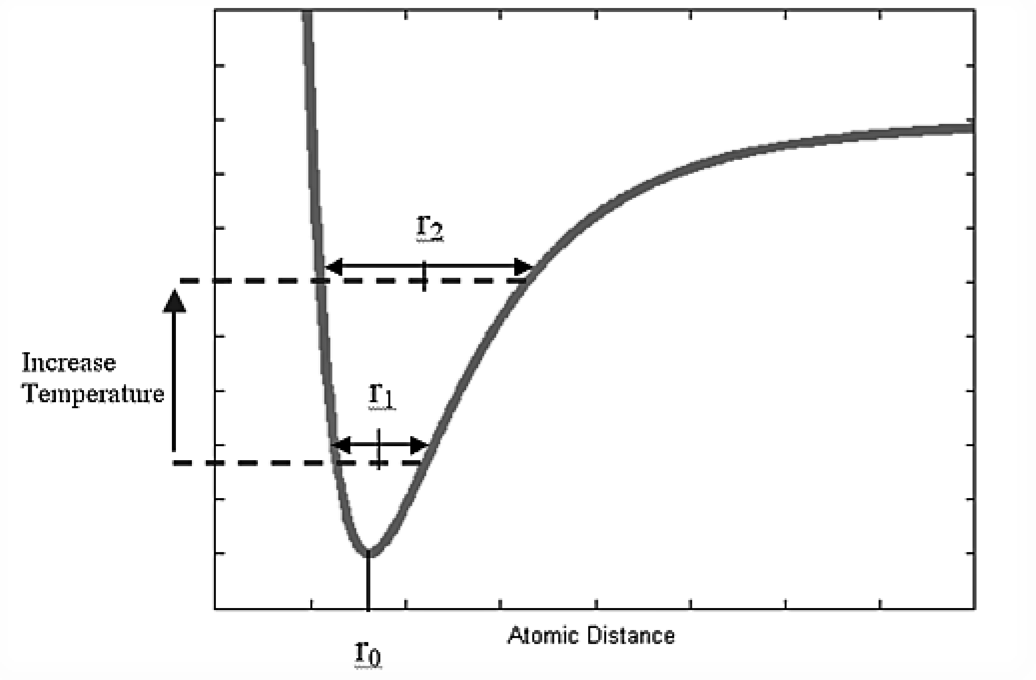

Figure 5: Intermolecular Lennard-Jones potential (Laboratory of Atomistic and Molecular Mechanics, 2016)

When it comes to more specific correlations for temperature – viscosity in polymeric solutions there is a great number of different models proposed. The comprehensive nature of this will not be explained in this report. One equation which have been extensively used is the Arrhenius equation (Gupta, 2000), which is seen below:

$$\eta = A * e^{\left(\frac{Ea}{RT}\right)} \qquad \textit{Equation 1}$$

The viscosity η is expressed in terms of a constant A, activation energy Ea, absolute temperature T and the universal molar gas constant R. The activation energy describes the difficulty of moving a chain from one position to another (Budtova & Navard, 2015). This is a particularly complex value as it depends on amongst others, molar mass, molecular structure of the polymer and the interchain reaction intensity.

As briefly mentioned in the previous section, HT testing is performed in order to simulate well conditions. Information given by a variety of manufacturers states that temperature influence to a certain level is a reversible process. If exposed to higher temperatures than i.e. 80° C over long periods of time, it will permanently reduce the viscosity (Aqualon, 2010). Given this information, it is important that the levels at where such permanently damage find place are identified, as well as knowledge about what temperatures to be expected in the wellbore.

2.7 Time dependency

Thixotropic behaviour is a time dependant behaviour which was observed for mixtures involved in this project (Saptharishi, 2016). With increasing shear over time, the reversible process results in lower shear stress than in the build-up phase. As this was discussed detailed by Saptharishi (2016), it is briefly mentioned here.

Temperature is of course important over time, as chemical reactions proceed in higher rate with increased energy to the system. For simplicity, imagine temperature is being kept constant, and other mechanisms of influence are being investigated. Micro-organisms uses organic matter as nutrient, this matter can then be seen as bio-degradable. During such a process, complex molecules are broken into simpler ones, and in respect to the main topic, reduces viscosity. The rate of which these organisms consume organic matter are dependant of water, light and oxygen (Science Learning Hub, 2017). A detailed review of the driving mechanisms is a biological topic which will not be examined in further detail here.

2.8 Statistical tools

To be able to run a statistical analysis, a statistical toolbox has to be applied. During this section, some definitions and equations will be presented. Statistical theory have a mathematical basis, of which is a probability distribution of some random variable (Mandel, 1964). This distribution represents an image of the frequencies for all possible outcomes of a quantity of interest – in this work – viscosity measurements for mud samples. The normal probability distribution is the most common, and is illustrated in Figure 6.

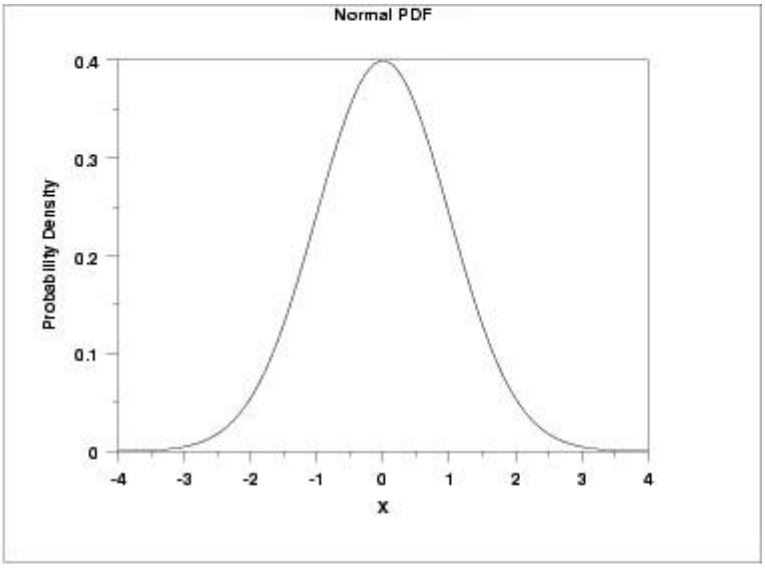

Figure 6: The normal probability distribution function (NIST/SEMATECH, 2017)

To be able to state with full validity that a material has a certain property, it would be necessary to measure every existent batch of this material, without any error regarding precision and accuracy of the systematic method involved. Samples of materials are used to create an image of a certain behaviour, with more or less probability for it being close to the true behaviour. The former of these would of course be preferable. A number of parameters are used to describe properties related to the fluctuations of sample measurements. The mean value is a description of where we expect a value to be located, based on a set of values obtained from an experiment. It is defined by Equation 2.

Estimate of the arithmetic mean value, derived from *N* measurements of value *x*:

$$E(x) = \bar{x} = \frac{x_1 + x_2 + \cdots + x_N}{N} = \frac{\sum_i x_i}{N}$$ *Equation 2*

The variance, Equation 3, is a parameter describing how the values are spread around the mean value. This is an average value of the square distance from the mean value, meaning that small variance have a dispersion more closely to the middle, than as of a larger variance. The use of hat, indicates an estimate of the true value.

Variance, by the estimate s^2:

$$Var(x) = E[x - E(x)]^2 = \hat{\sigma}^2 = s^2 = \frac{\sum_i (x_i - \bar{x})^2}{N-1} \qquad \textit{Equation 3}$$

As the variance is a squared unit, it is more practical to have a parameter containing the same unit as the measured quantity for description of the average fluctuations. The standard deviation is such, and is simply the square root of the variance, seen in Equation 4.

$$\hat{\sigma} = \sqrt{Var(x)} = s = \sqrt{s^2} = \sqrt{\frac{\sum_i (x_i - \mu)^2}{N-1}} \qquad \textit{Equation 4}$$

A term of particular interest is the confidence interval. When taking measurements of a sample, estimates for the true value are developed, without knowing what the true value is. In a limited interval, it may or may not be possible that the true value is located. From a set of sample-data, it is possible to calculate an interval which contains the true value with a certain level of possibility, or confidence. The formula for the confidence interval is given below, in Equation 5.

$$\bar{x} - \frac{t * s}{\sqrt{N}} < \mu^* < \bar{x} + \frac{t * s}{\sqrt{N}} \qquad \textit{Equation 5}$$

In Equation 5 μ^* represents the true value, which is unknown, and t represents the critical value of the t-distribution (Figure 7). For data not fitting into the confidence interval, the deviation from the mean value can be divided by the mean, in order to directly asses the partitioned error. This may be suitable for addressing errors when sample numbers are limited.

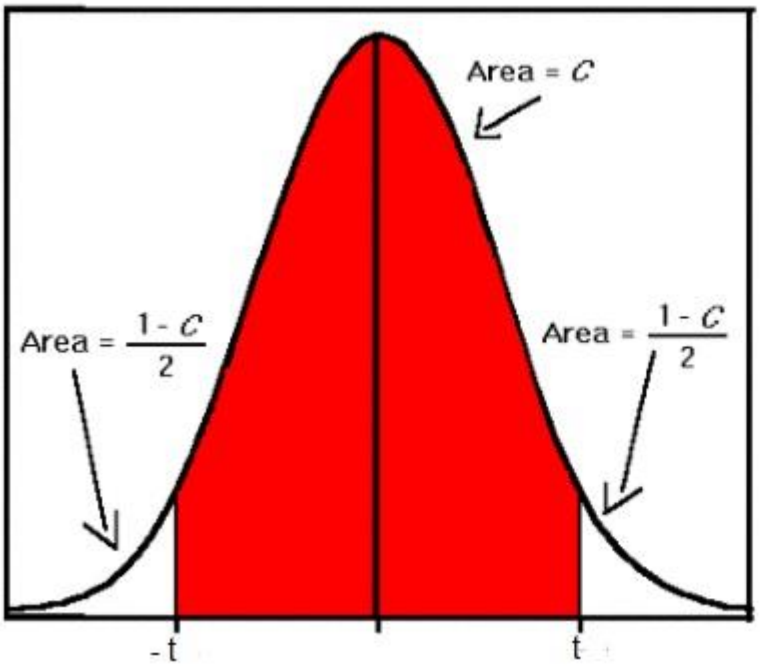

Figure 7: the probability t-distribution (Mackowiak, 1992)

Standard deviation is the parameter for expressing dispersion of samples, compared to the true value of the quantity. Often the true value and the standard deviation is not known. It could still be desirable to express the dispersion of samples, compared to the obtained mean from measurements, as a way to define the precision of the measurement method itself. The standard error (Equation 6) is an expression for such, and is dependent of the number of measurements N. When number of measurements goes to infinity, the standard error goes to zero, because the measurements will then get closer to a true representation.

$$standard\ error = \frac{s}{\sqrt{N}} \qquad \text{Equation 6}$$

The above equation represents a measure for method precision, which quantifies how repeatable a method of interest is. Another parameter is also needed to evaluate the quality of such. The systematic error gives information regarding the mean of the sample measurements compared to the true value, here called a reference value denoted by R^*. The systematic error (Equation 7) is simply:

$$systematic\ error = \bar{x} - R^* \qquad \text{Equation 7}$$

3 Laboratory Work

The laboratory work has been carried out at the Mud Engineering Lab at the Institute of Petroleum Technology (IPT), on the NTNU campus Lerkendal-Valgrinda. Materials have been provided by Statoil, through my supervisors John-Morten Godhavn and Aminul Islam.

The objectives of the lab work were mainly divided into the following two agendas. First, to investigate the repeatability and reliability related to manual experiments in Priyadharshini Saptharishi's Master's thesis. Thereby, a further look at time dependency of viscosity measurements, and also briefly at temperature effects on viscosity behaviour over time. The mixtures which were tested mainly consists of poly-anionic cellulose (PAC), but also some samples including Xanthan Gum (XG) and barite.

As reliability of the experiments was to be investigated, strict routines related to experimental procedures have been on top of the agenda. The preface in the lab contained some experimental variations to get familiar with the equipment available, and for selecting the best suited setup. A few changes were made compared to Saptharishi's procedure, due to some conditions set in correlation with supervisors. This concerns the mixing RPM and resting time of samples will be explained more detailed in the following subsections, where the equipment and procedures involved are outlined.

3.1 Equipment

Instruments and equipment used in these experiments are quite simple, but robust. Both the mud balance and the Fann 35 viscometer are widely used in the industry, and are found in both laboratories and at on-site installations, according to IPT lab-technician Roger Overå.

3.1.1 Weights

The weighing of materials included in the different mixtures is of essential importance, as this is the first step for making the preparation procedure repeatable within an accurate basis. When using precise balances, they have to be level- adjusted correctly, and should be placed at a location which is not affected by air streams, i.e. beneath air conditioning system or windows, due to temperature and air pressure influence. There have been used two different weights in the preparation of samples. The first is a Sartorius CP 4202 S, with a readability of 10 mg, which has been used for weighing additives. Weighing chemicals with a two-decimal reading for grams makes it accurate enough to ignore possible errors within amount of additives. Sartorius Combics 1 has also been used to weigh water.

3.1.2 Agitators

A proper mixer is required to form a homogenous fluid when dissolving or dispersing a chemical additive in water or other base fluid. Shearing time and speed may vary with the components involved, as volume capacity also has to be considered.

During Priyadharshini Saptharishi’s work, the two-speed Hamilton beach laboratory mixer and a two-speed laboratory blender from Waring were used. These did both excess 500 RPM at minimum speed. When using any of these, there were observed a lot of air bubbles, and samples needed to rest up till 24 hours for trapped air-bubbles to be released (Saptharishi, 2016). To be able to make good quality measurements of samples during the 24 first hours after mixing, an alternative mixer had to be used. A condition for maximum mixing speed was set to 500 RPM.

In the IPT lab, a mixer fulfilling this criteria was the Janke & Kunkel RW 20 DZM overhead, delivered by IKA-Labortechnik. This mixer has step-less speed options ranging from 0-480 RPM, which is set manually. The mixing blade used was a centrifugal stirrer. When adding XG to the PAC-mixtures, the viscosity increased radically, and in order to get the whole sample sheared, a magnetic mixing plate at the bottom was added. This could probably have been prevented by using a spiral stirrer of appropriate size, but such a stirrer was not available. The magnetic mixing plate was a MR Hei-End from Heidolph. This also comes with step-less speed options, ranging from 30-1400 RPM, which appropriately matches the overhead mixer. The setup for the mixing procedure is shown in Figure 8: Mixing setup with overhead mixer and magnetic plate

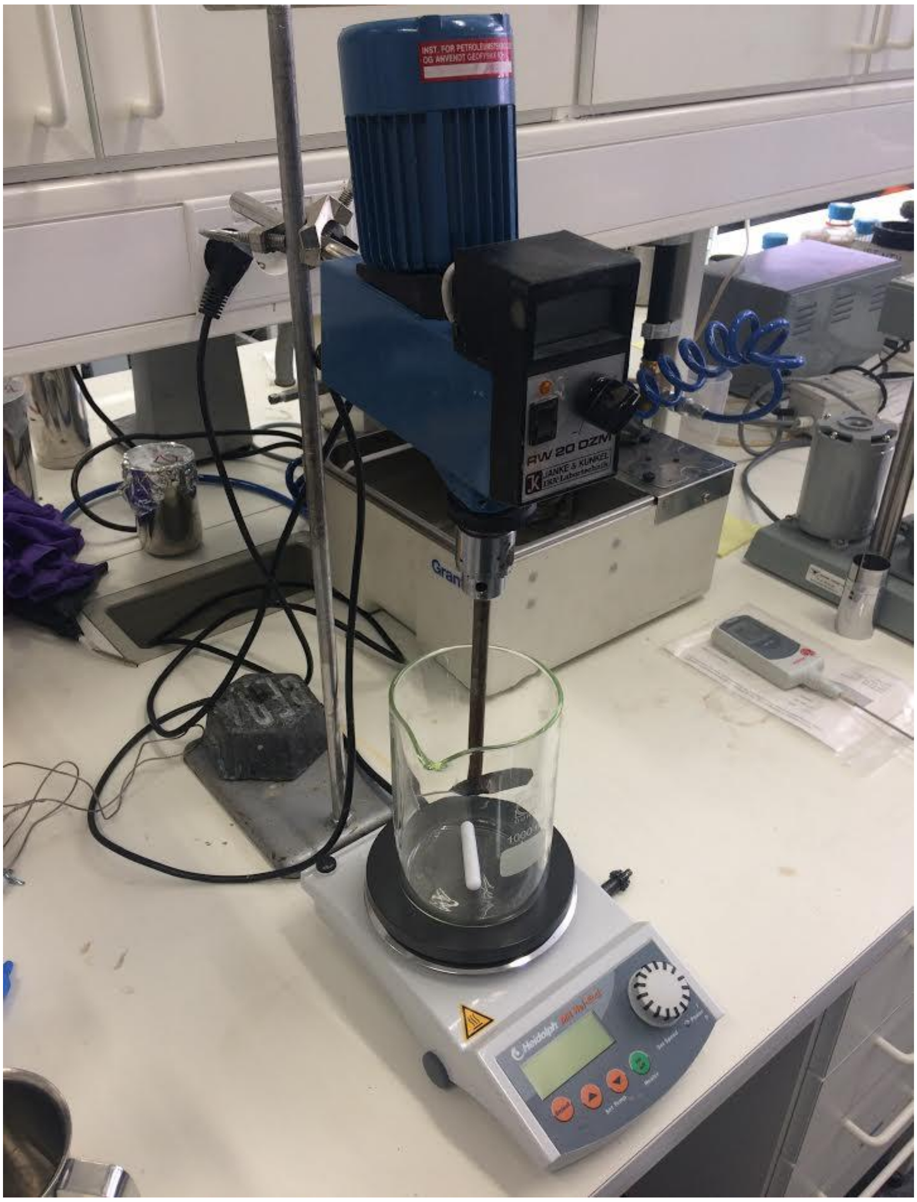

Figure 8: Mixing setup with overhead mixer and magnetic plate

3.1.3 Fann 35 viscometer.

The Fann 35 viscometer (Figure 9) is an instrument for measuring viscosity and gel strength. Fluid fills the annulus between a bob and a rotational cylinder. When the cylinder rotates, the fluid applies a viscous drag onto the bob, and is converted to shear force trough a precision spring. The deflection of the spring gives the shear stress. Instruments measuring shear force at a given shear speed are called Couette viscometers (Fann Instrument Company, 2016). Because viscosity can be read directly from the dial reading, it is also called a direct-indicating viscometer (Schlumberger, 2016).

There are two types of the Fann viscometer; a 6 speed and a 12 speed version. The 6 speed variant is used in this work, and has RPM options of 3, 6, 100, 200, 300 and 600. The ability of measuring shear force at multiple speeds makes it possible to calculate various coefficients for rheological models. There are several options of bobs, rotor sleeves and torsion springs. When the combination of these are standard; R1 rotor sleeve, B1 bob and F1 spring, plastic viscosity (PV) and yield point (YP) for the Bingham plastic fluid model can be calculated very easily. PV (cp) = dial 600 – dial 300, and YP (lb/100 ft^2) = dial 300- PV. Such easy calculations are quick, and offer a great application for interpretation of viscometer results in the field. Gel strength readings are also directly taken from the dial readings, with the unit lb/100 ft^2. Additional explanations of calculations connected to the Fann viscometer are found in section A.1 Fann 35 Viscometer calculations in appendices.

The Fann 35 viscometer was used to obtain viscosity and gel strength for the samples involved in this thesis, and measurements were performed due to the American Petroleum Institute (API) Recommended Practise for Field Testing Water Based Drilling Fluids, API RP 13B-2/ISO 10414-2 Specification.

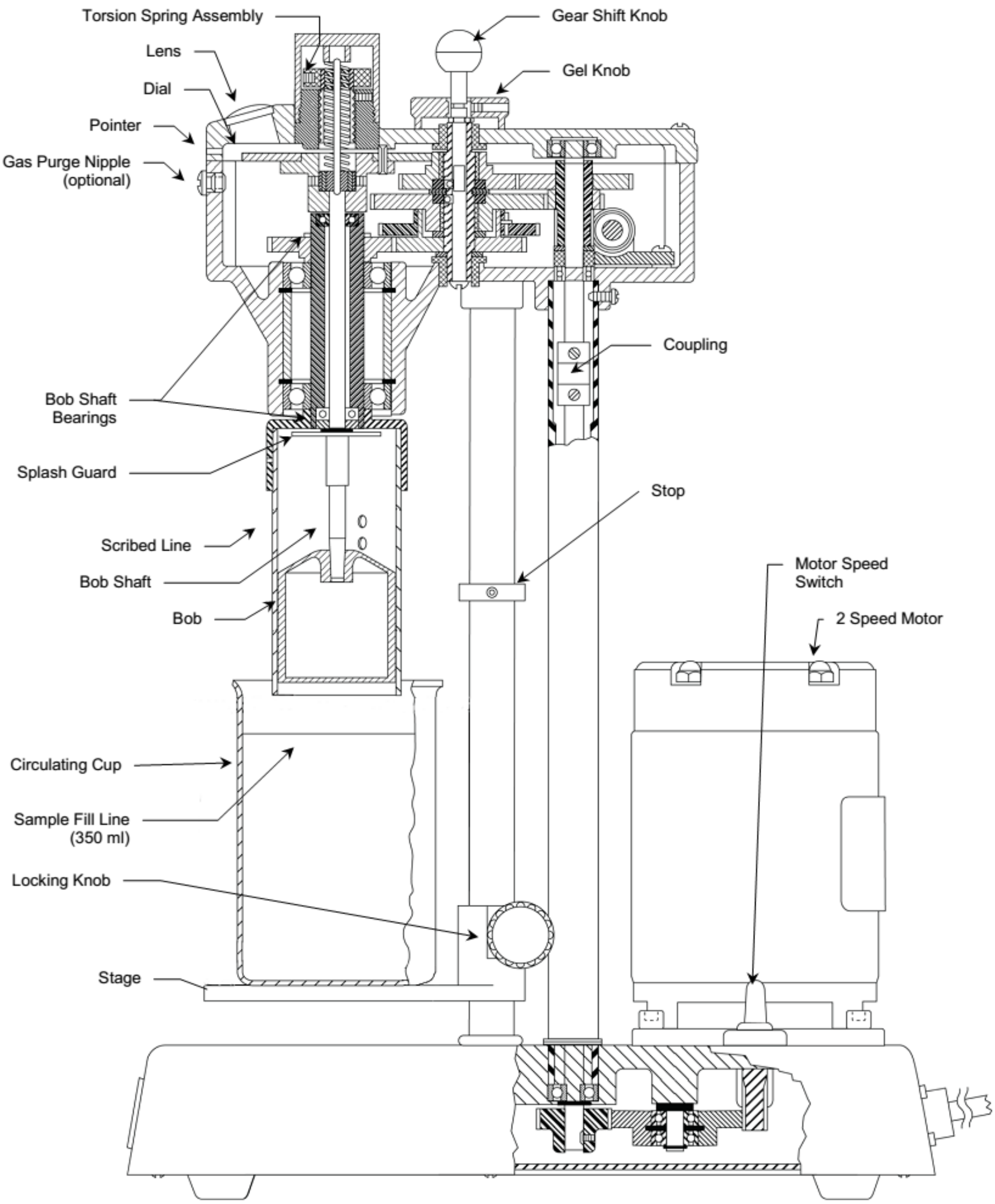

Figure 9: Fann 35 viscometer (Fann Instrument Company, 2016)

3.1.4 Mud Balance

A device for measuring density is the mud balance (Figure 10). Its simplicity makes it reliable in the field, and readings are not materially affected by the temperature of the sample (Fann Instrument Company, 2016). It is constructed by a sample cup with a lid in one end of a beam which is graduated and have a counterweight at the other end. A bubble is indicating whether the beam is level or not. The measuring range is from 0.72 -2.88 gm/cm^3. It is a small hole at the top of the lid to make sure gas bubbles can escape, but gas bubbles trapped within the sample may cause distortion to the accuracy. As long as the unpressurized version of the mud balance is used, it is important for the accuracy that the sample contain as little entrapped gas as possible (Fann Instrument Company, 2016). The unpressurized mud balance is used in this work. When it comes to precision, the accuracy of mud weight should lie in the range of +-0.01 gm/cm^3, or 0.1 lbm/gal (Schlumberger, 2016).

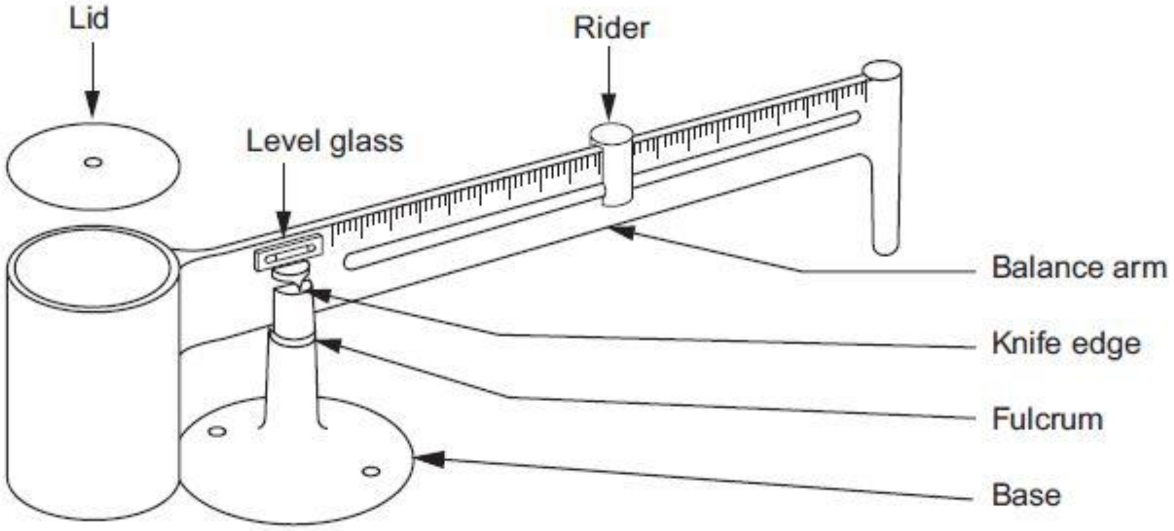

Figure 10: Mud balance (Petroleum Support, 2016).

3.1.5 Additional laboratory equipment

In addition to the essential instruments mentioned in previous subsections, multiple laboratory tools have been involved. These are blender jars, stirring spoons, storage jars, heating cups and a separating funnel.

3.2 Test setup

As one objective was a closer study of previous experiments (Saptharishi, 2016), it was necessary to include parts of the same test matrix to obtain comparative results. Time and temperature influence on viscosity was also to be investigated, so measurements over a wide time interval was added together with a few variations in temperature.

3.2.1 Initial matrix

Initially, the idea was to use 4% PAC as a base, and thereby adding XG and barite. After a few trials, it was clear that such a composition would be too viscous when adding XG, so that a new setup had to be made. The first test matrix is shown in and **Error! Reference source not found.**. They are divided into two parts to make them fit, and still be readable.

Table 1: Initial test matrix, part 1, viscosity

Test parameters			density	Fann viscometer RPM					
Mixture	Temp(°C)	Rest time(hrs)	g/cm3	3	6	100	200	300	600
4% PAC	5	0							
4% PAC	25	0							
4% PAC	50	0							
4% PAC	25	24							
4% PAC	25	48							
4% PAC	25	6 days							
4%PAC+2%XG	5	0							
4%PAC+2%XG	25	0							
4%PAC+2%XG	50	0							
4%PAC+2%XG	25	24							
4%PAC+2%XG	25	48							
4%PAC+2%XG	25	6 days							
4%PAC+2%XG+barite	5	0							
4%PAC+2%XG+barite	25	0							
4%PAC+2%XG+barite	50	0							
4%PAC+2%XG+barite	25	24							
4%PAC+2%XG+barite	25	48							
4%PAC+2%XG+barite	25	6 days							

Table 2: Initial test matrix, part 2, gel strength

Test parameters			Gel Strength		
Mixture	Temp(°C)	Rest time(hrs)	10sec	1min	10min
4% PAC	5	0			
4% PAC	25	0			
4% PAC	50	0			
4% PAC	25	24			
4% PAC	25	48			
4% PAC	25	6 days			
4%PAC+2%XG	5	0			
4%PAC+2%XG	25	0			
4%PAC+2%XG	50	0			
4%PAC+2%XG	25	24			
4%PAC+2%XG	25	48			
4%PAC+2%XG	25	6 days			
4%PAC+2%XG+barite	5	0			
4%PAC+2%XG+barite	25	0			
4%PAC+2%XG+barite	50	0			
4%PAC+2%XG+barite	25	24			
4%PAC+2%XG+barite	25	48			
4%PAC+2%XG+barite	25	6 days			

3.2.2 Final matrix

After reviewing results from initial test setup, which will be presented in chapter 4, the test matrix was modified as explained previously. Mixture concentrations were altered, and as the viscosity seemed to change over time, it was also decided to increase the frequency of time testing. Pure PAC with water in 4% and 2% concentrations was decided, and 2% PAC to be mixed with 1% and 2% XG. Barite was mixed together with 4% PAC. Varying temperature conditions was excluded for the new matrix, as it would break the time schedule for getting through the new matrix. The test matrix was completed several times, in order to have material for a statistical analysis. and show the final test matrix. Both tables have been shortened related to the four lower concentrations time dependency tests, but can be found together with full tabulation of results in Appendix C Complete .

Table 3: Final test matrix, part1. Rest times for the four lower mixtures are shortened in the table, but the same as for 2% PAC

Temp 25 °C			Fann viscometer RPM					
Mixture	Sample #	Rest time(hrs)	3	6	100	200	300	600
2% PAC		0						
2% PAC		1						
2% PAC		2						
2% PAC		10						
2% PAC		24						
2% PAC		48						
2% PAC		72						
2% PAC		6days						
2% PAC		10 days						
2% PAC		14days						
4% PAC		0-14days						
2%PAC + 1%XG		0-14days						
2%PAC + 2%XG		0-14days						
4%PAC + 20%barite		0-14days						

Table 4: Final test matrix, part 2. Rest times for the four lower mixtures are the same as for 2% PAC

Temp 25 °C			Gel Strength		
Mixture	Sample #	Rest time(hrs)	10sec	1min	10min
2% PAC		0			
2% PAC		1			
2% PAC		2			
2% PAC		10			
2% PAC		24			
2% PAC		48			
2% PAC		72			
2% PAC		6days			
2% PAC		10 days			
2% PAC		14days			
4% PAC		0-14days			
2%PAC + 1%XG		0-14days			
2%PAC + 2%XG		0-14days			
4%PAC + 20%barite		0-14days			

3.3 Routines and Contamination

In order to make the tests repeatable and reliable, it is of great importance that laboratory equipment are clean, and that procedures are the same. At the IPT lab, there are a lot of people working with different chemicals, using the same equipment. If instruments and blender jars are not perfectly cleaned after use, there is a big risk that particles from previously mixtures involved will be transferred to the current fluid in work. For experiments running over time, it is even more severe if contamination occurs, as chemical reactions could evolve, and influence the fluid properties (Smith, 2000). Because of this, it is not only important to clean properly after own use, but also to check equipment before using it. Every time finishing a procedure, the first priority should be to clean every tool with proper solvents. Good routines could prevent inadvertent addition of unwanted particles into the samples. In addition an orderly workplace saves time.

3.4 Sample Mixing

This subsection will explain how the different samples were prepared for testing. A few different procedures were conducted, in respect to type of mixer, mixing time, and mixing temperature. This was in order to get a brief outline of how these parameters seemed to affect the quality of the sample, before committing to a final setup.

Pre-calculations of amount of additive were done in regard to the outlined weight percentages that were chosen. Appropriate sized glass jars were used when weighing material on the laboratory scale; respectively spring water and the chemical substances. Because the Hamilton Beach mixer and the Waring blender both did produce a lot of entrapped air bubbles – as observed by Saptharishi (2016) – they were seen unfit for the procedure, as the Fann tests during the first 24 hours would risk being affected by the large amount of bubbles. The overhead mixer did not have the same problem, as it could manage lower speed, and was used at 480 RPM. This was chosen to be the best fit. The blender jars fit for this mixer were of sizes 500-1000ml. A disadvantage of using this agitator was a longer mixing time to get homogenous results. 2-3 hours appeared to be the ideal time for this type, due to the transparency of the mixture. As it was only possible to stir one sample at a time, it had to be shortened to 1 hour to be able to get the experiments done within the time-frame for this lab work, looking at the amount and time frequency for the test matrix. After 1 hour, a homogenous mixture was seen, but a slightly less transparent colour, indicating a lower degree of dissolution. Temperature were monitored with a thermometer during the stirring process, and seemed to be stable, within +-0.5 degrees C. This was clearly an advantage, compared to the Hamilton mixer and Waring blender, which produced more heat.

A sieve was first used to get the PAC evenly distributed into the water, but as the particle size distribution was not evenly enough for this, it had to be done manually by pouring. This had to be done very carefully and accurate, to avoid creation of big lumps which would take extra time to dissolve. This process were done over respectively 3 and 6 minutes for the 500ml and 1000ml blender jars. Addition of XG and barite could be done with a sieve, as the particle sizes was more evenly distributed.

The temperature was raised to 50 degrees C at some preface experiments, in order to get the mixing time done in less time. As the samples had to be cooled down after mixing, it turned out not to be time efficient, though it did reveal some interesting results, which will be presented during chapter 4. The standard for the final matrix was set to 25 degrees C. This was also found to be necessary to compare with Saptharishi’s results. Samples were stored in air sealed laboratory storage jars in between tests.

3.5 Experimental Procedure

A brief stepwise description of the experimental execution will follow below. To make the results comparable, this had to be done in accordance to Saptharishi´s (2016) procedures.

- The Fann 35 viscometer was calibrated at the beginning, with a commercial calibration fluid of known viscosity. This was not necessary to repeat for each experiment, following the advice of the head technician of the IPT lab, Roger Overå. There were two different Fann viscometers in use, and both were checked using the calibration fluid in accordance with the official Fann calibration procedure.
- The mud balance was checked before use, using spring water.
- Samples were prepared as described in the previous section. It should be emphasized that samples in these experiments did not rest 24 hours before the first test, as they did in Saptharishi’s work. Part of the experiments was to look at the behaviour during the first 24 hours.
- Samples which had been stored in between tests, were re-stirred for 2 minutes manually, and for 2 minutes at 300 RPM in the Fann viscometer.
- The measurements in the Fann viscometer were taken gradually from 600 RPM to 3 RPM.
- Gel strength readings were conducted after initial shear rate readings. Here, 10sec, 1 minute and 10 minute test were completed, with 2 minutes shearing at 600 RPM in between, also in accordance with previous routine (Saptharishi, 2016). The peak value when shearing at 3 RPM after rest times is the value of interest.
- Density was recorded using mud balance after shear rate and gel strength tests, using the mud balance.
- Samples were stored in labelled air sealed laboratory storage jars in between tests.

3.6 Comparative Samples

To be able to compare results obtained in this work with Saptharishi’s results, compatibility in regard to resting time of samples and test temperatures were chosen as main parameters.

Saptharishi had a resting time of minimum 24 hours and maximum 48 hours for samples prepared in the IPT lab. That is a bit vague, but it is chosen to use measured values after 24 hours of rest in experiments performed during this work, as comparable basis. Regarding to tests performed in a laboratory in Bergen, the mixing procedure was described to take about 10 hours. The tests taken from the flow loop at this place, was conducted after about 30 minutes and 1 hour 40 minutes. As comparable basis, the 12 hours measurements in this work will be used. There is of course a quantity of uncertainty which is difficult to define involved in such assumptions.

3.7 Statistical approach

The intention for doing these viscosity tests, was to use the measurements for describing the rheological properties of the mud types of interest. The experimental procedure accounted for previously in 3.5 was applied, -to measure viscosity as a property of the fluid compositions described in section 3.2 Test setup, to characterize rheological profiles for the mixtures. Errors can occur, both experimental and from mistakes made. It is important to stick to the procedure, so that the mistakes can be minimized. During this work, the most important parameters to monitor for sticking to a strict reliable procedure, are the mixture temperatures, the test times and the sample preparations. The more detailed variations in the surroundings is less possible to control. Natural variables lies in the polymer chain lengths, small temperature differences within the samples, homogeneity of the samples and the eye that read the measured values. The objective with the statistical analysis, is to establish what limitations lies within, and the validity in, a selection of the results made by Saptharishi, and the results which is presented in the next chapter.

3.7.1 Statistical Methods

In order to produce reliable results, it would be optimal to have a preface in the laboratory, for which sample sizes for desired level of certainty could be decided, based on an initial trial of small samples (Ryan, 2013). Results obtained by Saptharishi (2016) contained single-valued results for each mixture type, which made it insufficient in determination of such sample sizes. Results from measurements during this work, will be used to induce reliability, and also to consider whether the sampling sizes has been sufficient. In order to express something about the reliability for a selection of Saptharishi's (2016) results, they will be compared to results presented in the next chapter by the use of arithmetic averages, variance, standard deviations and confidence intervals. One agenda is to propose what sample proportions are necessary for the manual rheological Fann experiments, in order to be confident that they are reliable. An initial assessment of precision and accuracy of the measurement method (Fann viscometer rheological tests) will also be performed before presenting the main results.

3.7.2 Establishing reliability

To assess the reliability of manual results obtained in the background project for this work, the conditions have to be similar. It is established through observations that temperature changes affects the viscosity directly. The temperature was fluctuating for both automated and manual test at Sandsli (Saptharishi, 2016), as well as existing uncertainties regarding resting times. The Fann tests by PS (2016) at IPT was conducted at exact 25°C, and information regarding resting times was more precise. By establishing confidence intervals for the comparable tests performed in this work – with desired state of probability – one can assess reliability to the previous obtained results based on whether they fit into the intervals or not. As number of samples are limited in a statistical view, variation from arithmetic mean value could also be used to assess reliability of both measurements performed in this project and in the previous by Saptharishi (2016).

3.7.3 Statistical notation

It is chosen to look at results as estimations, as they do not represent the exact truth for all mixtures of the same concentrations. It is prepared a number of samples, to represent and create an image of the behaviour, not to state exact properties valid for all instances of these mixture types.

4 Measurement Results and Analysis

In this chapter, all results from the Fann viscometer tests will be presented and analysed from a statistical point of view. Complete tabulation of measurement results can be found in Appendix C Complete . A total of 49 samples were measured, distributed among the selection of fluids found in test matrices outlined in Chapter 3. Graphics will be used extensively.

4.1 Precision and Accuracy of the Measurement Method

The samples which are mixed and tested, are not a perfect representation of how the properties for the actual mixture is. The number of samples involved gives an image of tendencies, and thereby a glimpse of the behaviour. There is also a huge amount of parameters which will influence mud in different wells, under which the circumstances during this work is not possible to recreate or foresee. The conditions used for these samples are simplified, as may also reduce the risk in respect to the number of parameters which brings errors into the test results. Sample preparations are also unique processes every time they are repeated. Mixing equipment could differ, so will probably routines at different companies, together with surroundings that likely never will be exactly identical.

When it comes to precision of the measurements themselves, precision can be related to the scatter of results for the same system, repeated through a well-defined experimental procedure (Davis, 2008). As new samples in this work are mixed frequently and the preparation itself can contain differences, it is difficult to verify the precision of the measurements by these results.

For determining the accuracy of a measurement, it is common in technological analysis, to use a reference value (Davis, 2008). This value is often based on agreement among experts, or through procedures agreed upon within the field of interest. The reference value chosen to be best suited to state something about the accuracy of the Fann viscometer readings in this work, is the calibration fluid of 100 cp at 25 degrees C. By using this reference fluid, it is also possible to state something about how precise the measurements are, as they are repeatable without any procedural error during preparation. This topic will be resumed after a brief description of the fluid calibration check involved in order to determine values for these expressions. The official Fann site states that the fluid calibration check should lie within +- 1.5 cp of the reference viscosity value. The value is obtained from taking the 600 RPM reading, divided by 1.98 (Fann Instrument Company, 2016). In Table 5: Fluid calibration check of Fann viscometers below, it is clear that both viscometers are inside of this tolerance, with values of 100.3 and 99.7cp. Readings from instrument number 3 was adjusted with one unit, as it showed a negative value of -1 at the zero point. When the adjustment is taken into account, the readings seems to be consequent except a deviation of 0.5 at 200 RPM. This may also be due to the eye that reads.

Table 5: Fluid calibration check of Fann viscometers, where the tabulation represents the Fann dial readings.

25 °C	Fann	Fann viscometer RPM (shear rate below (1/s))						Viscosity
Mixture	Instrument	3	6	100	200	300	600	Reference (cp)
	Number #	5	10	170	340	511	1021	
Calibration fluid	1	1	2	34	68,5	101	198,5	100,3
Calibration fluid	3	0	1	33	67,5	100	197,5	99,7
Calibration fluid	1	1	2	34	68	101	198,5	100,3
Calibration fluid	3	0	1	33	67	100	197,5	99,7

As the calibration fluid is a Newtonian fluid, viscosity can easily be calculated at the different shear rates, to further investigate the precision and accuracy of the rheological tests. The precision of the measurement method is based on the scatter of viscosity calculated from the Fann readings. These are presented in Table 6: Viscosity after adjustment of deflected needle in Fann instrument #3, and appurtenant statistical properties, and shows that the variation from the mean is 0.24%. This will not influence rheological properties significantly, and it is possible to claim that the measurement method is by definition precise.

Table 6: Viscosity after adjustment of deflected needle in Fann instrument #3, and appurtenant statistical properties

25 °C	Fann	Fann viscometer RPM (shear rate below (1/s))					
Mixture	Instrument	3	6	100	200	300	600
	Number #	5	10	170	340	511	1021
Calibration fluid	1	100	100	102	102,75	101	99,25
Calibration fluid	3	100	100	102	102,75	101	99,25
Calibration fluid	1	100	100	102	102	101	99,25
Calibration fluid	3	100	100	102	102	101	99,25

Arithmetic mean (Equation 2Equation 2) = 100,77 cp
Variance (Equation 3) = 1,35
Standard deviation (Equation 4) = 1,16
Standard error (Equation 6) = 0,24
Variation from the mean = 0,24 %

From the resulting values in Table 6: Viscosity after adjustment of deflected needle in Fann instrument #3, and appurtenant statistical properties the arithmetic mean value $\bar{x}$ of the viscosity is 100.77 cp, while the reference value R^* of the calibration fluid was stated to be 100 cp. Applying Equation 7, we get that the systematic error is equal 0.77, which defines the accuracy of the method. This is a slightly overestimation – but a tolerable value – and supports the generalized view of Fann viscometers as reliable instruments.

4.2 Partition of pure PAC Samples

One of the first observations made, related to the sample preparation and testing of pure PAC samples was the evident partition which appeared in the samples after a relatively short amount of time. Such tendencies could be observed already after 12 hours of rest, and became obvious after 24. When reaching 48 hours, interfaces between layers were clear and contained no blurred transition between the interfaces. In Figure 11 the phenomenon is shown. It must be emphasized that all mixtures containing 2% and 4% PAC – except with addition of XG –

showed this behaviour in spite of a homogenous condition after preparation. Partition made re-stirring of samples before each test essential. Figure 12 displays a graphical representation of the phase proportions measured for 4% and 2% PAC.

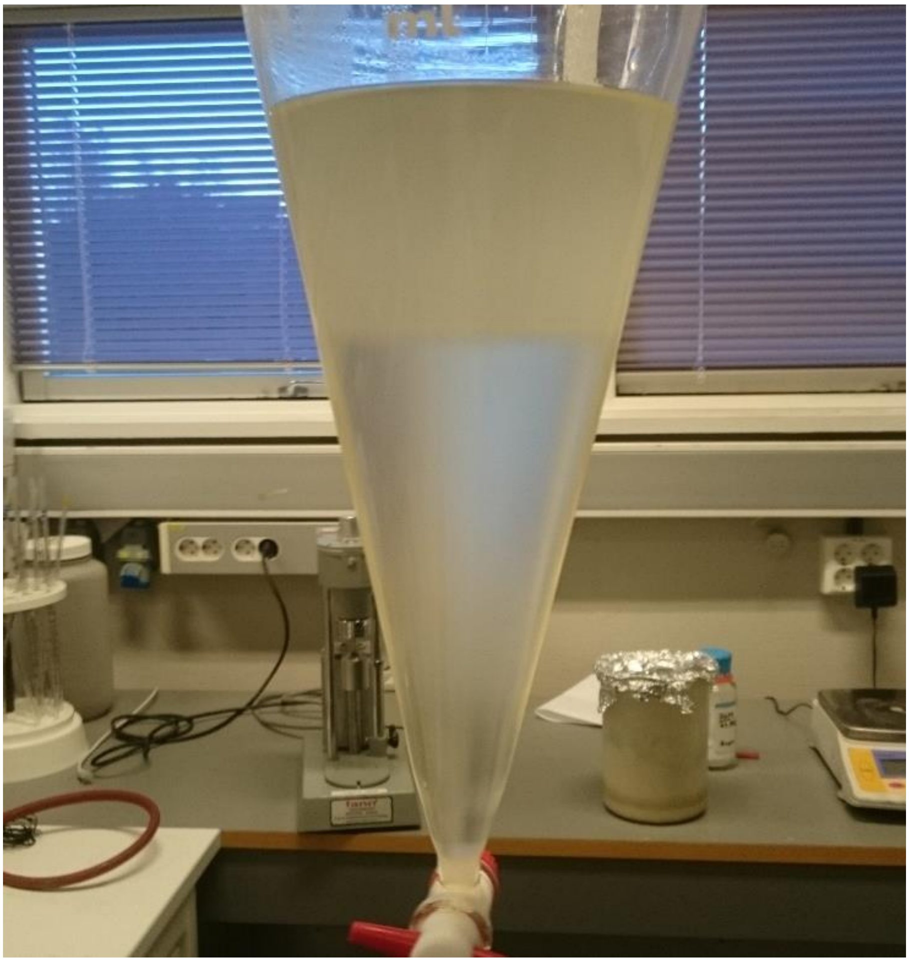

Figure 11: Partition of 4% PAC 24 hours after mixing.

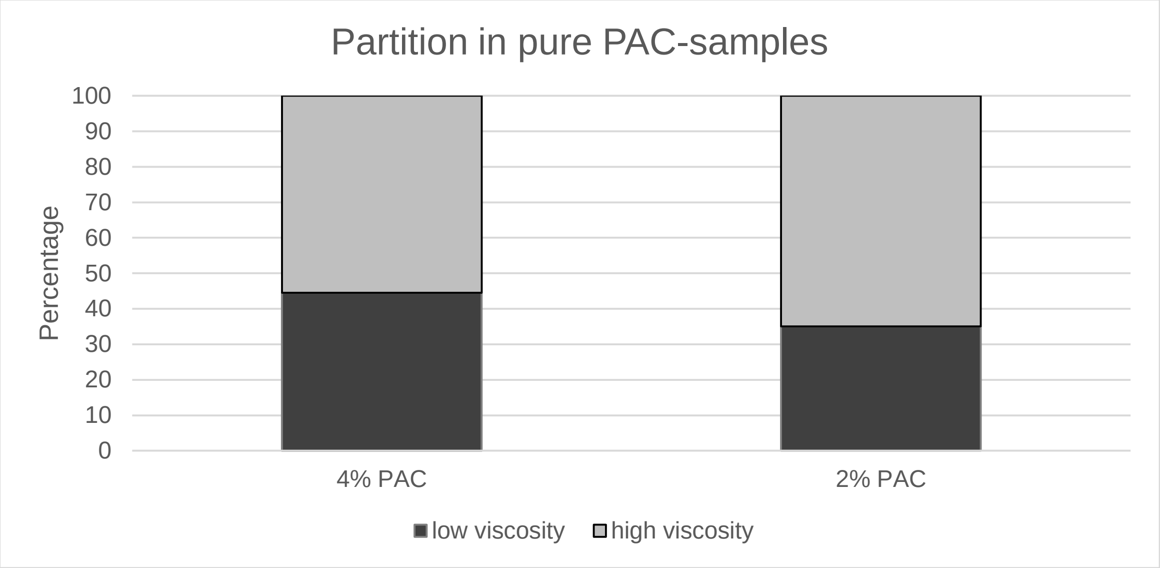

Figure 12: Proportions of partition in pure PAC samples

The properties for each of these phases in 4% PAC have evident dissimilarities which is shown in **Error! Reference source not found.**. The 2% PAC showing a less extreme curvature.

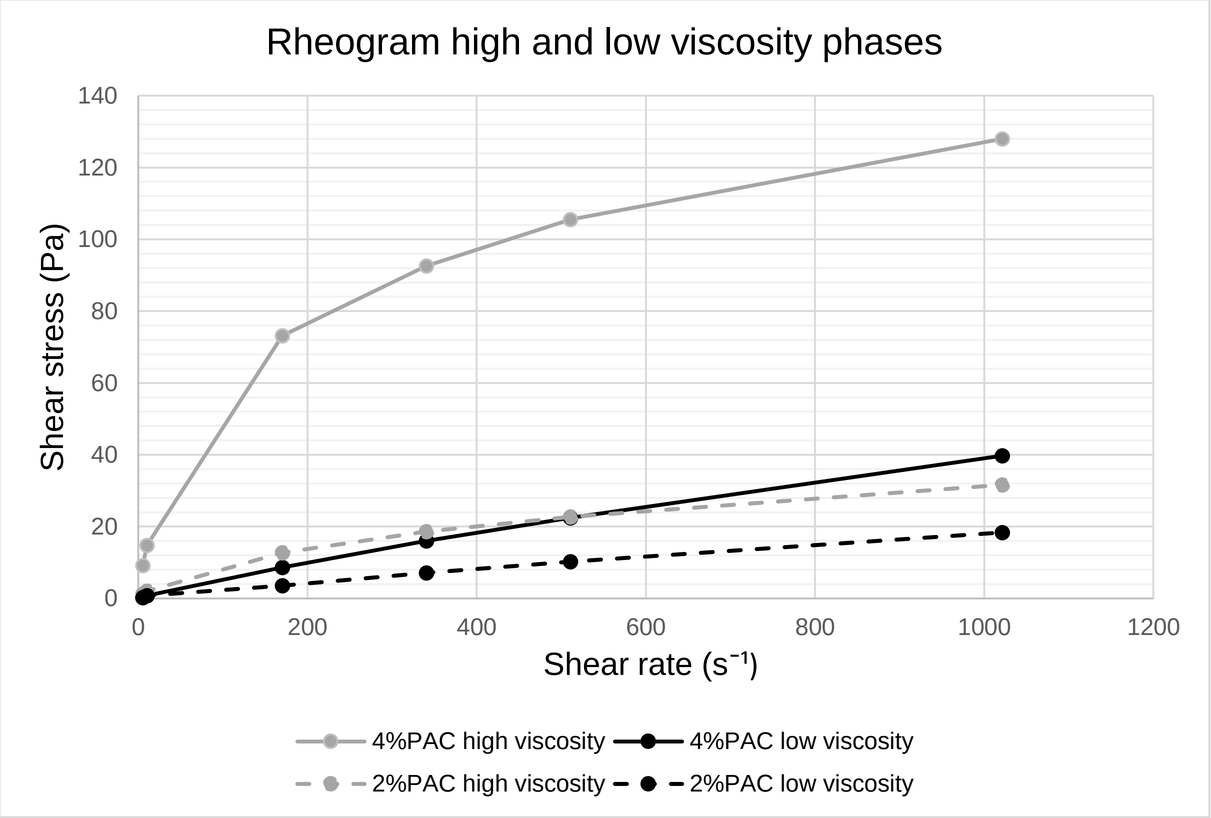

Figure 13: Rheogram displaying the rheological differences of the phases for 4% PAC and 2% PAC

An interesting observation is the relation between the high viscosity-layer on the top and the concentrations. While the low viscosity phase behaves like a Newtonian fluid, the high viscosity phases seem to look like the Herschel Bulkley model. According to section 2.2 Polymer fundamentals, it may imply that the main polymer additives are concentrated in the upper layer. Comparing to the partitions in Figure 12, it seems that the 4% concentration is less dispersed than 2%, which could indicate that the solubility lies in between these concentration levels. This is not inconceivable, as there is provided different information about the solubility as mentioned in section 2.3.1 Poly-anionic Cellulose (PAC).

The density of the sample after re-stirring compared to the separated phases are shown in Figure 14. The difference of the upper and lower layer is 0.01 g/cm^3. This is not a significant value, but a valid observation of the property.

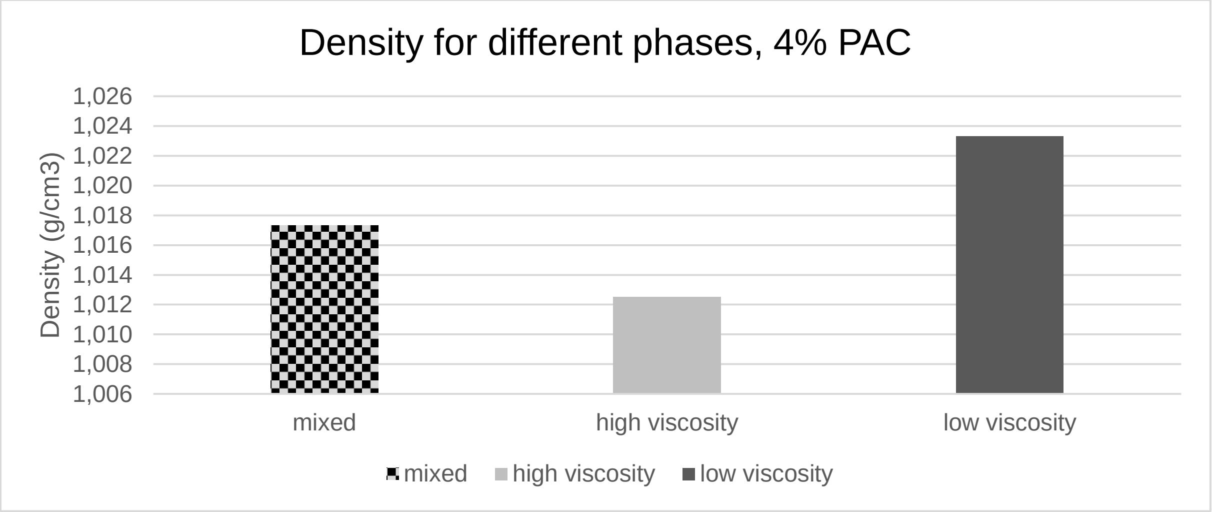

Figure 14: Density for different phases in 4% PAC partition after 24 hours

4.3 Mud balance density measurements

A comparison of the densities measured in these experiments compared to Saptharishi's (2016), gives an indication whether the mixtures contain evenly amounts of additive. The mud balance itself is not an accurate instrument for second decimal readings in the ppg-scale. All readings of more than more decimal are approximate values, and depends on the eye that reads. The results are presented in Figure 15: Anticipated vs measured densities, using Mud balance. below, and shows small differences taken the axis scale into consideration. The anticipated densities are Saptharishi's (2016) obtained values. Observations worth mentioning, is that the 2% PAC and 4% PAC measured densities are reversed compared to the anticipated ones, such that 4% PAC is slightly denser than 2% PAC. This makes sense as more additive is dissolved into the water, without making any real impact on the volume. The difference regarding 4% PAC + 20% barite is 0.013 g/cm^3. Taken the sedimentation of barite particles into consideration it seems to be an acceptable difference.

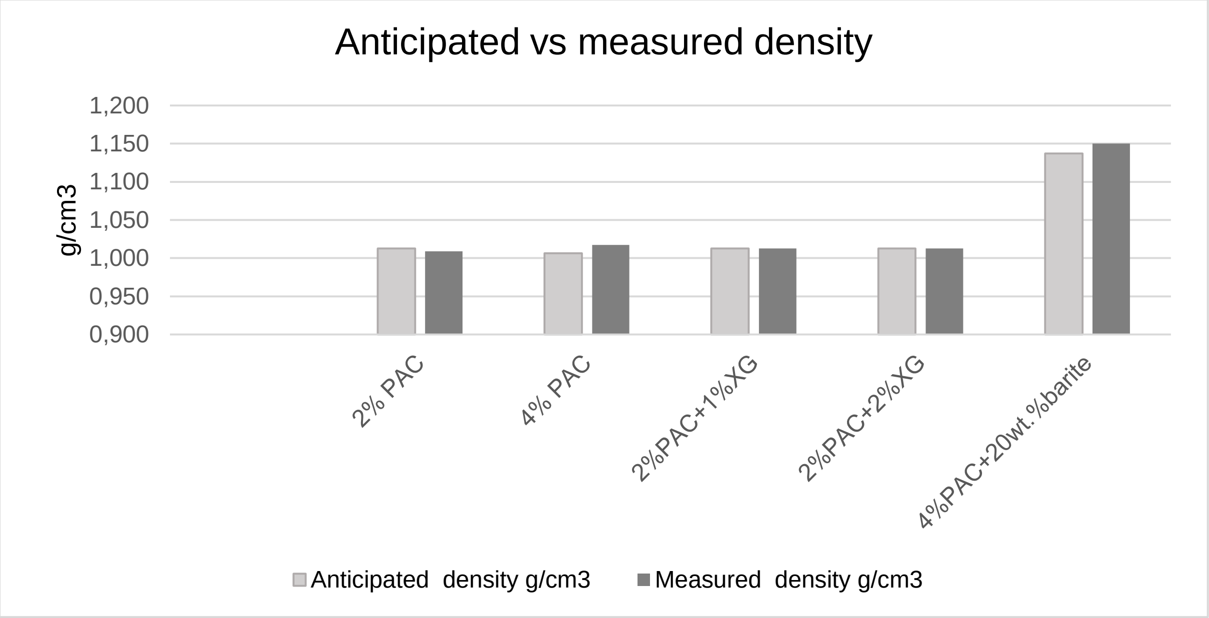

Figure 15: Anticipated vs measured densities, using Mud balance.

4.4 Temperature impact

Temperature is expected to have an evident impact on rheological behaviour. For deep water drilling a wide range of temperatures can be expected, from > 150°C downhole to approaching zero at the seafloor. Knowledge of behaviour at varying temperatures is therefore important.

4.4.1 Temperature-Viscosity

The initial test matrix which was presented in section 3.2.1 Initial matrix contained selections of three different test temperatures: 5°, 25° and 50°. This was completed for the pure PAC concentrations. As seen in Figure 16: Temperature impact 4% PAC below, the temperature affects the shear stress in 4% PAC directly, so there is no doubt of the inverse correlation between viscosity and temperature which were described in section 2.6 Temperature influence. The same can be seen for 2% PAC, which can be found in Figure 38 in appendices.

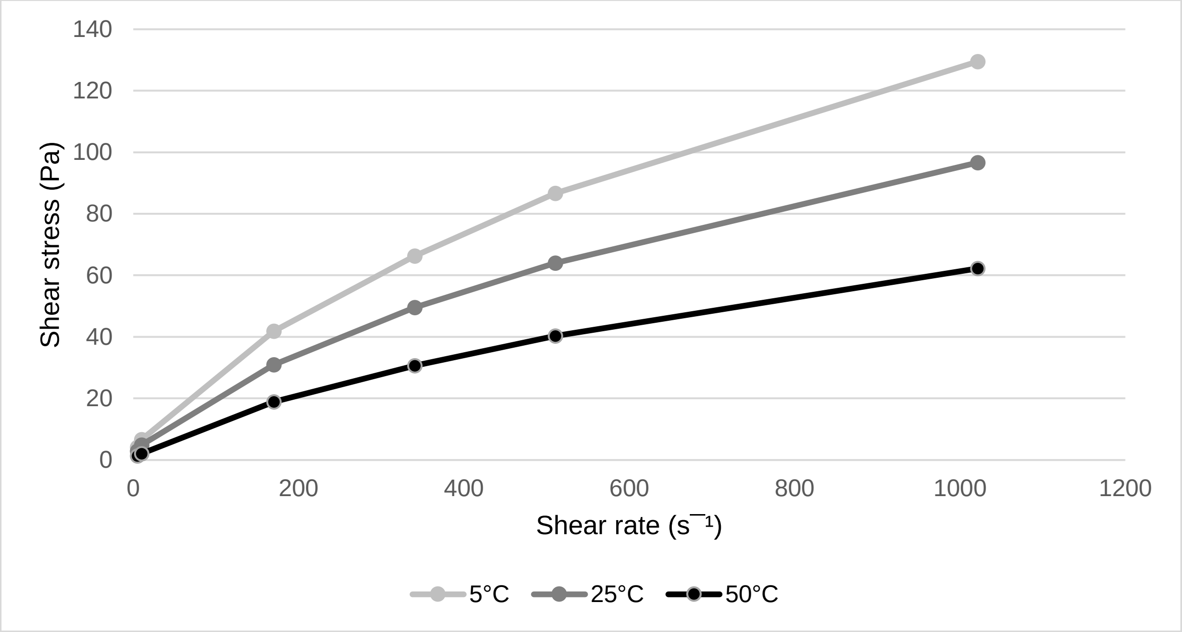

Figure 16: Temperature impact 4% PAC

Results obtained by (Saptharishi, 2016), will from now on be denoted by PS (Priyadharshini Saptharishi). She did compare results obtained by Offite viscometer, Fann viscometer at the laboratory in Sandsli Bergen and at IPT. At Sandsli the temperatures was not measured exactly at 25°C. They have been plotted with results from this project, Figure 17: 4% PAC measured with different instruments at approximately 25°C in order to see an approximation of how they compares to each other. The Offite viscometer has lower value at the highest shear rate, as might be because the temperature in that sample was 30°C. Fann test at Sandsli have slightly higher values, probably due to lower temperature of the sample. Taking temperature differences into account, results seems to be very similar, except an inconsistency in the trend at the shear rate of 170 for both Fann - PS IPT and PS Sandsli.

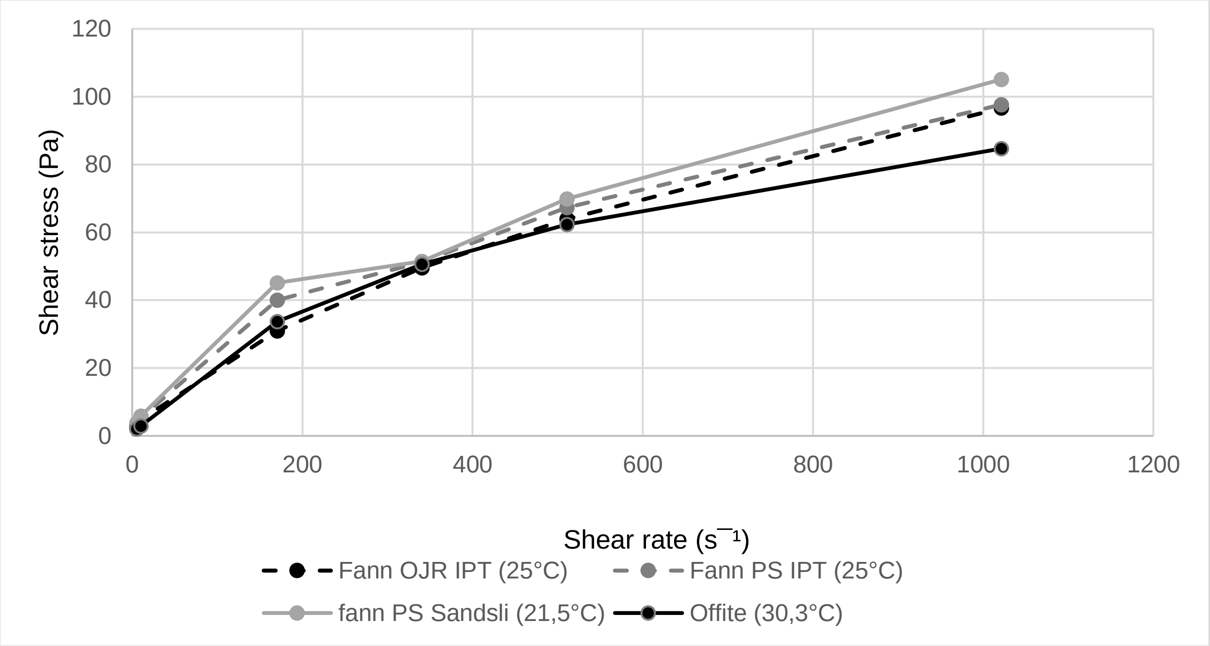

Figure 17: 4% PAC measured with different instruments at approximately 25°C

The same test were also performed at 50°C, and all these temperatures is given as exact temperatures. These are found in Figure 18 below and proves to very accurate – which implies that if conditions are set precisely – different measuring methods gives the same results. An additional comment is that mixing procedures for this project at IPT, PS at IPT and PS at Sandsli have all been different.

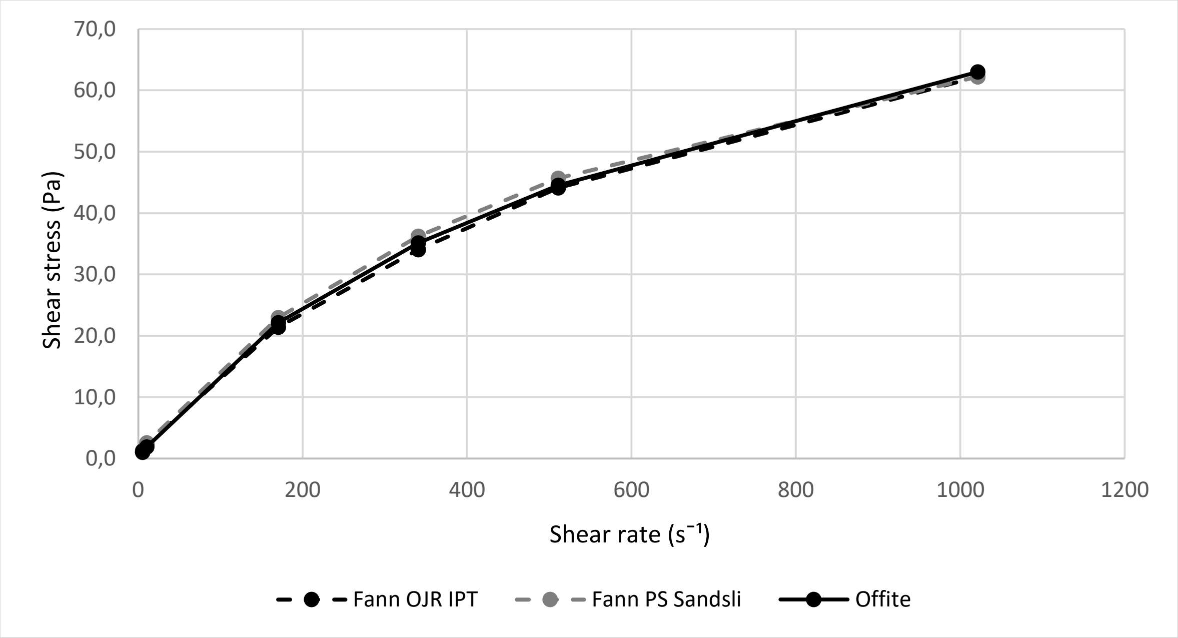

Figure 18: 4%PAC measured with different instruments at exactly 50°C

Looking more detailed at the outcome of the measurements, the variation from the mean values are doubled in the situation where the parameters are not consistently matching each other, compared to the one having exact same temperatures, seen in Table 7: Bias in different measuring processes. The percentage value is not an entirely good image because of huge errors in the lower RPM readings, where approximations need to be made in order to round up or down to nearest value. The standard deviation for the 50°C measurements is a fifth of the other, making it far more precise.

Table 7: Bias in different measuring processes

Shear rate (s^{-1})	5	10	170	340	511	1021	AVG
Standard deviation 25°C	0,7	1,3	6,4	0,9	3,4	8,4	3,5
Variation from the mean	7,06 %						
Standard deviation 50°C	0,1	0,3	0,8	1,1	0,8	0,4	0,7
Variation from the mean	3,46 %						

In progress with the initial test matrix, samples were mixed at 50°C before cooled down to 25°C and tested. Even if this was changed for the final setup, it revealed some interesting observations (Figure 19: Temperature effects over time align in 4% PAC (measured at same temperature). Initial differences seem to align after about 10 days. The same trend shows for other compositions as well, and figures can be found in appendices.

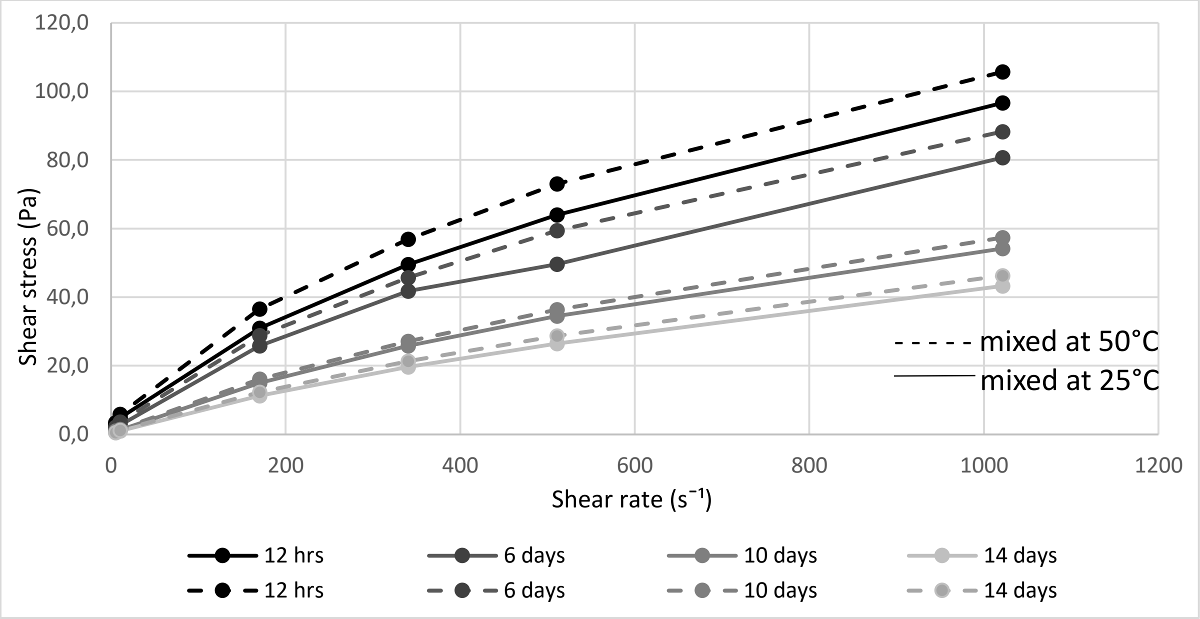

Figure 19: Temperature effects over time align in 4% PAC (measured at same temperature)

4.4.2 Temperature vs Gel strength

Decreasing tendency in gel strength has been observed consistently with pure PAC samples, but will be discussed in more detail later. Gel strength was measured at different temperatures, seen in Figure 20: Temperature impact on gel strength. As for the viscosity, the 4% PAC seems to be directly affected by the changes in temperature, while 2% PAC is almost unaltered. Observations worth mentioning is a stabilized gel strength in 4% PAC after 30 minutes for 5°C and 50°C, and that increasing values are observed beyond 10 minutes at 50°C. For 2% PAC, 25°C has the lowest measured values of the temperatures, but the differences are small.

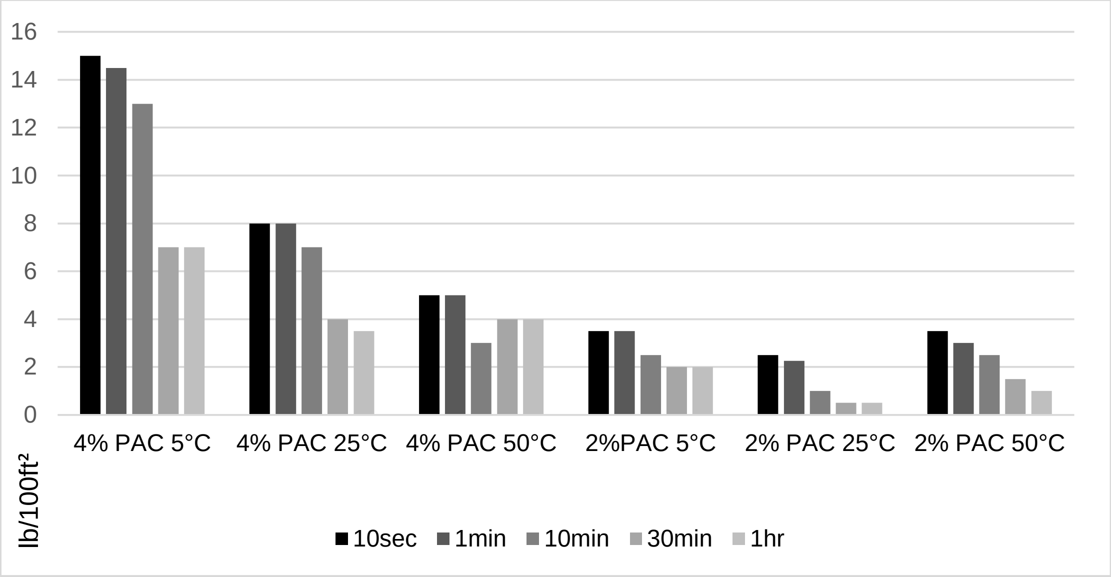

Figure 20: Temperature impact on gel strength for pure PAC solutions

4.5 Time-related degradation

Possible degradation over time was one of the main scopes for the experiments in this work. Degradation is here referred to as decreasing viscosity over time. After some measurements using the initial test matrix, the frequency of the testing times was increased in order to gain more precise knowledge about inflection points in the behaviour. The measurement numbers are extensive and results will be presented individually for the different compositions in order to make it easy to follow. All samples involved in this section are mixed at 25°C, and not exposed for higher temperatures. This is of course a simplification, and less probable for real applications, even if the influence of properties after HT exposure seems to be reversible.

4.5.1 2% PAC

Measurements for 2% PAC seemed to be very precise with little bias between different samples, especially at the two lower shear rates. From Figure 21 it can be seen that the viscosity (related to shear stress) is about stable during the first 3-6 days, before it decreases with almost linear rate to the 14th day. During the time window of measurements, shear stress is decreased by 10 Pa, or 26%. The samples had a transparent look during the whole aging process, but with partition after rest as explained previously.

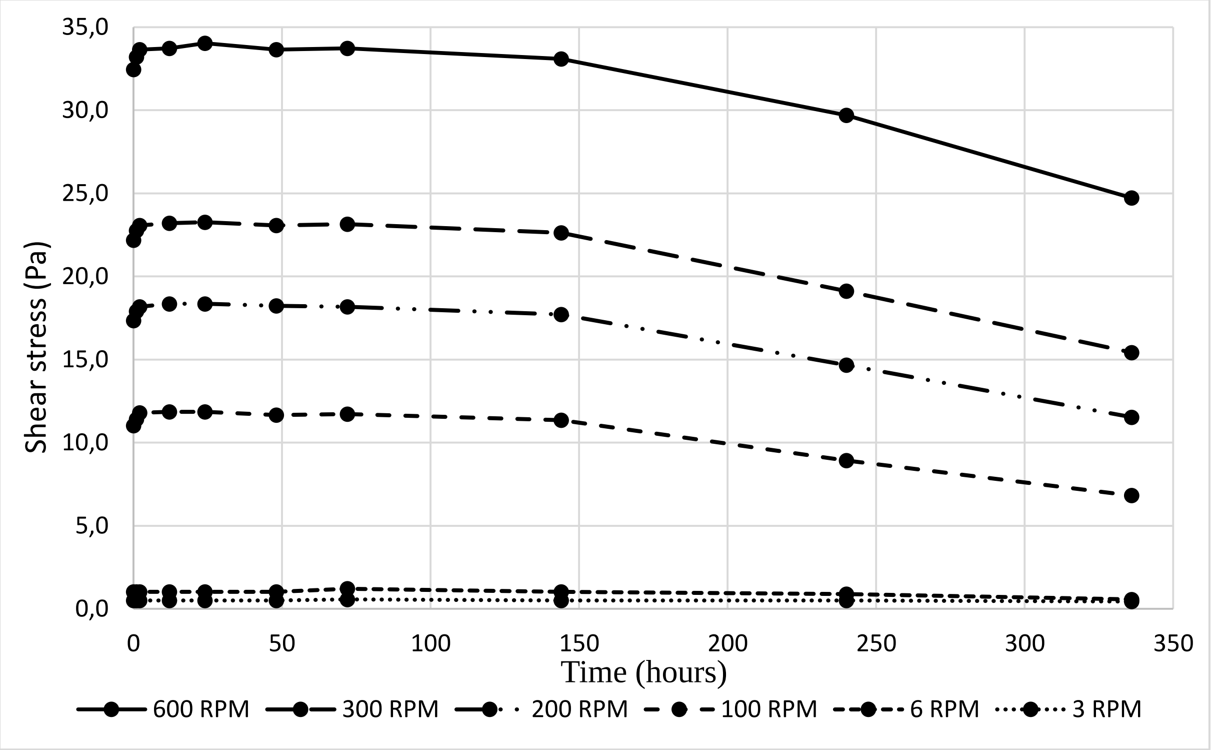

Figure 21: Degradation with time, 2% PAC

There were conducted time-measurements for a total of 9 samples, and the average standard deviation distributed for each time-frequency is shown in Figure 22. The maximum average standard deviation found is below 0.7 Pa, which is a very good result. Comparing the degradation and the deviation, it is obvious that degradation brings along more variation in measured values.

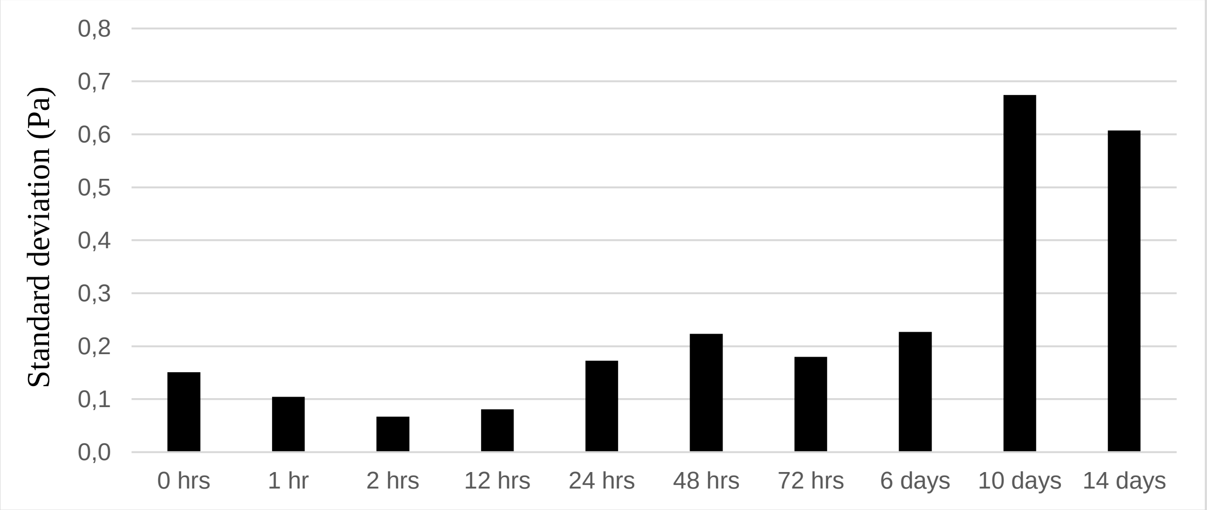

Figure 22: Standard deviation distributed for each time frequency 2% PAC

Gel strength (Figure 23) seems to be of a stable character for 6 days, before it decreases. It is proportional to the behaviour of viscosity. Consistent results was obtained for gel-values and deviations for these readings can be found in appendix B.2 Time-related degradation.

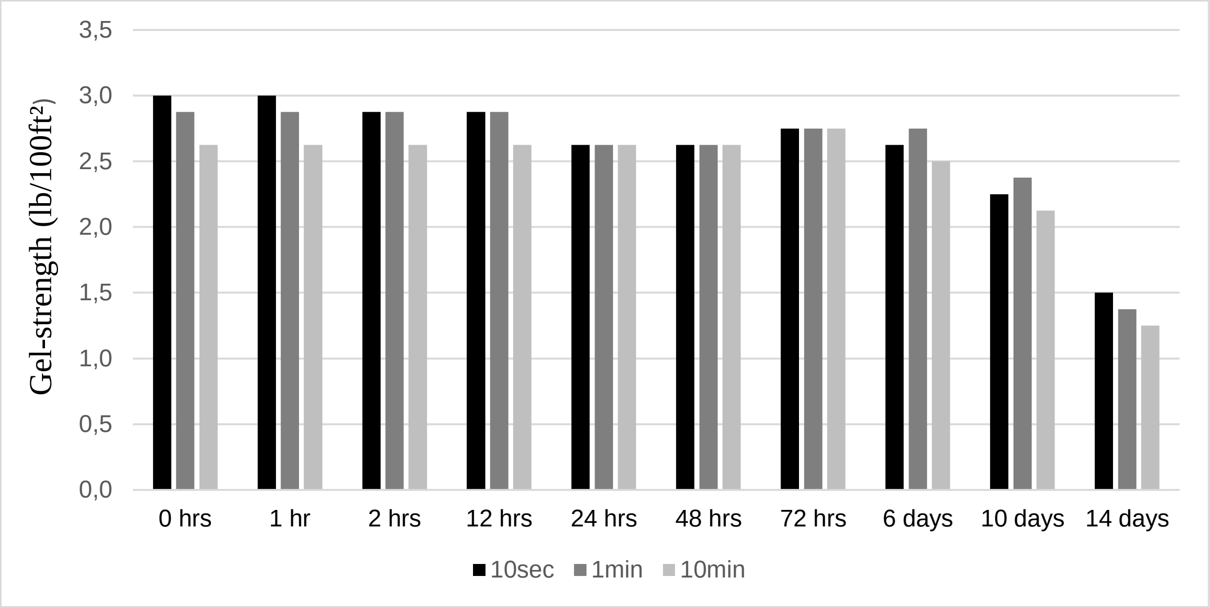

Figure 23: Gel strength vs time 2% PAC

4.5.2 4% PAC

A total of 6 samples were measured with respect to degradation (Figure 24). Measured shear stress appears stable for 3 days before it decreases rapidly till 10 days. The tendency flattens a bit between 10 and 14 days. Shear stress is more than halved during 14 days for all shear rates.

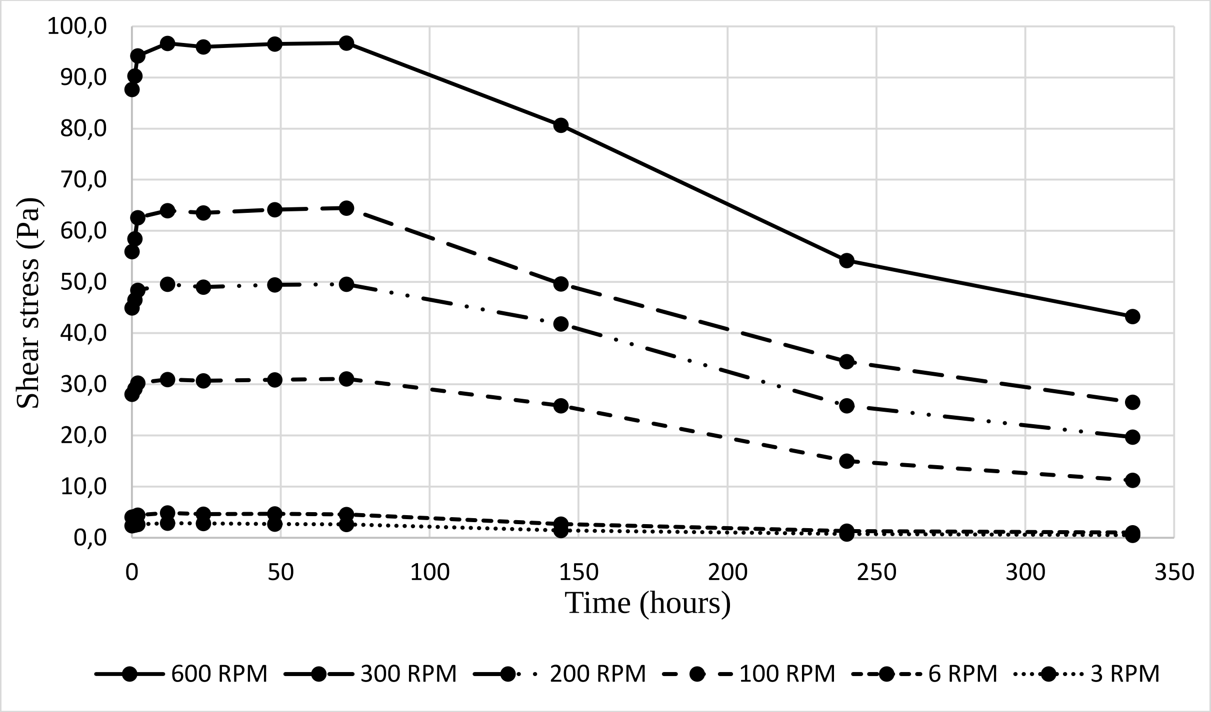

Figure 24: Degradation with time, 4% PAC

In the measuring process, heterogeneities was experienced in some of the samples, and more varying results than for lower concentration of PAC. The average standard deviation for the different points in time is presented in Figure 25. Magnitudes of deviation are increased compared to low concentration, and the largest values are found where the slope of degradation is the steepest – between 3 and 10 days.

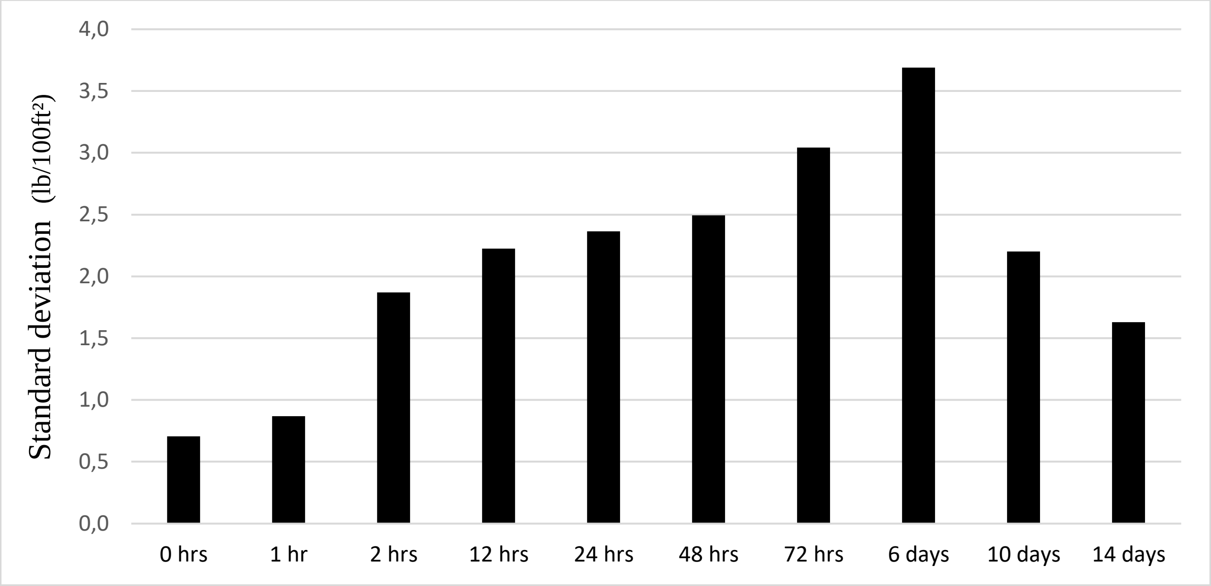

Figure 25: Standard deviation distributed for each time frequency 4% PAC

Gel strength (Figure 26) follows the same trend at the measured viscosities, with a steep decrease after 3 days. Measurements at 6 days was fluctuating more than for the other points while they were most stable for 10 and 14 days. The latter could be related to the magnitude of the values.

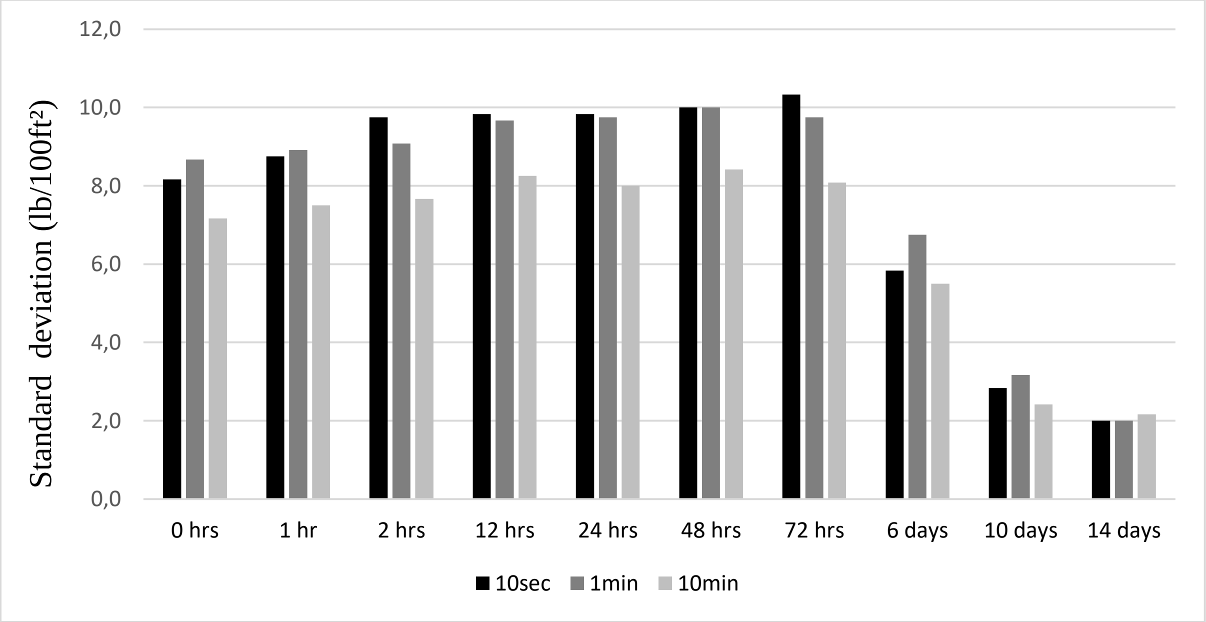

Figure 26: Gel strength vs time 4% PAC

4.5.3 4% PAC + 20 wt. % barite

Addition of barite to PAC solution resulted, not surprisingly, in a similar behaviour. Slightly increased viscosity at the beginning compared to 4% PAC, and also further degraded after 14 days. The 72 hours measurements had to be excluded due to some technical issues. 6 samples were tested for this mixture. Standard deviation correlated with 4% PAC- values is found in appendices.

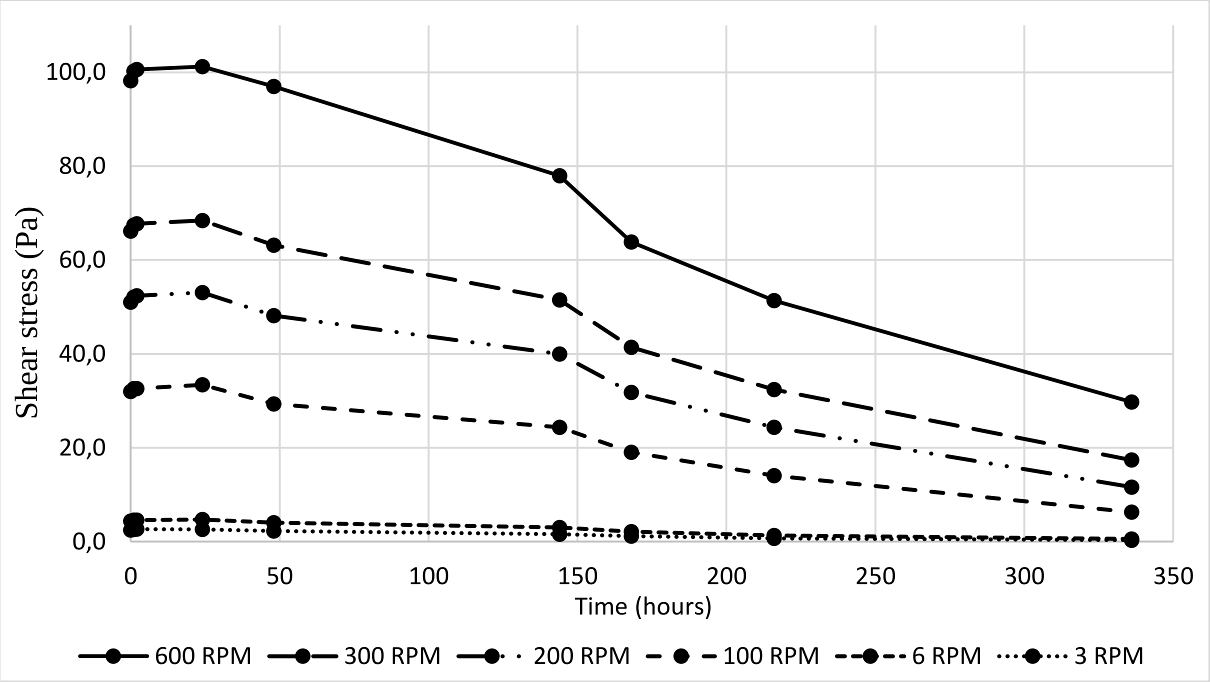

Figure 27: Degradation with time, 4% PAC+ 20 wt. % barite

Gel strength (Figure 28) did show dissimilarities comparing to pure PAC, which had a flat profile throughout 72 hours. Mixing with barite gave decreased gel strength already after 24 hours.

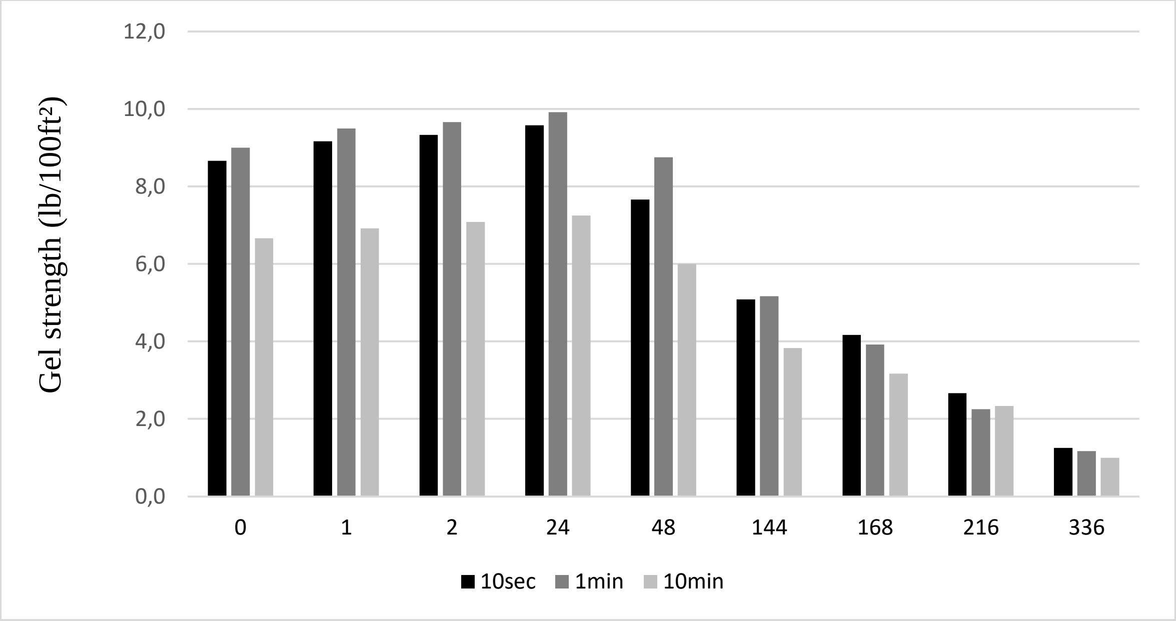

Figure 28: Gel strength vs time 4% PAC+ 20 wt. % barite

4.5.4 2% PAC + 1% XG

A total of 8 samples were measured for the time-dependency. Mixtures containing XG were more challenging in the mixing process, as agglomeration and lumps were easily created if careful attention was not paid. Gas was found in the samples after about 72 hours, and evolved each day after. This resulted in fluctuations for the viscometer dial in several of the sample measurements. In appendix B, there is a series of images showing the gas bubbles over three days. A finite stable yield stress is observed during the whole time window (Figure 29), but in a magnitude of about 5-10 less than Saptharishi (2016) reported. While the pure PAC compositions had stable values during the first three days, it is in this occasion decreasing with about 40 % during the same period. After 72 hours the viscosity seem to stabilize. Standard deviation was within 2.5 Pa, and the distribution is found in appendices.

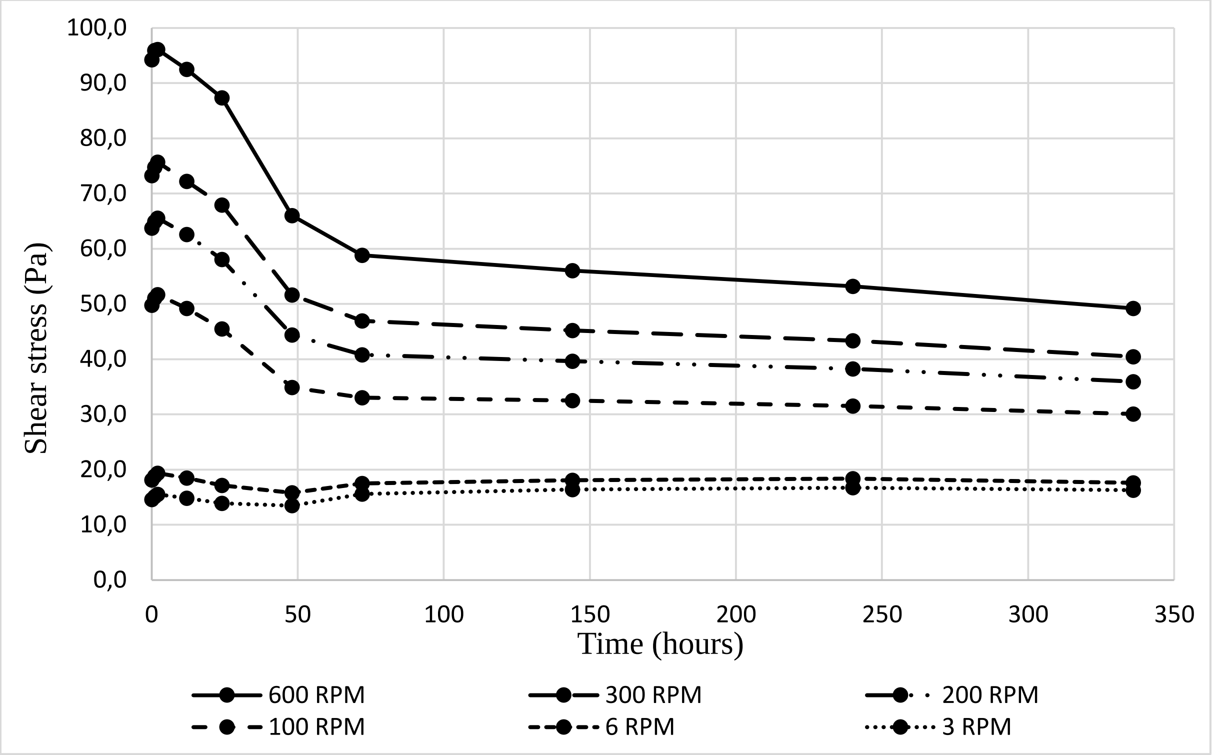

Figure 29: Degradation with time, 2% PAC+ 1%XG

Gel strength measurements (Figure 30) for this mixture brought some interesting results. A flat low increasing profile is observed for 24 hours, before it turns into a very progressive behaviour. The measured values was also of a low deviation which makes it reliable as well.

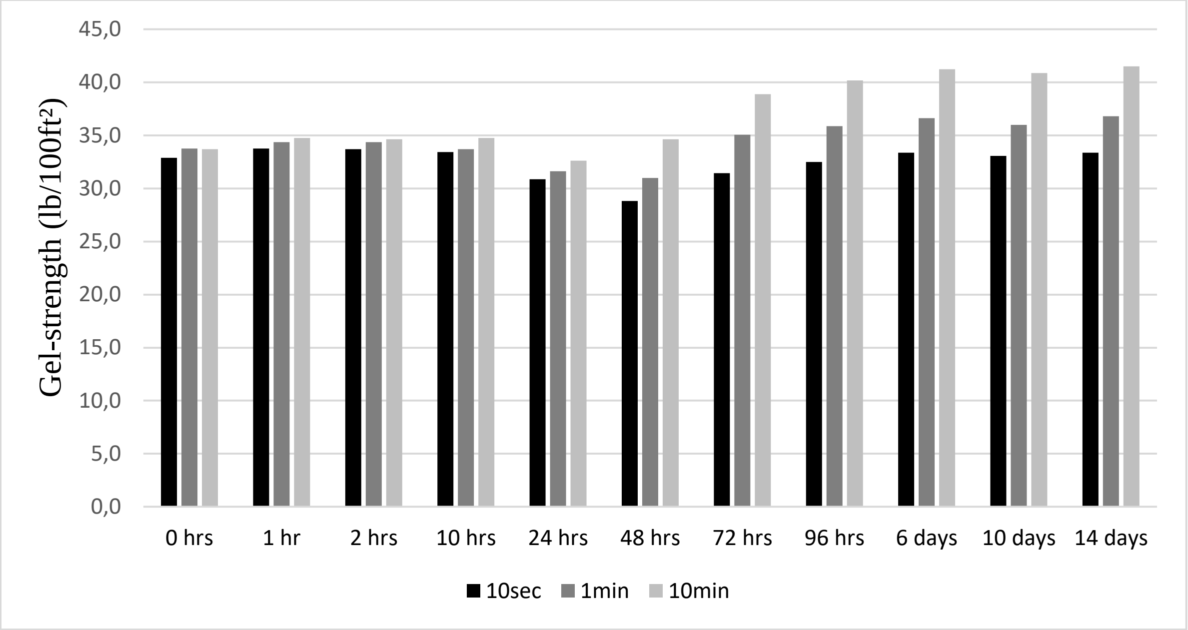

Figure 30: Gel strength vs time, 2%PAC+ 1%XG

4.5.5 2% PAC+2% XG

Compositions of 2% PAC mixed with 2% XG gave extreme viscosity values for high shear rates, and was not measureable at the higher rate during the first 12 hours, due to the maximum range of the Fann viscometer. Shear stress vs time is plotted in Figure 31. The viscosity behaviour seem to be exact the same as for the mixture containing 1%XG, but in lower proportions.

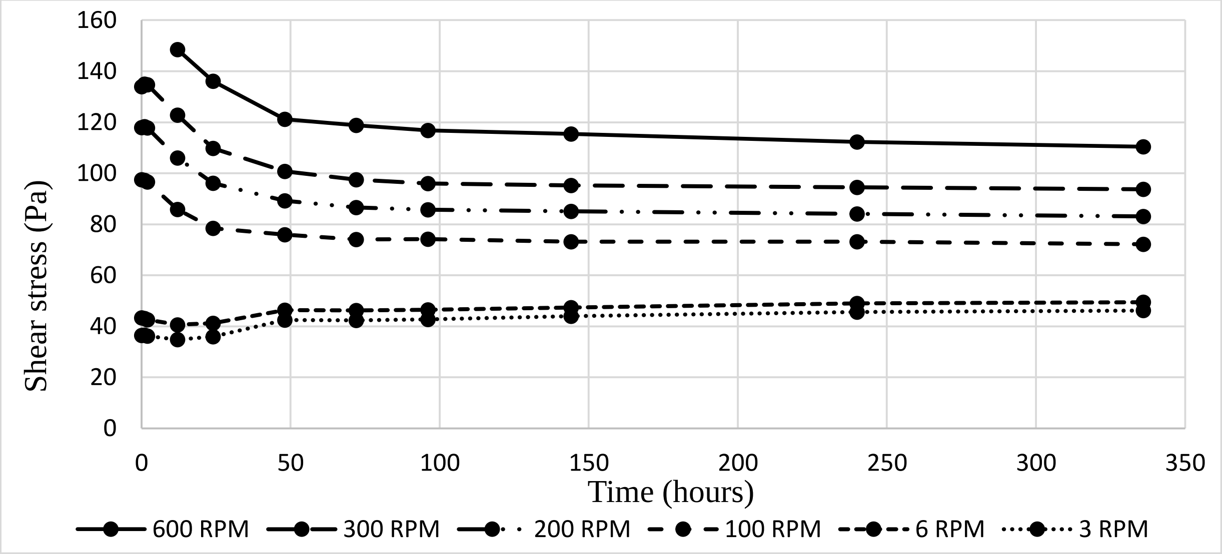

Figure 31: Degradation with time, 2% PAC+ 2%XG

Increased fluctuations for dial-readings also resulted in higher standard deviations (Figure 32), making the test results less reliable during the first 24 hours than for the other compositions. Gel strength values was of less value, as it seemed to slowly approach the finite yield stress when measured, without any peak as was observed for the other mixes.

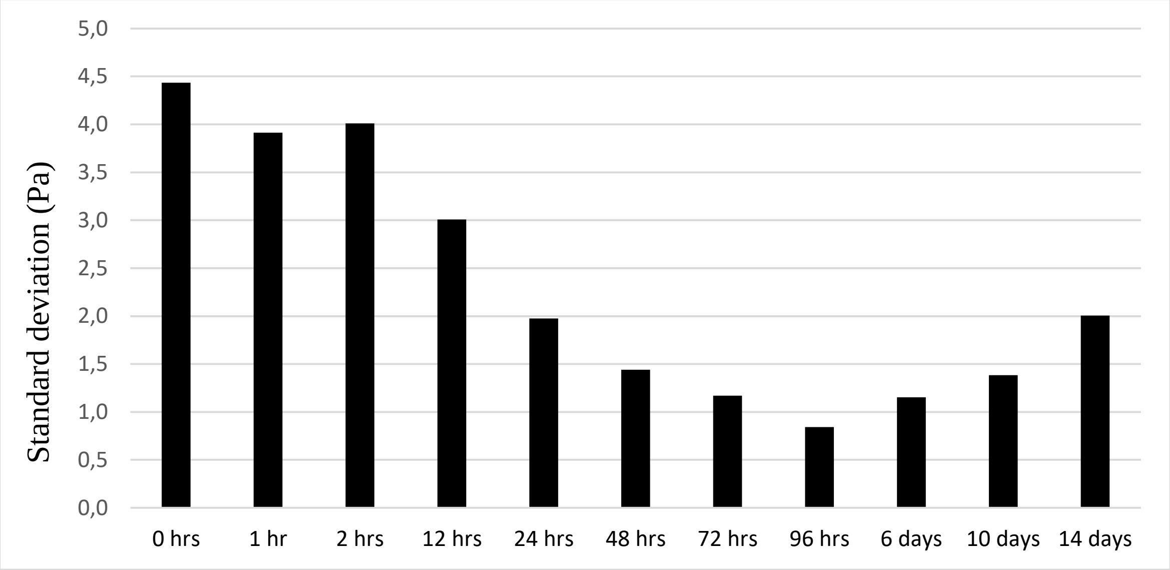

Figure 32: Standard deviation distributed for each time frequency 2% PAC+ 2%XG

4.6 Establishment of Reliability

Results from the measurements after 24 hours – and mixed at 25°C – are used, in order to establish 95 % confidence intervals which should be a desired level of certainty. As the number of samples are relatively small statistically speaking, the t-distribution will be used. Variation from the mean in percentage may be an even better way to directly assess reliability, if they do not fit into created confidence intervals. The Fann 300 RPM reading is chosen for comparison, as it is the value of which apparent viscosities (AP) has been used. 2% PAC, 4% PAC, 2% PAC + 1% XG, 2% PAC + 2% XG and 4% PAC + 20wt. % barite will be investigated. For all compositions, a rheogram will be plotted, containing values from (Saptharishi, 2016) and arithmetic mean value obtained during this project. Subsequently, statistical material is presented.

4.6.1 2% PAC

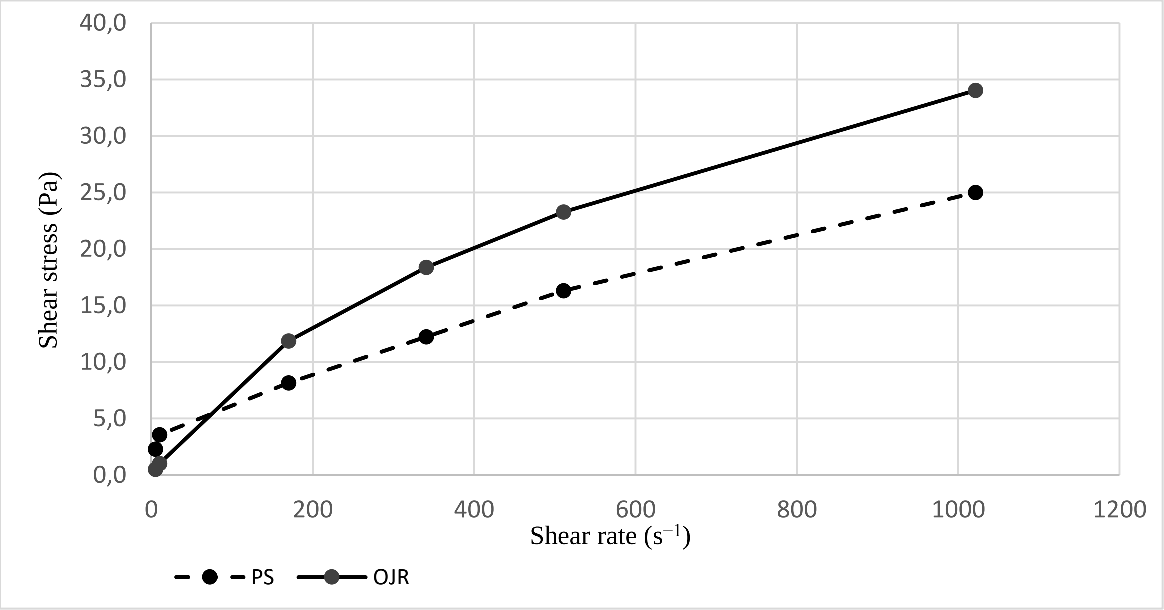

Figure 33: Rheogram compared values 2% PAC

Table 8: Statistical comparison 2% PAC

	Arithmetic mean	Standard deviation	Significance	Samples
300 RPM	45,63	0,48	0,05	4
95 % Confidence interval ±			0,76	
Resulting confidence range		44,86	to	46,39
Value PS			32	
Variation from mean value PS			28,2 %	
Average variation from mean value OJR			0,5 %	

By using Figure 33 and Table 8, the differences are clearly remarkable. As the variation from the mean in this work is 0.5 %, the experiments certainly seem to be repeatable and reliable under the same procedural conditions. Comparing value from PS IPT, the variation is 28.2 %, which is far away from what to be expected.

4.6.2 4% PAC

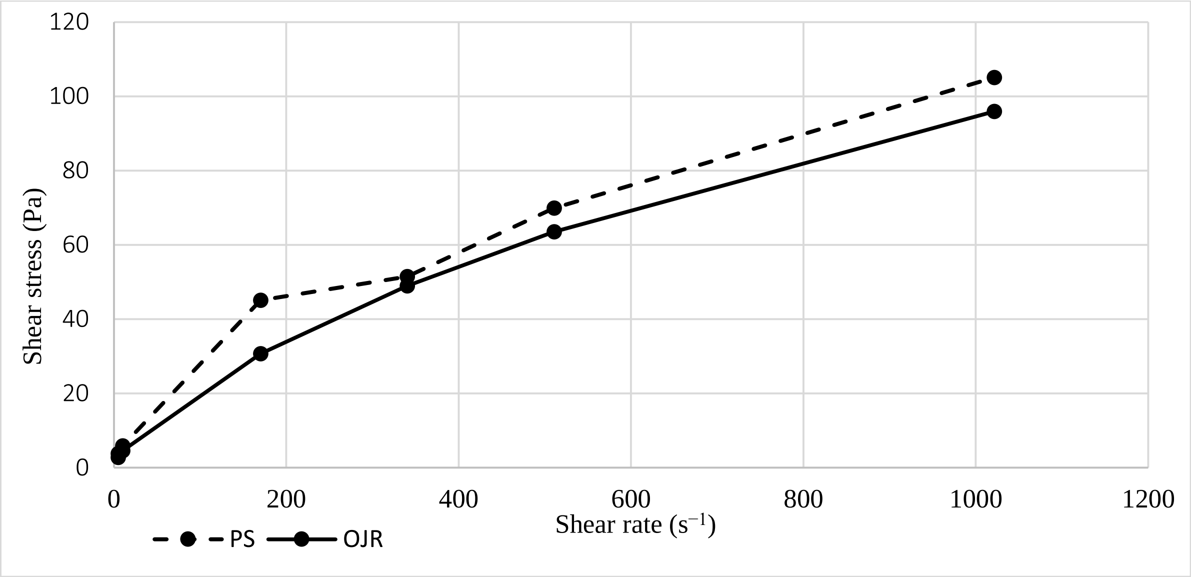

Figure 34: Rheogram compared values 4% PAC

Table 9: Statistical comparison 4% PAC

	Arithmetic mean	Standard deviation	Significance	Samples
300 RPM	124,50	7,14	0,05	6
95 % Confidence interval ±			7,49	
Resulting confidence range		117,01	to	131,99
Value PS			137	
Variation from mean value PS			10,0 %	
Average variation from mean value OJR			2,3 %	

Results obtained in this work compared to results by Saptharishi (2016) are seen in **Error! Reference source not found.** and Table 9: Statistical comparison 4% PAC. The rheogram shows same trending behaviour except a deviating curvature at a shear rate of 170 s^{-1}. The 6 conducted experiments for this project are resulting in 2.3 % variation from the mean, which could be classified as tolerable, but not very precise. Measured value by PS IPT has a 10 % variation from the mean, which means it will not fit in the confidence interval.

4.6.3 2% PAC + 1% XG

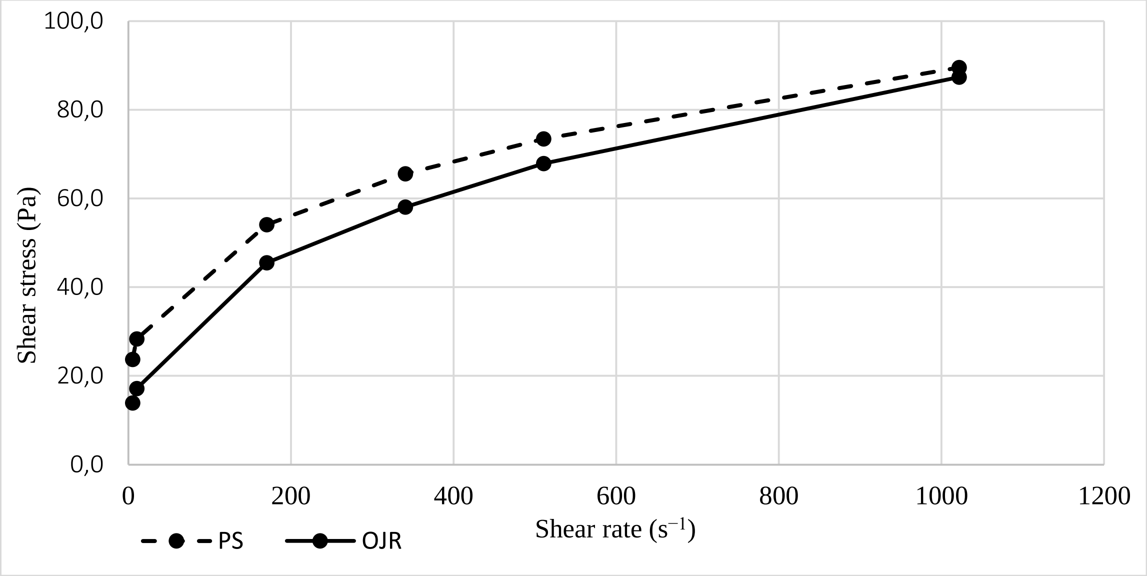

Figure 35: Rheogram compared values 2% PAC+ 1% XG

Table 10: Statistical comparison 2% PAC+ 1% XG

		Arithmetic mean	Standard deviation	Significance	Samples
300 RPM		133,13	3,85	0,05	8
95% Confidence interval ±				3,22	
Resulting confidence range			129,90	to	136,35
Value PS				144	
Variation from mean value PS				8,2 %	
Average variation from mean value OJR				1,0 %	

Results for 2% PAC + 1 % XG are compared in **Error! Reference source not found.** and Table 10. A total of 8 samples constitutes the measured values in this work, resulting in a 1 % variation from obtained mean value – which is good. As for the values by PS IPT, the rheogram shows that behaving trends are approximately the same, and the variation from the mean is 8.2 %. This does not fit into the confidence interval, but is closer than for the pure PAC measurements.

4.6.4 2% PAC + 2% XG

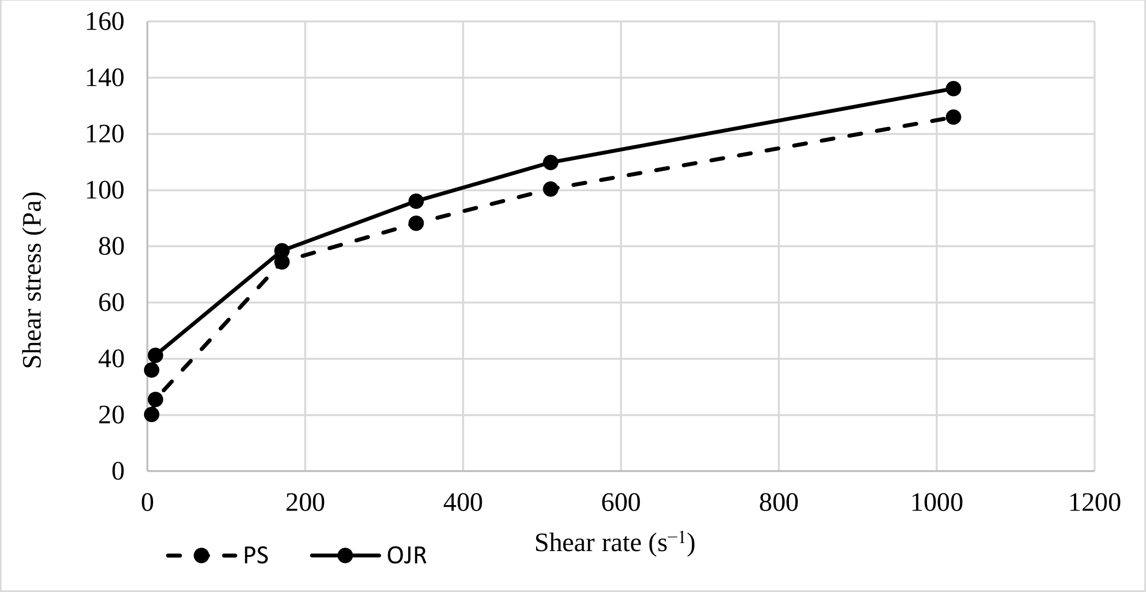

Figure 36: Rheogram compared values 2% PAC+ 2% XG

Table 11: Statistical comparison 2% PAC+ 2% XG

	Arithmetic mean	Standard deviation	Significance	Samples
300 RPM	215,33	4,93	0,05	6
95% Confidence interval ±			5,17	
Resulting confidence range		210,16	to	220,50
Value PS			197	
Variation from mean value PS			8,5 %	
Average variation from mean value OJR			0,9 %	

Comparing results for 2% PAC + 2% XG in Figure 36 and Table 11, the trend is about the same – except a deviation from the smooth curvature at a shear rate of 170. Measurements gathered in this project have a variation of 0.9 % from the mean value. Value from PS IPT does not fit into the confidence interval, and has a variation of 8.5 % from the mean.

4.6.4 4% PAC + 20 wt. % barite

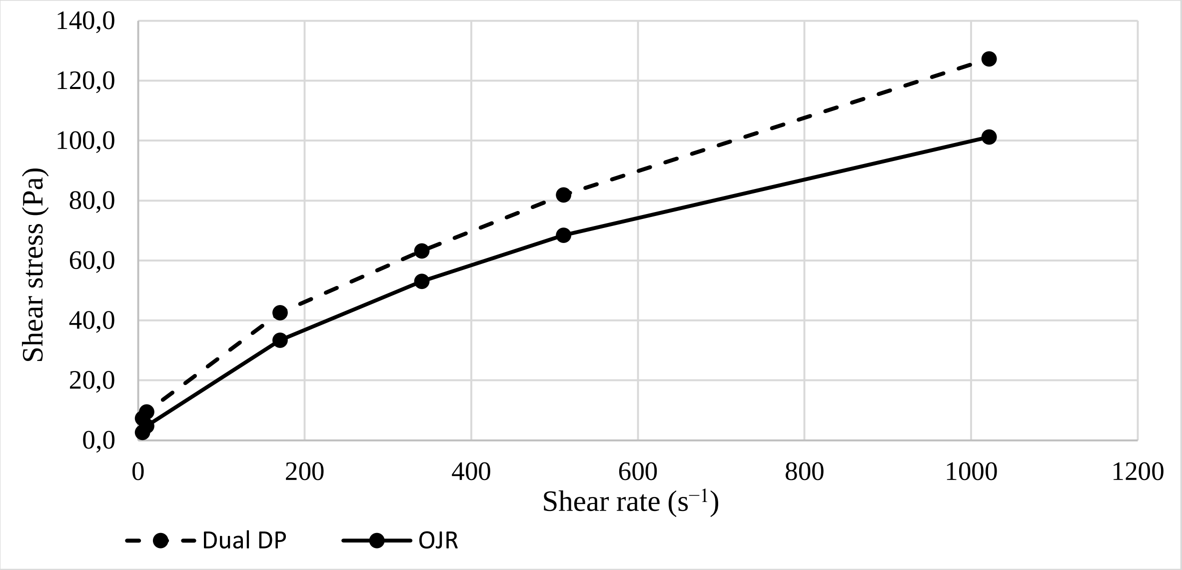

Figure 37: Rheogram compared values 4% PAC + 20wt. % barite, between OJR at IPT and Dual DP at Sandsli

Table 12: Statistical comparison 4% PAC + 20wt. % barite, between OJR IPT and dual DP Sandsli

	Arithmetic mean	Standard deviation	Significance	Samples
300 RPM	68,43	1,91	0,05	6
95% Confidence interval ±			2,01	
Resulting confidence range		66,42	to	70,43
Value dual DP (23°C)			81,9	
Variation from mean value dual DP			19,7 %	
Average variation from mean value OJR			1,1 %	

Comparing the manual measurements for PAC and barite vs the automated dual DP at Statoil Sandsli Bergen, in Figure 37 and Table 12. The rheogram shows the same trends, but an extra offset for manual measurements at the higher shear rate. As for comparing the values, they differ with almost 20 %, which is a lot. The dual DP having temperature at 23°C, which do possibly affect a little bit. There may also be differences in mixing procedures. Saptharishi (2016) did report higher measured values from manually measurements than for dual DP, explained by not taking frictional effects into account. In the above graphics it is evident that the opposite is the case here.

5 Discussions

Key observations and deviations will be summarized and discussed in this chapter.

The initial fluid calibration checks did confirm that with a reference fluid and given temperature conditions, Fann 35 viscometer is a reliable instrument. This is also supported by the extended use within the oil and gas industry

Partitions in the pure PAC samples was surprising, as it was an unknown behaviour in such extent. It is difficult to pin-point if this did impact the measurements, as all mixtures were re-stirred before testing. What is worth mentioning is that this behaviour was not reported by Saptharishi (2016), as makes it susceptible for being related to the mixing procedure which was performed at lower RPM. This may not have been sufficient to get it properly dispersed into the water, even if the solutions appeared to be homogenous and slightly transparent. Measurements of pH values should possibly have been integrated in the test setup, in order to have an increased number of parameters in the toolbox for explaining a phenomenon as the partition.

The experimental results confirmed the related relationship between viscosity and temperature, as an inverse correlation. Comparison of results from Sandsli and IPT, with different instruments did also reveal how tightly related this relationship is. The results based on measurements at approximately 25°C had five times the standard deviation as the measurements which was completed at an exact temperature of 50°C. It is of that essentially important to keep an eye on such a parameter when producing results for comp ability with others. Samples exposed to 50°C during mixing had altered properties for a period of time compared to those only kept only at 25°C. It seemed to take between 7 and 10 days before the properties aligned for the different procedures. It would be reasonable to call that reversible effects, which also is in line with what a number of manufacturers states in their product specifications.

For the time window of 14 days which were investigated, degradation were evident for all samples. 2% PAC had a decrease of about 30 % at 300 RPM readings, 4% PAC decreased by

50 %, and the 4% PAC samples were barite was added did decrease with 75%. The stable window varied from 6 days in low concentration PAC, and did narrow down to 2 and 3 days for higher concentrations of PAC, respectively with and without barite. Mixtures containing XG decreased rapidly the first 48 hours in a level of 25 and 30 %, but did approximately stabilize at these levels. Those are high values, and it would be realistic to think that this will have an impact on a mud system. Simplifications were made for experiments conducted in this project, so they are not directly comparable with conditions in real mud systems, as multiple other additives also will be applied. On the other hand, it emphasizes the need for addition of stabilizers in order to prevent oxidation, scission and other mechanisms driving the degradation.

Measured gel strength did show correlated behaviour to viscosity with varying parameters. The composition of 2% PAC + 1 % XG was an exception, as it evolved a progressive gel after 72 hours. The only observation which matches the timeline for this, is the gas development in the samples. What is most prominent related to measured gel strength is comparing results in this work with (Saptharishi, 2016). In her results, gel strength was only observed for samples containing XG. In this experimental work, gel strength has been clearly observed for all compositions. The latter seems most reliable, especially with the numerous measurements involved. There is no doubt that something in the two procedures must have been quite different. This is also supported by the statistical evaluations which was presented. Errors were small for measured values obtained for this project, while they did vary more than what should be acceptable for the other sets of values. Procedural differences, as they are stated to be in the two representative projects mainly differs at mixing process. The speed we do know about, and a question raises in regard to how high temperatures arises while using the Waring blender. There may also have been different habits capable of influencing the results. Comparison with the dual DP, which should be fully reliable, revealed a huge difference to the manual result compared from this project. Temperature deviation is certainly one factor, but cannot be assigned the error in its fullness alone.

6 Conclusions

The main agendas for this project was to investigate for time and temperature dependency, and attempt to assess the reliability and certainty of experiments completed by Saptharishi (2016), by the use of statistical methods. This involved a large number of measurements, which became about 3000 manual readings when finishing the laboratory work.

Observing the results for temperature changes, it verified what already exists of knowledge about the increased temperature-decreased viscosity relationship. Altered properties due to heat exposure has been observed to be a slowly reversible process for both pure PAC and PAC+XG compositions. Gel strength follows the same behaviour as viscosity when tested against temperatures of 5°C, 25°C and 50°C.

Behaviour over time was highly affected by degradation, showing 4% PAC mixed with barite as the composition having the most significant decrease in viscosity and gel strength. 4% PAC without barite kept stable for 3 days, and 2% PAC was stable for 6 days, being the least affected of the mixtures investigated. XG mixtures had a significant drop in viscosity during the 48 first hours, but was the most stable throughout the rest of the time window. Gel strength was also linked to the time related viscosity behaviour. 2% PAC + 1% XG was an exception, as it did develop a progressive-like gel after 72 hours, even though the viscosity behaviour seemed to keep stable. Use of stabilizers seem to be important in order to constrain the degradation driving mechanisms.

Fann viscometers are precise, accurate and reliable instruments. Measurements conducted by such seems to be highly reliable when being subject to identical procedures and surrounding conditions. This was supported by the precision and accuracy analysis, as well as comparison of Fann viscometer tests at Sandsli and IPT together with Offite viscometer. The comparative plotting did show that deviations were extremely low with exact same temperature conditions, while the deviations quintupled when temperatures were differing.

Measurements in this work proved to be repeatable and reliable. The latter is concluded on the basis that both 2% PAC, 2% PAC +1% XG and 2% PAC +2%XG did show variation from mean values of less than 1%. These results was obtained using values from 300 RPM dial readings – after 24 hours – when the mixtures behaved stable, or stable-decreasing. 4% PAC and 4% PAC with barite had variations of 2.3 % and 1.1 % respectively, which is also satisfying. As the results seems to be reliable and with such small errors, it is difficult to address a need for increased sampling sizes. On the basis of the numeral material collected in this work, 95 % confidence intervals were created, with an expectation that Saptharishi's (2016) results should be found within. This was not the occasion for any of the four mixtures compared. Deviation was the largest for 2% PAC, which did vary with 28.2% to the mean values. For 4 % PAC and the samples containing XG, the deviation was between 8 and 10 %. These are not reliable values and the certainty has to be considered as poor. It must be emphasized that the mixing procedures was not the same, and could be the main reason for the differences. Comparing dual DP and results from this work did also differ with almost 20 %, knowing that temperature was not exactly equal. The main conclusion regarding reliability and uncertainty is that experiments being subject to the same procedures and conditions are highly repeatable and reliable, but the certainty cannot be accounted for unless a detailed standard have been implemented.

7 Potential Improvements and Way Forward

There are a few key points which is worth considering for further work on topics discussed in this report, as well as improving quality of similar work:

- Comparative analysis of mixing types and speeds, influence on viscosity. This would have been interesting in order to find out if the differences from the compared results in this work could be ascribed to this.
- Influence of heat exposure over short time vs hot rolling over time as done for more advanced aging tests.
- PH measurements in order to explain the chemical reactions happening in mixtures discussed. Also to possibly explain the partition and what driving mechanisms influencing this in PAC solutions.
- Use of salts, to examine what impact it has on the properties for PAC solutions, and what tolerance values exists.

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Appendix A Additional information

A.1 Fann 35 Viscometer calculations

Viscosity calculations based on dial readings are very simple for Newtonian fluids. At 300 RPM the viscosity can be read directly, while for the other speeds, they need to be converted related to the speed factor (SF) which evolves around the 300RPM speed. These relations are shown in Table 13: Correlation between RPM, shear rate and speed factorTable 13.

Table 13: Correlation between RPM, shear rate and speed factor

Fann viscometer RPM	3	6	100	200	300	600
Shear rate (1/s)	5	10	170	340	511	1021
Speed factor (SF)	100	50	3	1,5	1	0,5

The equation for calculating Newtonian viscosity:

$$\eta_N = SF * \theta * f * C \qquad \text{Equation 8}$$

Newtonian viscosity represented by η_N, θ the dial reading, *SF* the speed factor, *f* the spring factor and *C* the rotor-bob factor. In the setup used, the combination R1-B1-F1 gives *f* and *C* the value of 1, such that Equation 8 reduces to:

$$\eta_N = SF * \theta \qquad \text{Equation 9}$$

Appendix B Additional graphical Material

B.1 Temperature influence

Relation between temperature and measured shear stress for 2% PAC in Figure 39

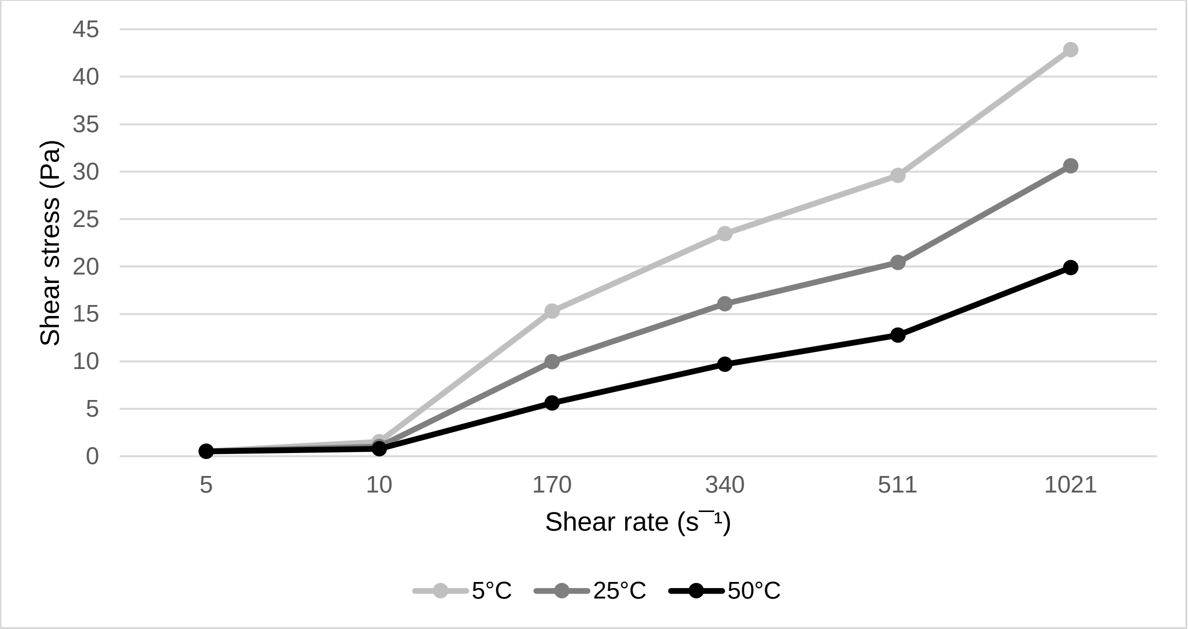

Figure 38: Temperature impact 2% PAC

The mixtures which were exposed to 50°C during mixing, had altered properties compared to samples only kept at 25°C. This is illustrated for 2% PAC and 2% PAC+2%XG in Figure 39 Figure 40.

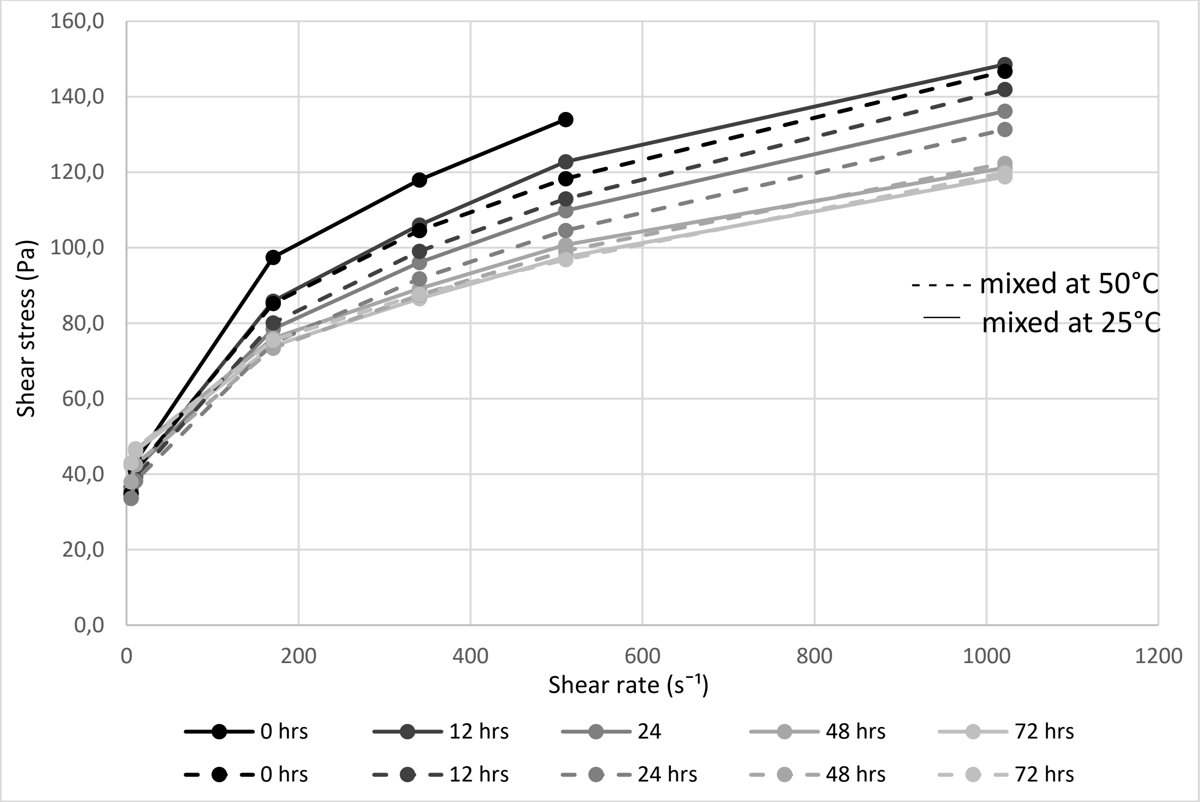

Figure 39: Temperature impact align over time for 2%PAC +2% XG

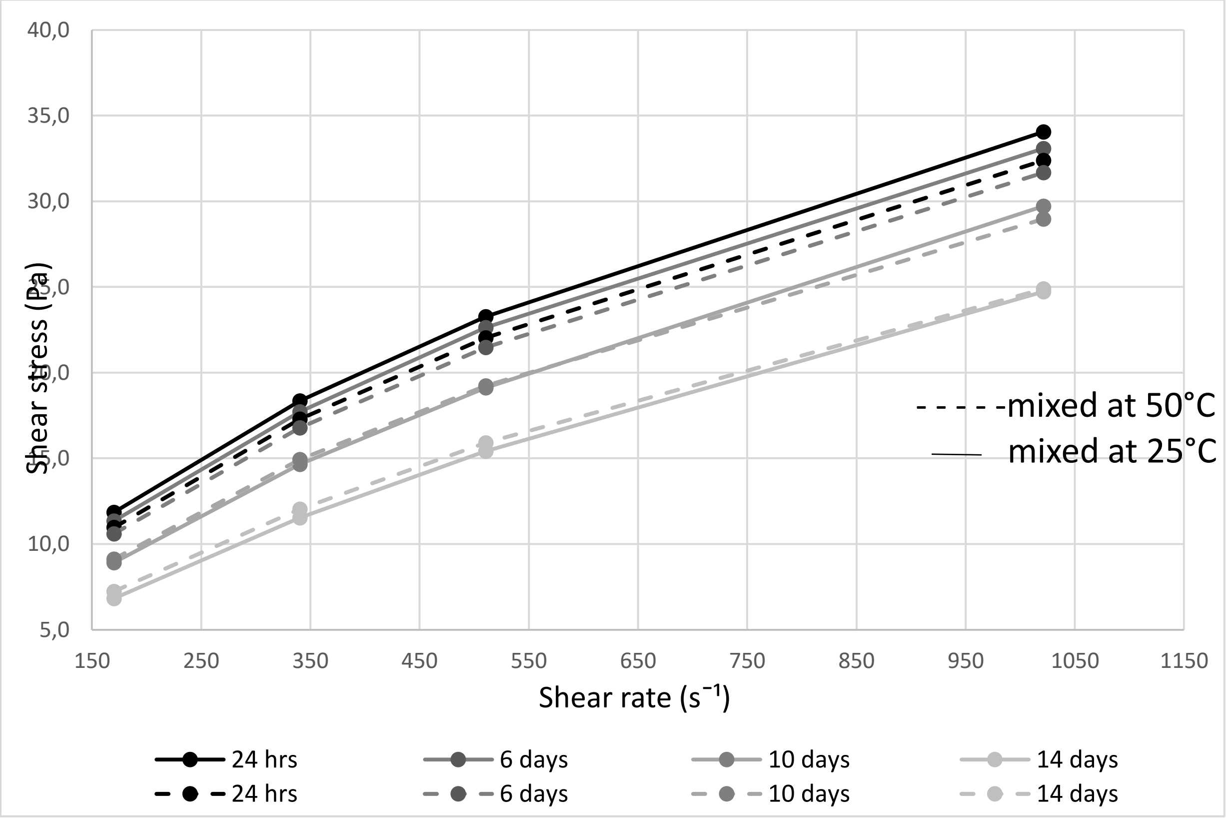

Figure 40: Temperature impact align over time 2%PAC

B.2 Time-related degradation

Figures 41-44 shows standard deviations related to the degradation observations.

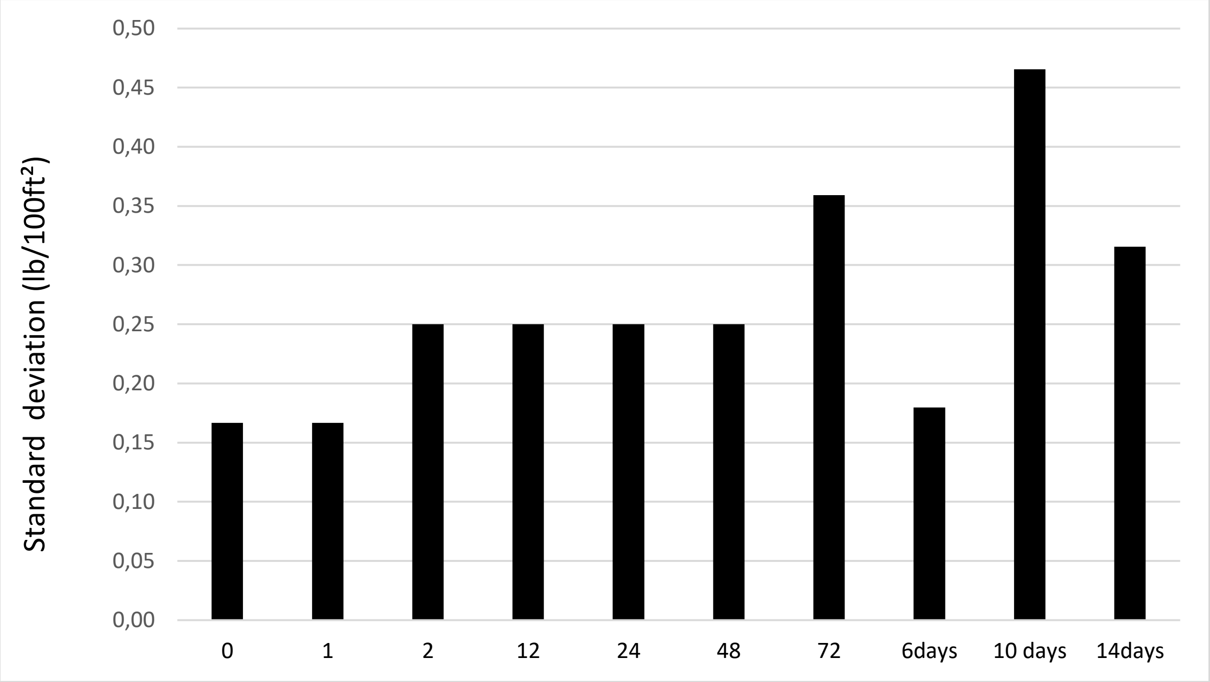

Figure 41: Average standard deviation for gel strength readings 2% PAC

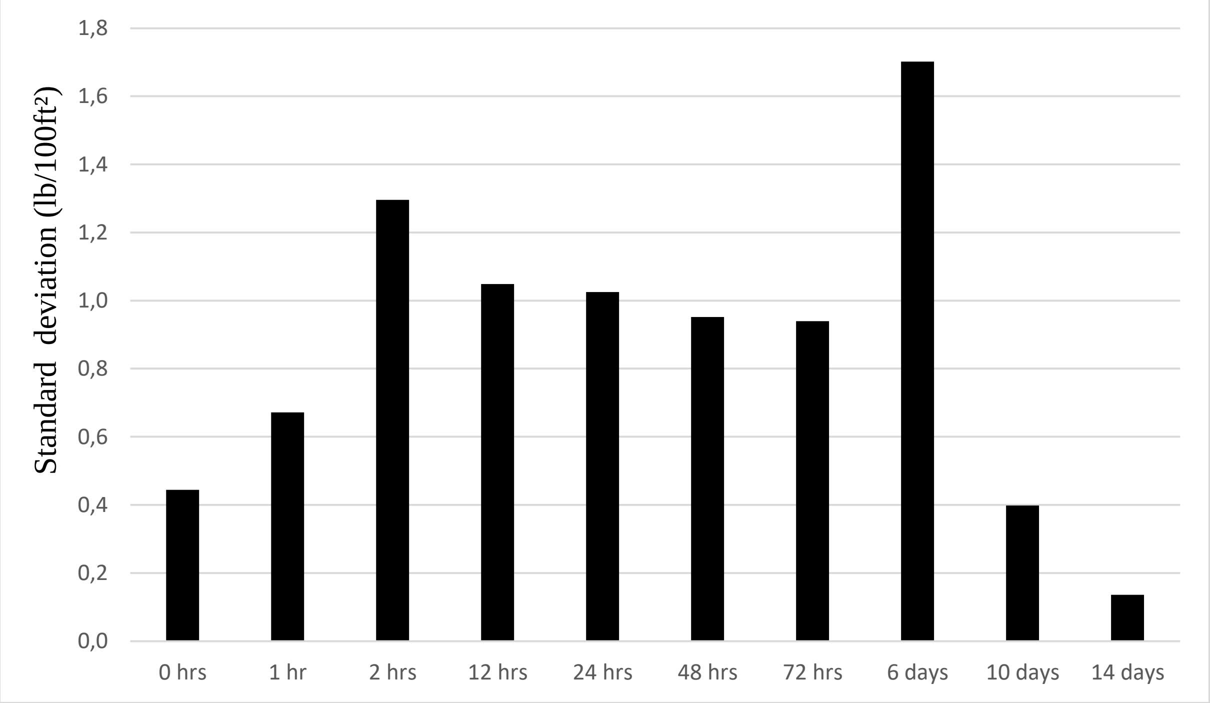

Figure 42: Average standard deviation for gel strength readings 4% PAC

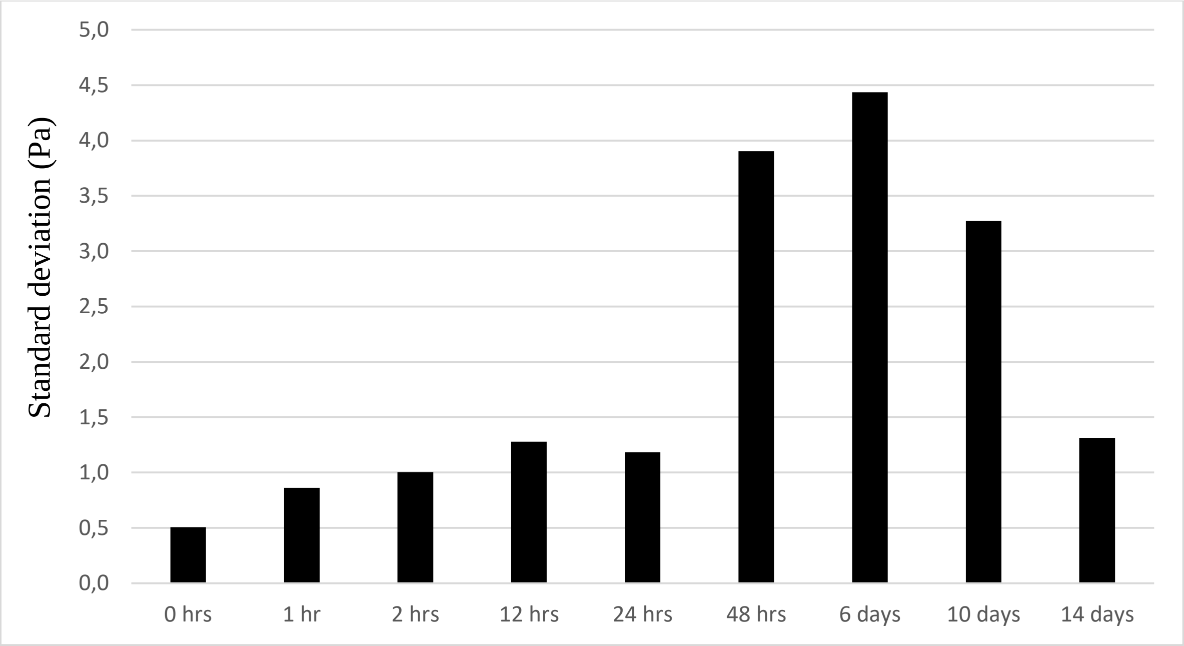

Figure 43: Standard deviation distributed for each time frequency 4% PAC+20 wt. % barite

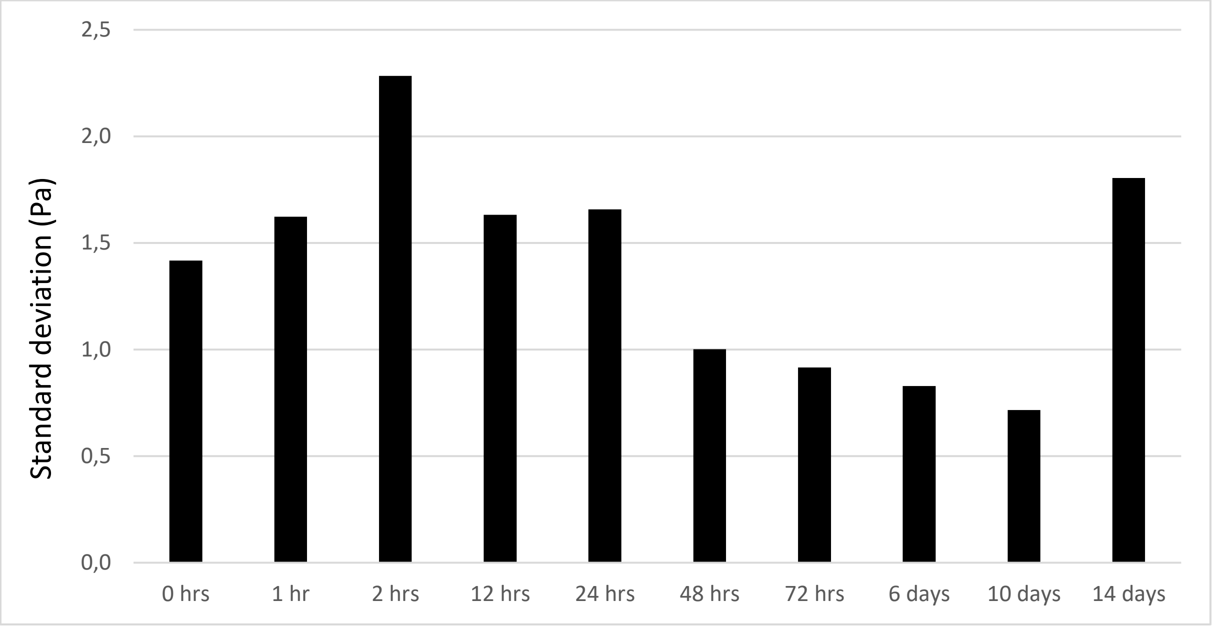

Figure 44:Standard deviation distributed for each time frequency 2% PAC+1%XG

During the aging time of XG samples, gas was found in the mixtures. Below there is a series of three images, showing how this gas evolved between 3 and 5 days for a mixture of 2% PAC + 1%XG. Samples containing 2% XG had an even more rapid and extensive behaviour.

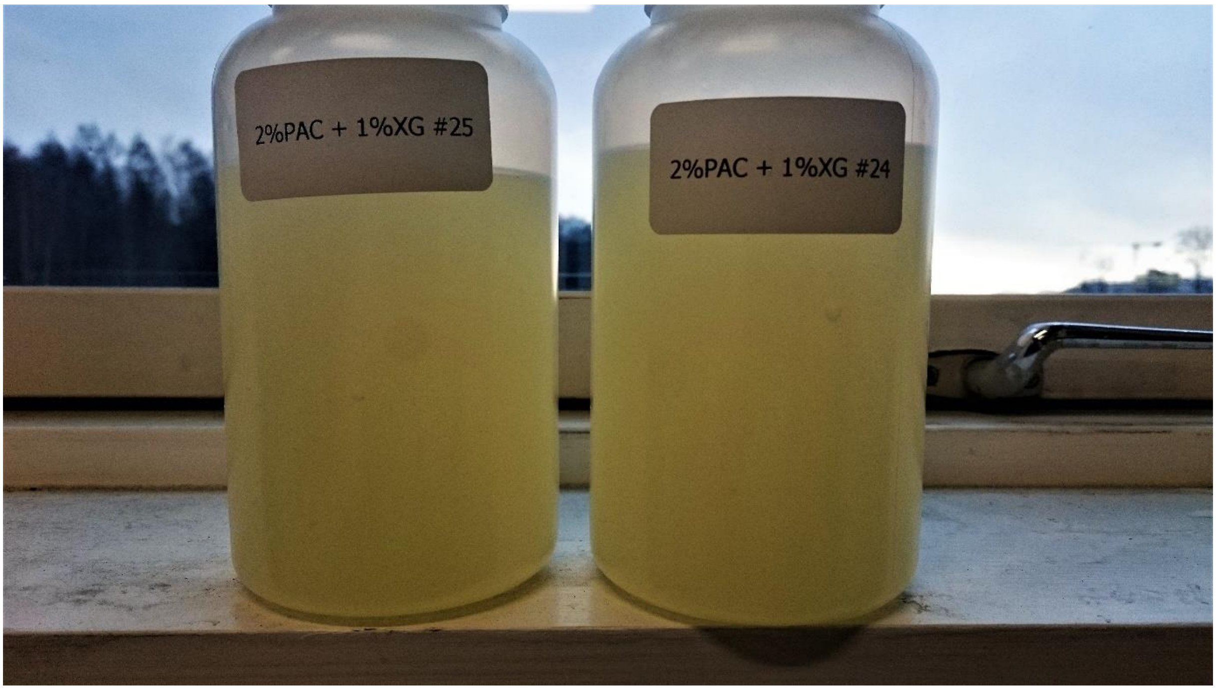

Figure 45: Gasification after 72 hours, 2% PAC+1%XG

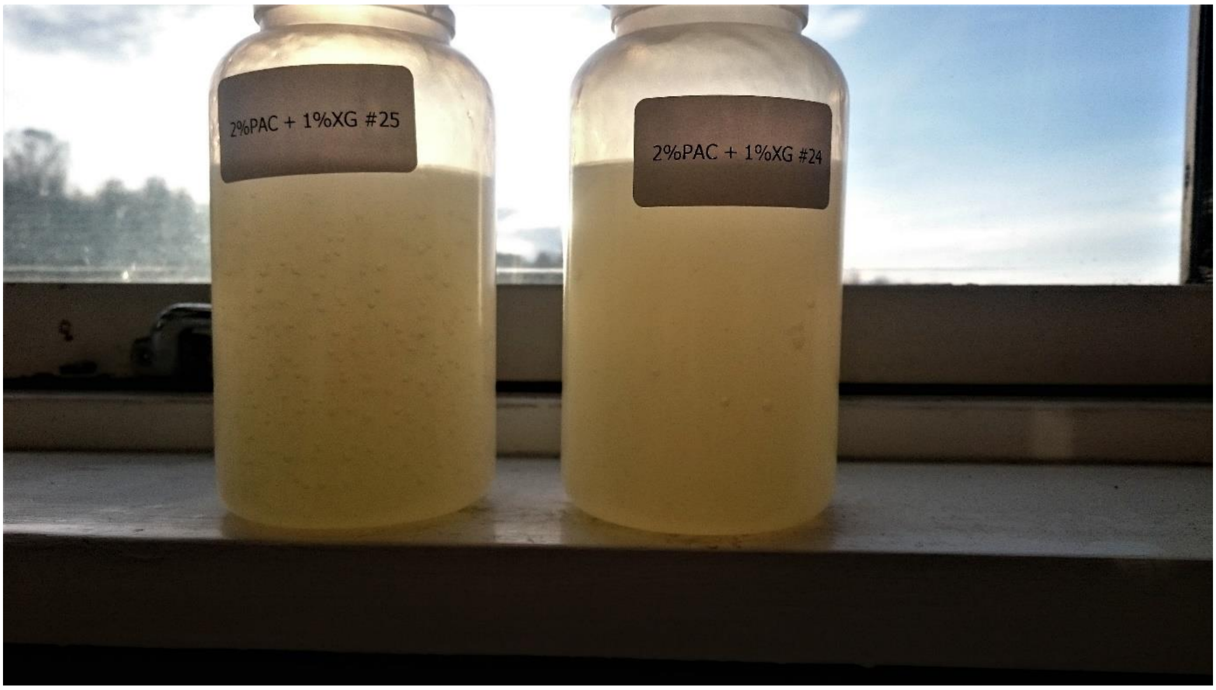

Figure 46: Gasification after 96 hours, 2% PAC+1%XG

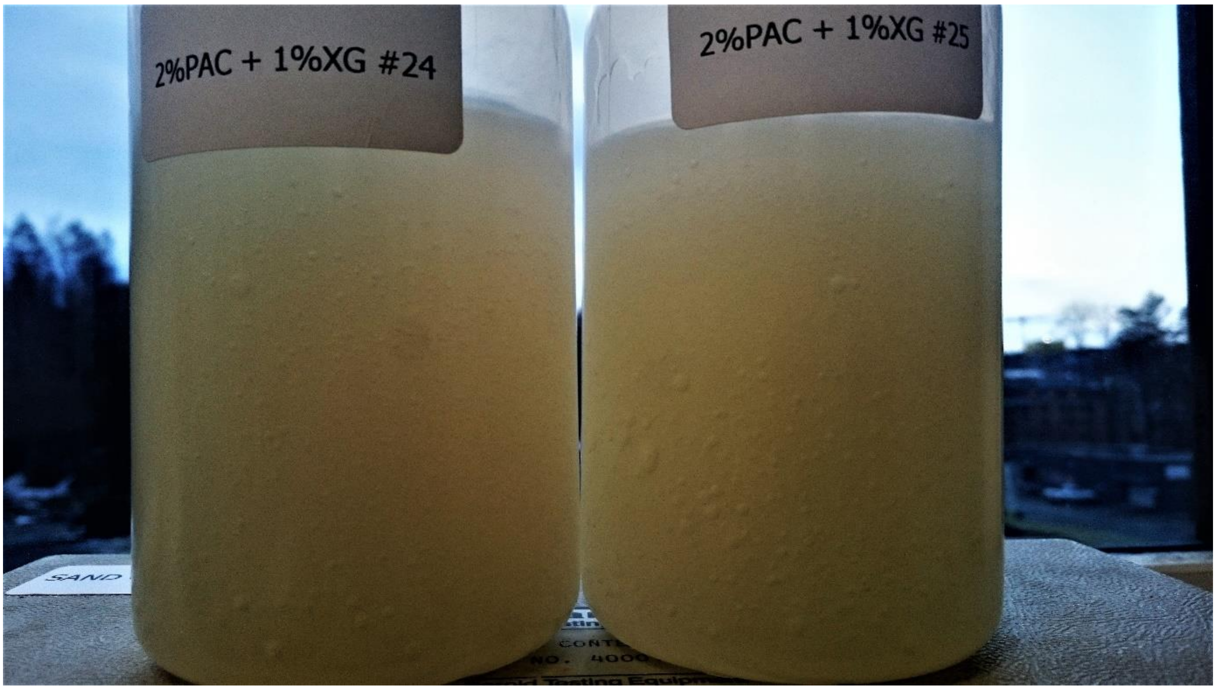

Figure 47: Gasification after 120 hours, 2% PAC+1%XG

Appendix C Complete measurement tabulations

C.1 Samples prepared at 50 degrees C

C.1.1 Dynamic rheological measurements

Table 14: 2% PAC mixed at 50°C, rheological measurements

Mixing Parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
2% PAC	14	0	1	2	20	31,5	40	59
2% PAC	14	1	1	2	22	34	43	63
2% PAC	14	2	1	2	22,5	35	44,5	65
2% PAC	14	12	1	2	22,5	34,5	44	64
2% PAC	14	24	1	2	22	34,5	43,5	64
2% PAC	14	48	1	2	22	34,5	44	64
2% PAC	14	72	1	2	22,5	35	44	65
2% PAC	14	6days	1	2	21	33	42	62,5
2% PAC	14	10 days	1	2	21	33,5	42,5	62,5
2% PAC	14	14days	1	2	20,5	32,5	41,5	61
2% PAC	15	0	1	2	19	30,5	39	58
2% PAC	15	1	1	2	20,5	32,5	41,5	61,5
2% PAC	15	2	1	2	21	33,5	43	63
2% PAC	15	12	1	2	21	33,5	43	63
2% PAC	15	24	1	2	21	33,5	43	63
2% PAC	15	48	1	2	21	33,5	42,5	63
2% PAC	15	72	1	2	21	33,5	42,5	63
2% PAC	15	6days	1	2	20	32,5	41	61
2% PAC	15	10 days	1	1,5	15	25,5	33,5	52
2% PAC	15	14days	1	1,5	10	18	24	40
2% PAC	16	0	1	2	20,5	32,5	41	60,5
2% PAC	16	1	1	2	22	34	43	63
2% PAC	16	2	1	2	22	34,5	43,5	64
2% PAC	16	12	1	2	22	34,5	43,5	64
2% PAC	16	24	1	2	22	34,5	43,5	64
2% PAC	16	48	1	2	22	34,5	43,5	64
2% PAC	16	72	1	2	21,5	34	43,5	64
2% PAC	16	6days	1	2	21,5	33,5	42,5	62,5
2% PAC	16	10 days	1	2	20,5	32,5	41,5	61

2% PAC	16	14days	1	1,5	15	24,5	32,5	51
2% PAC	17	0	1	2	19,5	31	40	59
2% PAC	17	1	1	2	20,5	33	42	61,5
2% PAC	17	2	1	2	21	33,5	43	63
2% PAC	17	12	1	2	21	33,5	43	63
2% PAC	17	24	1	2	21	33,5	43	63
2% PAC	17	48	1	2	21	33	42,5	62,5
2% PAC	17	72	1	2	21	33,5	43	63
2% PAC	17	6days	1	2	20,5	32,5	42	61,5
2% PAC	17	10 days	1	2	16	26,5	34,5	53,5
2% PAC	17	14days	1	1,5	12	20,5	28	45
2% PAC	4	0	1	2	20	31,5	40,5	59,5
2% PAC	4	1	1	2	21	33	42,5	62,5
2% PAC	4	2	1	2	21,5	33,5	43,5	63,5
2% PAC	4	12	1	2	22	33,5	43	63,5
2% PAC	4	24	1	2	21,5	33,5	43	63,5
2% PAC	4	48	1	2	21,5	33,5	43,5	63
2% PAC	4	72	1	2	21,5	33	43	63
2% PAC	4	6d	1	2	21	33	43	63
2% PAC	4	10 days	1	2	17	28,5	36,5	55
2% PAC	4	14days	1,5	2,5	13,5	22,5	30	47

Table 15: 4% PAC mixed at 50°C, rheological measurements

Mixing Parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
4% PAC	12	0 hrs	5,5	9	64	100,5	130	192
4% PAC	12	1 hr	5,5	10	66	104	133	198
4% PAC	12	2 hrs	6	10	65,5	104	132,5	197
4% PAC	12	12 hrs	6	10	65	102	132	195,5
4% PAC	12	24 hrs	6	10	67	105	136	200
4% PAC	12	48 hrs	6	9,5	67,5	105,5	136	200,5
4% PAC	12	72 hrs	5,5	10	65,5	103	133,5	197,5
4% PAC	12	6 days	4	7	57	90	116	172
4% PAC	12	10 days	2	2	25	43,5	60	96
4% PAC	12	14 days	1	2	20	36,5	48,5	80
4% PAC	13	0 hrs	5	9,5	62,5	99,5	129,5	193
4% PAC	13	1 hr	5,5	9,5	63	100	131,5	195
4% PAC	13	2 hrs	6	10	63,5	101	132	196

4% PAC	13	12 hrs	6,5	10,5	64	101,5	132,5	196,5
4% PAC	13	24 hrs	6	10,5	64,5	102,5	133,5	198
4% PAC	13	48 hrs	6	10	65	103	134,5	198,5
4% PAC	13	72 hrs	6	10,5	64,5	102	133	198
4% PAC	13	6 days	5	9	62	98,5	129	190,5
4% PAC	13	10 days	2,5	4	38,5	63	83	128,5
4% PAC	13	14 days	1	2,5	28	47	64	101
4% PAC	20	0 hrs	5,5	10	69,5	108	138	202
4% PAC	20	1 hr	6,5	11	71,5	111,5	142	208
4% PAC	20	2 hrs	6,5	11	74	115	146	212
4% PAC	20	12 hrs	7	12	77	118	150	215
4% PAC	20	24 hrs	7	11	73	113,5	145,5	212
4% PAC	20	48 hrs	6,5	11,5	74,5	116	148	214,5
4% PAC	20	72 hrs	7	12	75,5	116,5	147,5	215
4% PAC	20	6 days	5,5	9,5	69	107	137	199,5
4% PAC	20	10 days	2	3,5	37	61,5	81,5	127,5
4% PAC	20	14 days	1	2,5	27	46,5	62,5	100
4% PAC	21	0 hrs	5,5	10,5	68	107	138,5	203,5
4% PAC	21	1 hr	6,5	11	70,5	110,5	142,5	208
4% PAC	21	2 hrs	6,5	11,5	73	116	147	212,5
4% PAC	21	12 hrs	7	13	81	125	158	222
4% PAC	21	24 hrs	6,5	10,5	68,5	108	140	206
4% PAC	21	48 hrs	6	10,5	70	110	142,5	207,5
4% PAC	21	72 hrs	5,5	10	69	108,5	140	205
4% PAC	21	6 days	2	3	38	63	84	130,5
4% PAC	21	10 days	1	2	26	45	61	98
4% PAC	21	14 days	1	2	21	37	50	82

Table 16: 2%PAC+2%XG mixed at 50°C, rheological measurements

Mixing Parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
2%PAC + 2%XG	10	0	68,5	80,5	166,5	204	231	287,5
2%PAC + 2%XG	10	1	67	79	167	206	234	292,5
2%PAC + 2%XG	10	2	67,5	79	167	207	233,5	293
2%PAC + 2%XG	10	10	66,5	77	157,5	194,5	221,5	278

2%PAC + 2%XG	10	24	66,5	75	146,5	179,5	204	255,5
2%PAC + 2%XG	10	48	75	84	144	171	195	240
2%PAC + 2%XG	10	72	85,5	93	148,5	170	196	237
2%PAC + 2%XG	11	0	68,5	80,5	168	206	233	288
2%PAC + 2%XG	11	1	66,5	78,5	167,5	206,5	234,5	292,5
2%PAC + 2%XG	11	2	67	79	167,5	207	234	291
2%PAC + 2%XG	11	10	65,5	76,5	156,5	194	221,5	278,5
2%PAC + 2%XG	11	24	65,5	75	147	180,5	206	259,5
2%PAC + 2%XG	11	48	74	83	144	172	194	240
2%PAC + 2%XG	11	72	83,5	90,5	147,5	173,5	184	233

Table 17: Various trial samples mixed at 50°C, rheological measurements

Temp 50°C	Mixing Parameters			Fann viscometer RPM					
Mixing type	Sample number	test temp	rest time	3	6	100	200	300	600
4% PAC	1.1	25	1	4	6,5	57	91	120,5	182
4% PAC	1.1	25	24	3	5,5	41	86	109	167
4% PAC	1.1	25	7d	2,5	4	31	52	69,5	110
4% PAC	1.2	25	1	3,5	6,5	53,5	89	116	175,5
4% PAC	1.2	5	24	8	13	84	134	174,5	258
4% PAC	1.3	50	3	2,5	5	47	73,5	94	142
4% PAC	5.1	25	48	18	29	143,5	181,5	207	251
4% PAC	5.2	25	48	0,5	1,5	17	31,5	44	78
4% PAC	1.4	50	1	2,5	4	37	60	79	122
4% PAC	1.4	5	24	8	13	82	130	170	254
4% PAC	1.4	25	26	4,5	8	59	94	123,5	186
2%PAC	2.1	25	1	1	2	20,5	33	42,5	62,5
2%PAC	2.1	25	6d	1,5	2,5	13,5	22,5	30	47
2%PAC	8	50	0	2	3	10,5	16,5	22	35
2%PAC	8	5	24	1,5	3	29	43,5	55	80
2%PAC	8	25	48	1	2	19	30	38,5	57,5
2%PAC	9	50	0	1	1,5	11	19	25	39
2%PAC	9			1	1,5	11	19,5	26	40
2%PAC	9	5	24	1	3	30	46	58	84
2%PAC	9	25	26	1	2	19,5	31,5	40	60
4%PAC+2%XG	6	50	0	60	74	222	301	300+	300+
4%PAC+2%XG	6	25	2	60	77	252	300+	300+	300+
4%PAC+1%XG	4.1	25	0	31	41	162	235	289	300+

2%PAC+2%XG	7	50	0	69	79	159	193	218	269
2%PAC+2%XG	7			69,5	80	163	197	221	272
2%PAC+2%XG	7	5	24	67	79	174	218	251	300+
2%PAC+2%XG	7	14		66	78	169	211	242	300+
2%PAC+2%XG	7	25	48	76	86	151,5	183	202	252
2%PAC+2%XG	7	25	20days	96	103	151,5	173	196	229
2%PAC+1%XG	9	25	0	31,5	39	102,5	129	147	185,5
2%PAC+1%XG	9	25	20days	32	35	58	69	77	93,5

C.1.2 Gel strength

Table 18: 2% PAC, mixed at 50°C, gel strength

Mixing Parameters			Gel Strength		
Mixture	Sample #	rest time	10sec	1min	10min
2% PAC	14	0	2,5	3	2,5
2% PAC	14	1	3,5	3	3
2% PAC	14	2	3	3	3
2% PAC	14	12	3	3	2,5
2% PAC	14	24	3	3	3
2% PAC	14	48	3	3	3
2% PAC	14	72	3	3	2,5
2% PAC	14	6days	2,5	3	2,5
2% PAC	14	10 days	2,5	3	2,5
2% PAC	14	14days	2,5	2,5	2,5
2% PAC	15	0	2,5	2,5	2,5
2% PAC	15	1	2,5	3	2,5
2% PAC	15	2	3	3	2,5
2% PAC	15	12	2,5	2,5	3
2% PAC	15	24	2	3	2,5
2% PAC	15	48	2,5	3	2,5
2% PAC	15	72	2,5	2,5	2,5
2% PAC	15	6days	2,5	2,5	2,5
2% PAC	15	10 days	2	2,5	2
2% PAC	15	14days	2	2	2
2% PAC	16	0	2,5	2,5	3
2% PAC	16	1	2,5	3	3
2% PAC	16	2	2,5	2,5	2,5

2% PAC	16	12	2,5	2,5	2,5
2% PAC	16	24	3	2,5	2,5
2% PAC	16	48	3	2,5	2,5
2% PAC	16	72	3	2,5	2,5
2% PAC	16	6days	3	2,5	2,5
2% PAC	16	10 days	3	2,5	2,5
2% PAC	16	14days	2	1,5	1,5
2% PAC	17	0	2,5	3	2,5
2% PAC	17	1	2,5	3	2,5
2% PAC	17	2	3	3	2,5
2% PAC	17	12	3	3	3
2% PAC	17	24	2,5	2,5	2,5
2% PAC	17	48	2,5	2,5	2,5
2% PAC	17	72	2,5	2,5	2,5
2% PAC	17	6days	2,5	2,5	2,5
2% PAC	17	10 days	2,5	2,5	2,5
2% PAC	17	14days	1,5	1	1
2% PAC	4	0	2,5	2,5	2,5
2% PAC	4	1	2,5	2,5	2,5
2% PAC	4	2	2,5	3	2,5
2% PAC	4	12	2,5	3	2,5
2% PAC	4	24	2,5	3	2,5
2% PAC	4	48	2,5	3	2,5
2% PAC	4	72	2,5	2,5	2,5
2% PAC	4	6d	2,5	2,5	2,5
2% PAC	4	10 days	2,5	2,5	2,5
2% PAC	4	14days	2	2	1,5

Table 19: 4 % PAC, mixed at 50°C, gel strength

Mixing Parameters			Gel Strength		
Mixture	Sample #	rest time	10sec	1min	10min
4% PAC	12	0 hrs	10	10	8
4% PAC	12	1 hr	11	11	9
4% PAC	12	2 hrs	11	11	9
4% PAC	12	12 hrs	10,5	11	9
4% PAC	12	24 hrs	11	11	9
4% PAC	12	48 hrs	11	11	8

4% PAC	12	72 hrs	11	11	9
4% PAC	12	6 days	3	4	3
4% PAC	12	10 days	2	2,5	2
4% PAC	12	14 days	2	2	2
4% PAC	13	0 hrs	9,5	9,5	7
4% PAC	13	1 hr	10	10	7,5
4% PAC	13	2 hrs	10	10,5	8
4% PAC	13	12 hrs	10,5	10,5	8,5
4% PAC	13	24 hrs	10,5	10	8
4% PAC	13	48 hrs	11	10,5	8
4% PAC	13	72 hrs	11	10,5	9
4% PAC	13	6 days	9	9	6
4% PAC	13	10 days	4	4,5	3,5
4% PAC	13	14 days	2	2,5	2
4% PAC	20	0 hrs	12,5	12	8
4% PAC	20	1 hr	12,5	12	8
4% PAC	20	2 hrs	13	12,5	8,5
4% PAC	20	12 hrs	14	13,5	12
4% PAC	20	24 hrs	13	12,5	10,5
4% PAC	20	48 hrs	13	12,5	10
4% PAC	20	72 hrs	13,5	13	10
4% PAC	20	6 days	11,5	11	9
4% PAC	20	10 days	3	4	3
4% PAC	20	14 days	2,5	2,5	2
4% PAC	21	0 hrs	12	12	8
4% PAC	21	1 hr	12	12	8
4% PAC	21	2 hrs	12,5	12,5	8
4% PAC	21	12 hrs	14,5	13,5	12
4% PAC	21	24 hrs	12	11,5	10
4% PAC	21	48 hrs	12	12	9,5
4% PAC	21	72 hrs	11,5	11,5	9
4% PAC	21	6 days	3	4	3
4% PAC	21	10 days	2	2,5	2
4% PAC	21	14 days	2	2,5	2

Table 20: Various trial samples mixed at 50°C, gel strength

Temp 50°C	Mixing Parameters			Gel Strength						
Mixing type	Sample number	test temp	resting time	10 sec	1 min	10 min	30 min	1 hr	1.5 hr	24 hrs

4% PAC	1.1	25	1	8	8	7	4	3,5		
4% PAC	1.1	25	24	7	7,5	6	4	3,5		7,5
4% PAC	1.1	25	7d	3,5	2,5	2,5	2	2		
4% PAC	1.2	25	1	8	8	7	4	3,5	2,5	
4% PAC	1.2	5	24	15	14,5	13	7	7		25
4% PAC	1.3	50	3	6,5	7	4	3	2		
4% PAC	5.1	25	48	32	31	30	31,5	32		
4% PAC	5.2	25	48	1	1	0,5	0,5			
4% PAC	1.4	50	1	5	5	3	4	4		
4% PAC	1.4	5	24	13	13	7	6	8		20
4% PAC	1.4	25	26	8,5	8,5	7	4	4		
2%PAC	2.1	25	1	2,5	2,5	1	0,5	0,5		
2%PAC	2.1	25	6d	2	2	1,5	1,5	1		
2%PAC	8	50	0	4	3,5	3	1,5	1		
2%PAC	8	5	24	3	3	2,5	2	1		4,5
2%PAC	8	25	48	2	2,5	3	2	1		
2%PAC	9	50	0	3	2	2	1,5	1		
2%PAC	9									
2%PAC	9	5	24	3,5	3,5	2,5	2	2		3
2%PAC	9	25	26	2,5	2	1	1	0,5		
4%PAC+2%XG	6	50	0	66	64	63	59	57		
4%PAC+2%XG	6	25	2	73	70	69,5	67	64		
4%PAC+1%XG	4.1	25	0	38	38	38	36	34		
2%PAC+2%XG	7	50	0	68	68	71	72	73,5		
2%PAC+2%XG	7									
2%PAC+2%XG	7	5	24	66	65	65	65	67		69
2%PAC+2%XG	7	14								
2%PAC+2%XG	7	25	48	76	76	75				
2%PAC+2%XG	7	25	20days	93	95					
2%PAC+1%XG	9	25	0	35	34,5	35,5	36	36,5		
2%PAC+1%XG	9	25	20days	32	34					

C.2 Samples prepared at 25 degrees C

C.2.1 Dynamic rheological measurements

Table 21: 2 % PAC mixed at 25°C, rheological measurements

Mixing Parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
2% PAC	18	0	1	2	22	34,5	44	64
2% PAC	18	1	1	2	23	35,5	45	65,5
2% PAC	18	2	1	2	23,5	36	45,5	66
2% PAC	18	12	1	2	23,5	36	45,5	66
2% PAC	18	24	1	2	23,5	36,5	46	67
2% PAC	18	48	1	2	23,5	36,5	46	66,5
2% PAC	18	72	1	2,5	23,5	36	46	66,5
2% PAC	18	6days	1	2	23	35,5	45	65,5
2% PAC	18	10 days	1	2	19	30,5	39,5	59,5
2% PAC	18	14days	0,5	1	14,5	24,5	32,5	50,5
2% PAC	19	0	1	2	21	33,5	43	63
2% PAC	19	1	1	2	22	35	44,5	65
2% PAC	19	2	1	2	23	35,5	45	66
2% PAC	19	12	1	2	23	36	45	66
2% PAC	19	24	1	2	22,5	35,5	45	66
2% PAC	19	48	1	2	22	35	44,5	65
2% PAC	19	72	1,5	2,5	22,5	35,5	45	65,5
2% PAC	19	6days	1	2	21,5	34	43,5	64
2% PAC	19	10 days	1	1,5	16	27	35,5	54,5
2% PAC	19	14days	1	1,5	12	21	28	45,5
2% PAC	42	0	1	2	21,5	34	43,5	63,5
2% PAC	42	1	1	2	22	35	44,5	65
2% PAC	42	2	1	2	23	35,5	45,5	66
2% PAC	42	12	1	2	23	36	46	66,5
2% PAC	42	24	1	2	23,5	36	46	66,5
2% PAC	42	48	1	2	23	35,5	45,5	66,5
2% PAC	42	72	1	2,5	23	35,5	45,5	66,5
2% PAC	42	6days	1	2	22	35	45	65
2% PAC	42	10 days	1	2	18	28,5	37	57
2% PAC	42	14days	1	1	13,5	23	30	48
2% PAC	43	0	1	2	22	34	43,5	64
2% PAC	43	1	1	2	22,5	35	44,5	65

2% PAC	43	2	1	2	23	35,5	45	66
2% PAC	43	12	1	2	23,5	36	45,5	66
2% PAC	43	24	1	2	23,5	36	45,5	67,5
2% PAC	43	48	1	2	23	36	45	66
2% PAC	43	72	1	2	23	35,5	45	66
2% PAC	43	6days	1	2	22,5	34,5	44	65
2% PAC	43	10 days	1	1,5	17	29	38	62
2% PAC	43	14days	1	1	13,5	22	30,5	50

Table 22: 4 % PAC mixed at 25°C, rheological measurements

Mixing Parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
4% PAC	22	0 hrs	4,5	8	56,5	89,5	106,5	173,5
4% PAC	22	1 hr	5	8,5	58,5	92	111	179
4% PAC	22	2 hrs	5	9	62	97,5	127	188,5
4% PAC	22	12 hrs	6	10	64	101	131,5	195
4% PAC	22	24 hrs	6	9,5	63	99,5	130	193,5
4% PAC	22	48 hrs	6	10	65	103	133,5	197
4% PAC	22	72 hrs	6	10	67	106	136	199,5
4% PAC	22	6 days	3	6	52,5	84	109,5	164
4% PAC	22	10 days	1,5	2,5	30,5	52	69,5	110,5
4% PAC	22	14 days	1	2	24,5	42	57	92,5
4% PAC	23	0 hrs	4,5	8	54,5	88	111,5	171,5
4% PAC	23	1 hr	5	8	57	92	117	177
4% PAC	23	2 hrs	5	8	57,5	93	118	182
4% PAC	23	12 hrs	5,5	9	58	95	120	188
4% PAC	23	24 hrs	5,5	8,5	58	93	119,5	187,5
4% PAC	23	48 hrs	5	8,5	57	92	120	188
4% PAC	23	72 hrs	4	8	57	92	119	189
4% PAC	23	6 days	2,5	5	57	91	78	167
4% PAC	23	10 days	1,5	3	31	52	69	105,5
4% PAC	23	14 days	1	2	22	39	54	86,5
4% PAC	28	0 hrs	4,5	8	54,5	87,5	114	170
4% PAC	28	1 hr	5	8	57,5	91,5	119	176
4% PAC	28	2 hrs	4,5	8	57,5	91,5	119	177,5
4% PAC	28	12 hrs	5	9	58,5	93	121	179,5
4% PAC	28	24 hrs	5	8,5	57,5	91,5	119,5	178
4% PAC	28	48 hrs	4,5	8,5	57	91,5	119	177
4% PAC	28	72 hrs	4,5	8	57	91	118	175

4% PAC	28	6 days	4,5	7,5	57,5	91	117,5	174
4% PAC	28	10 days	1,5	3	32	54	72	114
4% PAC	28	14 days	1	2	24	41	55	90
4% PAC	29	0 hrs	5	8	53	85	111	169
4% PAC	29	1 hr	4	8	54	87	114	172
4% PAC	29	2 hrs	5	8	55	89	116	175
4% PAC	29	12 hrs	5	8	56	90,5	118	178
4% PAC	29	24 hrs	5	8	55	89	116	175
4% PAC	29	48 hrs	4,5	8	56,5	91	118,5	178
4% PAC	29	72 hrs	4,5	8	55,5	89	116,5	175,5
4% PAC	29	6 days	2,5	5	47,5	77	100	151,5
4% PAC	29	10 days	2	3	33	56	75	118
4% PAC	29	14 days	1	2	23,5	40,5	55	88,5
4% PAC	48	0 hrs	4,5	8	55,5	89	109	173
4% PAC	48	1 hr	5	8,5	58	92	114,5	178
4% PAC	48	2 hrs	5,5	9,5	61	98	126	192
4% PAC	48	12 hrs	6	10,5	62,5	100	127,5	197
4% PAC	48	24 hrs	5,5	10	62,5	100,5	128	196
4% PAC	48	48 hrs	5,5	10	62,5	100,5	129	196
4% PAC	48	72 hrs	6	9,5	63	101	132,5	198,5
4% PAC	48	6 days	2	4	47	79	87	150,5
4% PAC	48	10 days	1	2	26	48	63	97
4% PAC	48	14 days	1	2	20	37	47	79
4% PAC	49	0 hrs	4,5	8	56	89,5	106,5	174,5
4% PAC	49	1 hr	5	8,5	58	92,5	112	180
4% PAC	49	2 hrs	5,5	9,5	63	100	130	193,5
4% PAC	49	12 hrs	6	10,5	65	103,5	134,5	199,5
4% PAC	49	24 hrs	6	10	65	103	134	199
4% PAC	49	48 hrs	6	10	65,5	103,5	135	199,5
4% PAC	49	72 hrs	6	10	66	104	136,5	200,5
4% PAC	49	6 days	2	4	42	70	92	142
4% PAC	49	10 days	1	2	24	42	57	92,5
4% PAC	49	14 days	1	2	18	32	43,5	72,5

Table 23: 2 % PAC + 1% XG mixed at 25°C, rheological measurements

Mixing parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
2%PAC + 1%XG	24	0 hrs	30	36	99,5	132,5	147	188
2%PAC + 1%XG	24	1 hr	33,5	41,5	107	134	152,5	192,5
2%PAC + 1%XG	24	2 hrs	37	45	111,5	136	157	196
2%PAC + 1%XG	24	10 hrs	33	40,5	103,5	129,5	149	191
2%PAC + 1%XG	24	24 hrs	31	38	95,5	121	140	179
2%PAC + 1%XG	24	48 hrs	30	34,5	72,5	91,5	106	135
2%PAC + 1%XG	24	72 hrs	32,5	36,5	68,5	84	96	121
2%PAC + 1%XG	24	96 hrs	34	37,5	67	81	91,5	114,5
2%PAC + 1%XG	24	6 days	34	37,5	64,5	77,5	87	109,5
2%PAC + 1%XG	24	10 days	34	37,5	63,5	76	86	105,5
2%PAC + 1%XG	24	14 days	34,5	37,5	63	74,5	83	101,5
2%PAC + 1%XG	25	0 hrs	28	35	99	126	146	188
2%PAC + 1%XG	25	1 hr	29,5	37	102,5	131	151,5	192,5
2%PAC + 1%XG	25	2 hrs	31	39	106	135	155	195
2%PAC + 1%XG	25	10 hrs	29,5	37	98	124,5	143,5	183
2%PAC + 1%XG	25	24 hrs	27,5	34,5	92	118	137,5	177,5
2%PAC + 1%XG	25	48 hrs	26	30,5	68,5	88	102	130
2%PAC + 1%XG	25	72 hrs	28,5	32,5	64	80	93,5	117,5
2%PAC + 1%XG	25	96 hrs	30	33	61	75	87	106,5
2%PAC + 1%XG	25	6 days	30,5	34	61	75	86	108
2%PAC + 1%XG	25	10 days	32	35	60	73	83	101
2%PAC + 1%XG	25	14 days	29	32	54	65	73	89
2%PAC + 1%XG	30	0 hrs	29,5	37	100	126,5	145	185
2%PAC + 1%XG	30	1 hr	30	37	100,5	127,5	146	190
2%PAC + 1%XG	30	2 hrs	29,5	37	100	127,5	146	188,5
2%PAC + 1%XG	30	10 hrs	29,5	36,5	97	123,5	142,5	182
2%PAC + 1%XG	30	24 hrs	28	34,5	90,5	111	134	172,5
2%PAC + 1%XG	30	48 hrs	26	31	68	86,5	100	129
2%PAC + 1%XG	30	72 hrs	31	35	65,5	80,5	92	116,5
2%PAC + 1%XG	30	96 hrs	32	35,5	64	78	89	111,5
2%PAC + 1%XG	30	7 days	32,5	35,5	61	74	83	102,5
2%PAC + 1%XG	30	10 days	32	35	59	70	79	97
2%PAC + 1%XG	30	14 days	33	36	59	70	78,5	95
2%PAC + 1%XG	31	0 hrs	28	35	98,5	126	145,5	188
2%PAC + 1%XG	31	1 hr	28	36	99	126,5	146,5	188,5
2%PAC + 1%XG	31	2 hrs	28,5	35,5	98,5	125,5	146	185
2%PAC + 1%XG	31	10 hrs	28,5	35,5	97	123	142	183

2%PAC + 1%XG	31	24 hrs	26	33	88,5	114	133	170,5
2%PAC + 1%XG	31	48 hrs	25,5	30	67,5	86,5	101	128,5
2%PAC + 1%XG	31	72 hrs	30	34	64	80	92	115
2%PAC + 1%XG	31	96 hrs	31,5	35	63,5	78	89	108
2%PAC + 1%XG	31	7 days	33	36	61,5	75	85	102
2%PAC + 1%XG	31	10 days	30	33	55	66	74,5	92
2%PAC + 1%XG	31	14 days	33	35	59,5	71	79	94,5
2%PAC + 1%XG	32	0 hrs	29,5	36,5	97,5	124	143	186,5
2%PAC + 1%XG	32	1 hr	30	37	100	127	146	186
2%PAC + 1%XG	32	2 hrs	31	39	102,5	130,5	150	189
2%PAC + 1%XG	32	10 hrs	29	36	95	121,5	138,5	178
2%PAC + 1%XG	32	24 hrs	27	33	87	111,5	129,5	166,5
2%PAC + 1%XG	32	48 hrs	27	31,5	69	87	101	129,5
2%PAC + 1%XG	32	72 hrs	31	34,5	64,5	79	90,5	114
2%PAC + 1%XG	32	96 hrs	33	36	63,5	77	87,5	110
2%PAC + 1%XG	32	7 days	33	36	61,5	74	84	103
2%PAC + 1%XG	32	10 days	33	31,5	60,5	71,5	79	96,5
2%PAC + 1%XG	32	14 days	32,5	35,5	59	69,5	78	94
2%PAC + 1%XG	33	0 hrs	28	35,5	98	125,5	145	187
2%PAC + 1%XG	33	1 hr	28,5	36	99	126,5	146,5	189,5
2%PAC + 1%XG	33	2 hrs	29	36,5	100	127	147,5	189
2%PAC + 1%XG	33	10 hrs	28	35	95	121,5	141	179,5
2%PAC + 1%XG	33	24 hrs	26	32	86,5	111,5	130,5	168
2%PAC + 1%XG	33	48 hrs	26	30,5	67	85	99,5	126,5
2%PAC + 1%XG	33	72 hrs	30,5	34,5	64	79	91	111
2%PAC + 1%XG	33	96 hrs	32	35	63	77	88	108,5
2%PAC + 1%XG	33	7 days	33	36	62	75	85	103
2%PAC + 1%XG	33	10 days	30	32,5	55	66	75	87
2%PAC + 1%XG	33	14 days	33	36	59	70,5	80	92
2%PAC + 1%XG	34	0 hrs	29	36	96	121,5	140,5	180
2%PAC + 1%XG	34	1 hr	29,5	36,5	97,5	124	143	184
2%PAC + 1%XG	34	2 hrs	29,5	37	97,5	124	143	182,5
2%PAC + 1%XG	34	10 hrs	28,5	35,5	95	120,5	139	178,5
2%PAC + 1%XG	34	24 hrs	27	33	88	112,5	130,5	169
2%PAC + 1%XG	34	48 hrs	26	30,5	68	86,5	100,5	128,5
2%PAC + 1%XG	34	72 hrs	31	34,5	64	79	91	114
2%PAC + 1%XG	34	96 hrs	33	36,5	64,5	78	89	110
2%PAC + 1%XG	34	7 days	33,5	36,5	62	75	85	104
2%PAC + 1%XG	34	10 days	34,5	38	62,5	74	82	100
2%PAC + 1%XG	34	14 days	34,5	38	61,5	73	81,5	98
2%PAC + 1%XG	35	0 hrs	27	33,5	92	118	137	176

2%PAC + 1%XG	35	1 hr	27,5	34,5	95	121,5	141	181,5
2%PAC + 1%XG	35	2 hrs	27,5	34,5	95	122,5	142,5	182
2%PAC + 1%XG	35	10 hrs	26,5	33,5	91,5	117,5	137	176
2%PAC + 1%XG	35	24 hrs	25,5	31	85,5	111	130	167
2%PAC + 1%XG	35	48 hrs	24,5	29	66,5	85	99,5	128
2%PAC + 1%XG	35	72 hrs	29,5	33	63	78,5	90,5	113,5
2%PAC + 1%XG	35	96 hrs	31	35	63	78	88	109,5
2%PAC + 1%XG	35	7 days	32,5	36	61	74	84,5	102
2%PAC + 1%XG	35	10 days	30	33,5	56	67	76	93
2%PAC + 1%XG	35	14 days	32	35	57	71	80	94,5

Table 24: 2 % PAC + 2 % XG mixed at 25°C, rheological measurements

Mixing Parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
2%PAC + 2%XG	26	0	75	90	203	247	278	300+
2%PAC + 2%XG	26	1	73,5	89	201	246	278	300+
2%PAC + 2%XG	26	2	73	87	198,5	245,5	278,5	300+
2%PAC + 2%XG	26	10	72	85	180	221,5	250	300+
2%PAC + 2%XG	26	24	74	84	158	191	216	271
2%PAC + 2%XG	26	48	85	93,5	151	177	201	241
2%PAC + 2%XG	26	72	84,5	92	146,5	169,5	191,5	238
2%PAC + 2%XG	26	96	84,5	92	146	170	191	225,5
2%PAC + 2%XG	26	6	85	90,5	141	165	186	225
2%PAC + 2%XG	26	10	84	91,5	140,5	160,5	181	216,5
2%PAC + 2%XG	26	14	82	89	138	160	180	215
2%PAC + 2%XG	27	0	71,5	84,5	193,5	231	269	300+
2%PAC + 2%XG	27	1	72	84,5	192	232,5	272	300+
2%PAC + 2%XG	27	2	71	84	191	232	272,5	300+
2%PAC + 2%XG	27	10	69,5	81	168	209	252,5	295
2%PAC + 2%XG	27	24	72	81	154,5	190,5	221	267
2%PAC + 2%XG	27	48	83	90	149	176	200	238
2%PAC + 2%XG	27	72	84,5	91	146	171	193	233
2%PAC + 2%XG	27	96	84	91,5	145	167,5	188,5	231
2%PAC + 2%XG	27	6	87	94	143,5	165	187,5	226
2%PAC + 2%XG	27	10	91	99	143	164	187,5	221
2%PAC + 2%XG	27	14	94,5	102	141	162	188	217
2%PAC + 2%XG	36	0	70,5	84	180,5	220	249	300+
2%PAC + 2%XG	36	1	71	83	180,5	220,5	249,5	300+
2%PAC + 2%XG	36	2	70	82,5	179	219,5	248	300+

2%PAC + 2%XG	36	10	65	76,5	160,5	197,5	226	287
2%PAC + 2%XG	36	24	68,5	79	148,5	182	207	259
2%PAC + 2%XG	36	48	82,5	90	145,5	170	190	231
2%PAC + 2%XG	36	72	82	89,5	143	167,5	186,5	224
2%PAC + 2%XG	36	96	84	91,5	144,5	166,5	186	227
2%PAC + 2%XG	36	7	83,5	91	142,5	166	184	222
2%PAC + 2%XG	36	10	88	94	143	164,5	185	220
2%PAC + 2%XG	36	14	88,5	95	139,5	161	180,5	213,5
2%PAC + 2%XG	37	0	68	80	177	216	245	300+
2%PAC + 2%XG	37	1	69	80,5	179	219,5	249	300+
2%PAC + 2%XG	37	2	68,5	80	177,5	218	247	300+
2%PAC + 2%XG	37	10	67	77	164	202	231	292
2%PAC + 2%XG	37	24	68	78	152	187	213	266
2%PAC + 2%XG	37	48	82	90	147	174	197	239,5
2%PAC + 2%XG	37	72	81,5	90	144	169,5	190,5	234
2%PAC + 2%XG	37	96	83,5	91	144,5	167	186,5	228
2%PAC + 2%XG	37	7	89,5	96,5	146	170	187,5	229
2%PAC + 2%XG	37	10	93	100	147	169	188	222
2%PAC + 2%XG	37	14	96,5	103	148	169	188	222,5
2%PAC + 2%XG	46	0	74	88	201,5	245,5	276	300+
2%PAC + 2%XG	46	1	73	87	200	244	276,5	300+
2%PAC + 2%XG	46	2	72,5	85,5	200,5	243,5	275,5	300+
2%PAC + 2%XG	46	10	69,5	81	174	212	241	300+
2%PAC + 2%XG	46	24	73	83,5	158	192	219	275
2%PAC + 2%XG	46	48	85,5	93	151	177	201	242
2%PAC + 2%XG	46	72	83	91	147	172	195	238,5
2%PAC + 2%XG	46	96	83	90	147	172	191	234
2%PAC + 2%XG	46	6	86,5	93	145	169	189	232
2%PAC + 2%XG	46	10	92	98	145	166	185,5	219,5
2%PAC + 2%XG	46	14	90	96	142	164	183	216
2%PAC + 2%XG	47	0	70	83,5	191	228	259,5	300+
2%PAC + 2%XG	47	1	71	83	191	229	263	300+
2%PAC + 2%XG	47	2	70	82,5	190	227	263	300+
2%PAC + 2%XG	47	10	66	77,5	164	205	244	291
2%PAC + 2%XG	47	24	67,5	79	152	188	216	264
2%PAC + 2%XG	47	48	81,5	89	150,5	175	196	234
2%PAC + 2%XG	47	72	82	89,5	144	169	191	230
2%PAC + 2%XG	47	96	84	90,5	146	166	187	228
2%PAC + 2%XG	47	7	85,5	92	143	165,5	186	224
2%PAC + 2%XG	47	10	88	94	142,5	164,5	184,5	222
2%PAC + 2%XG	47	14	91,5	96,5	140,5	161,5	183	215

Table 25: 4%PAC + 20 wt. % barite mixed at 25°C, rheological measurements

Mixing Parameters			Fann viscometer RPM					
Mixture	Sample #	rest time	3	6	100	200	300	600
4%PAC + 20%barite	38	0	5	9	62	99	127,5	188,5
4%PAC + 20%barite	38	1	5,5	9	62	99	129,5	192
4%PAC + 20%barite	38	2	5,5	9	62	99,5	129,5	191,5
4%PAC + 20%barite	38	24	5,5	9	62,5	100	129,5	191,5
4%PAC + 20%barite	38	48	5	8,5	59	95	124	184
4%PAC + 20%barite	38	144	2	4	40	67	89	140
4%PAC + 20%barite	38	168	1,5	3	30	51,5	70	114
4%PAC + 20%barite	38	216	1	2	22,5	39	53,5	89
4%PAC + 20%barite	38	336	0,5	1	11	22	33	58
4%PAC + 20%barite	39	0	4,5	8	62	99,5	130	195
4%PAC + 20%barite	39	1	4,5	8	62	100	130,5	196
4%PAC + 20%barite	39	2	4,5	8	62	100	130,5	196
4%PAC + 20%barite	39	24	4,5	8	62	100,5	131	196,5
4%PAC + 20%barite	39	48	4	7	58	95	124	187
4%PAC + 20%barite	39	144	2,5	4	39	65	87,5	137,5
4%PAC + 20%barite	39	168	1,5	2,5	27	47	65	106
4%PAC + 20%barite	39	216	1	2	20	38	55	90
4%PAC + 20%barite	39	336	0,5	1	11	21	33,5	59
4%PAC + 20%barite	40	0	5,5	9	63,5	100,5	130	191
4%PAC + 20%barite	40	1	6	9,5	65,5	103,5	133,5	197
4%PAC + 20%barite	40	2	6	9,5	65,5	104,5	135	198,5
4%PAC + 20%barite	40	24	6	10	68	107,5	138	201,5
4%PAC + 20%barite	40	48	4,5	8	54	89,5	120	196
4%PAC + 20%barite	40	144	4,5	7,5	55	89,5	116	172,5
4%PAC + 20%barite	40	168	3,5	5,5	46	74,5	98	150
4%PAC + 20%barite	40	216	2	3,5	35,5	59	79	122,5
4%PAC + 20%barite	40	336	1	1,5	17	30	40	65
4%PAC + 20%barite	41	0	4,5	8,5	63	100,5	131	195,5
4%PAC + 20%barite	41	1	5	9	65	104	135,5	200,5
4%PAC + 20%barite	41	2	5,5	9	65,5	104,5	136,5	202
4%PAC + 20%barite	41	24	5	9	66	105,5	137,5	202,5
4%PAC + 20%barite	41	48	4,5	8	61	98,5	129	192,5
4%PAC + 20%barite	41	144	3,5	6	49	79,5	104,5	160
4%PAC + 20%barite	41	168	2,5	4	38	63	84	132
4%PAC + 20%barite	41	216	1,5	2,5	26,5	45,5	62,5	100
4%PAC + 20%barite	41	336	0,5	1	10	19	30	51

4%PAC + 20%barite	44	0	4,5	8,5	62,5	100	129,5	193
4%PAC + 20%barite	44	1	4,5	9	63,5	103	130,5	196
4%PAC + 20%barite	44	2	4,5	9	63,5	103,5	130,5	196
4%PAC + 20%barite	44	24	4,5	9,5	67	105	132	197
4%PAC + 20%barite	44	48	4	8	58	95	122	188
4%PAC + 20%barite	44	144	2,5	7	51	85	98	142
4%PAC + 20%barite	44	168	1,5	5	38	70	77,5	108
4%PAC + 20%barite	44	216	1	2,5	29,5	50	58	95
4%PAC + 20%barite	44	336	0,5	1	11	20	32	56
4%PAC + 20%barite	45	0	5	9	63,5	100,5	130,5	192
4%PAC + 20%barite	45	1	5,5	9,5	65,5	103,5	134	198
4%PAC + 20%barite	45	2	5,5	9,5	65,5	104,5	134,5	199,5
4%PAC + 20%barite	45	24	5,5	10	67,5	106	137	201,5
4%PAC + 20%barite	45	48	4,5	8,5	55	93,5	124	193
4%PAC + 20%barite	45	144	4	7,5	53	84	111	165
4%PAC + 20%barite	45	168	3,5	5,5	45	68	93	141
4%PAC + 20%barite	45	216	2	3	32	55	73	108
4%PAC + 20%barite	45	336	1	1,5	14	25	36	61

C.2.2 Gel strength

Table 26: 2 % PAC mixed at 25°C, gel strength

Mixing Parameters			Gel Strength		
Mixture	Sample #	rest time	10sec	1min	10min
2% PAC	18	0	3	3	3
2% PAC	18	1	3	3	3
2% PAC	18	2	3	3	3
2% PAC	18	12	3	3	3
2% PAC	18	24	3	3	3
2% PAC	18	48	3	3	3
2% PAC	18	72	3,5	3	3
2% PAC	18	6days	3	3	2,5
2% PAC	18	10 days	2,5	3	2,5
2% PAC	18	14days	2	1,5	1,5
2% PAC	19	0	3	3	2,5
2% PAC	19	1	3	3	2,5
2% PAC	19	2	3	3	2,5
2% PAC	19	12	3	3	2,5

2% PAC	19	24	2,5	2,5	2,5
2% PAC	19	48	2,5	2,5	2,5
2% PAC	19	72	2,5	3	3
2% PAC	19	6days	2,5	3	2,5
2% PAC	19	10 days	2,5	2,5	2,5
2% PAC	19	14days	1	1	1
2% PAC	42	0	3	2,5	2,5
2% PAC	42	1	3	3	2,5
2% PAC	42	2	2,5	2,5	2,5
2% PAC	42	12	2,5	2,5	2,5
2% PAC	42	24	2,5	2,5	2,5
2% PAC	42	48	2,5	2,5	2,5
2% PAC	42	72	2,5	2,5	2,5
2% PAC	42	6days	2,5	2,5	2,5
2% PAC	42	10 days	2	2,5	2
2% PAC	42	14days	1,5	1,5	1
2% PAC	43	0	3	3	2,5
2% PAC	43	1	3	2,5	2,5
2% PAC	43	2	3	3	2,5
2% PAC	43	12	3	3	2,5
2% PAC	43	24	2,5	2,5	2,5
2% PAC	43	48	2,5	2,5	2,5
2% PAC	43	72	2,5	2,5	2,5
2% PAC	43	6days	2,5	2,5	2,5
2% PAC	43	10 days	2	1,5	1,5
2% PAC	43	14days	1,5	1,5	1,5

Table 27: 4 % PAC mixed at 25°C, gel strength

Mixing Parameters			Gel Strength		
Mixture	Sample #	rest time	10sec	1min	10min
4% PAC	22	0 hrs	9	9	8
4% PAC	22	1 hr	9	9,5	8,5
4% PAC	22	2 hrs	10,5	10,5	9
4% PAC	22	12 hrs	11	11	9,5
4% PAC	22	24 hrs	10	10,5	8
4% PAC	22	48 hrs	11	11	9
4% PAC	22	72 hrs	11	10,5	9
4% PAC	22	6 days	6	7	6
4% PAC	22	10 days	3	3	2,5

4% PAC	22	14 days	2	2	2
4% PAC	23	0 hrs	8	9	7
4% PAC	23	1 hr	8,5	9	7
4% PAC	23	2 hrs	8,5	8	6,5
4% PAC	23	12 hrs	8,5	9	7,5
4% PAC	23	24 hrs	9	9	7,5
4% PAC	23	48 hrs	9	9	8
4% PAC	23	72 hrs	9	9	8
4% PAC	23	6 days	8	9	7
4% PAC	23	10 days	3	3	2
4% PAC	23	14 days	2	2	2
4% PAC	28	0 hrs	8	9	7
4% PAC	28	1 hr	8,5	9	7
4% PAC	28	2 hrs	8,5	8	6,5
4% PAC	28	12 hrs	8,5	9	7,5
4% PAC	28	24 hrs	9	9	7,5
4% PAC	28	48 hrs	9	9	8
4% PAC	28	72 hrs	9	9	8
4% PAC	28	6 days	8	9	7
4% PAC	28	10 days	3	3	2
4% PAC	28	14 days	2	2	2
4% PAC	29	0 hrs	8	9	7
4% PAC	29	1 hr	8	8	6
4% PAC	29	2 hrs	9	8	6
4% PAC	29	12 hrs	9	9	7
4% PAC	29	24 hrs	9	8	7
4% PAC	29	48 hrs	9	9	7,5
4% PAC	29	72 hrs	9	9	7,5
4% PAC	29	6 days	5	5,5	5
4% PAC	29	10 days	2	4	3
4% PAC	29	14 days	2	2	3
4% PAC	48	0 hrs	8	8	7
4% PAC	48	1 hr	9	9	8
4% PAC	48	2 hrs	11	10	9
4% PAC	48	12 hrs	11	10	9
4% PAC	48	24 hrs	11	11	9
4% PAC	48	48 hrs	11	11	9
4% PAC	48	72 hrs	12	10,5	8
4% PAC	48	6 days	4	5	4
4% PAC	48	10 days	3	3	2,5
4% PAC	48	14 days	2	2	2

4% PAC	49	0 hrs	8	8	7
4% PAC	49	1 hr	9,5	9	8,5
4% PAC	49	2 hrs	11	10	9
4% PAC	49	12 hrs	11	10	9
4% PAC	49	24 hrs	11	11	9
4% PAC	49	48 hrs	11	11	9
4% PAC	49	72 hrs	12	10,5	8
4% PAC	49	6 days	4	5	4
4% PAC	49	10 days	3	3	2,5
4% PAC	49	14 days	2	2	2

Table 28: 2 % PAC+ 1% XG mixed at 25°C, gel strength

Mixing parameters			Gel Strength		
Mixture	Sample #	rest time	10sec	1min	10min
2%PAC + 1%XG	24	0 hrs	37,5	37,5	36
2%PAC + 1%XG	24	1 hr	37,5	37,5	36
2%PAC + 1%XG	24	2 hrs	37	37,5	36
2%PAC + 1%XG	24	10 hrs	36,5	36	37
2%PAC + 1%XG	24	24 hrs	33,5	34	34,5
2%PAC + 1%XG	24	48 hrs	31	33	36
2%PAC + 1%XG	24	72 hrs	32,5	36	40
2%PAC + 1%XG	24	96 hrs	33	36	39
2%PAC + 1%XG	24	6 days	33	37	41
2%PAC + 1%XG	24	10 days	33	37	41
2%PAC + 1%XG	24	14 days	34	37	41
2%PAC + 1%XG	25	0 hrs	34	35	33
2%PAC + 1%XG	25	1 hr	34	35	33
2%PAC + 1%XG	25	2 hrs	33	35	33
2%PAC + 1%XG	25	10 hrs	35	34	36
2%PAC + 1%XG	25	24 hrs	32,5	32,5	33
2%PAC + 1%XG	25	48 hrs	29	31	34
2%PAC + 1%XG	25	72 hrs	30	35	39
2%PAC + 1%XG	25	96 hrs	32,5	34	38
2%PAC + 1%XG	25	6 days	33	36,5	41
2%PAC + 1%XG	25	10 days	33,5	37	41,5
2%PAC + 1%XG	25	14 days	32	35	42
2%PAC + 1%XG	30	0 hrs	32	33	34,5

2%PAC + 1%XG	30	1 hr	33	33,5	34,5
2%PAC + 1%XG	30	2 hrs	33	33,5	34,5
2%PAC + 1%XG	30	10 hrs	32,5	33	34
2%PAC + 1%XG	30	24 hrs	31	31,5	32,5
2%PAC + 1%XG	30	48 hrs	28	30	33
2%PAC + 1%XG	30	72 hrs	31,5	34,5	38
2%PAC + 1%XG	30	96 hrs	32	35	38,5
2%PAC + 1%XG	30	7 days	32	35,5	40
2%PAC + 1%XG	30	10 days	33	35	40
2%PAC + 1%XG	30	14 days	33	36,5	40,5
2%PAC + 1%XG	31	0 hrs	32,5	33	34,5
2%PAC + 1%XG	31	1 hr	33	34	35
2%PAC + 1%XG	31	2 hrs	33	34	35
2%PAC + 1%XG	31	10 hrs	34	35	35,5
2%PAC + 1%XG	31	24 hrs	31	31,5	33
2%PAC + 1%XG	31	48 hrs	29	31	34
2%PAC + 1%XG	31	72 hrs	32	35,5	39
2%PAC + 1%XG	31	96 hrs	33,5	37	41
2%PAC + 1%XG	31	7 days	34	37,5	42
2%PAC + 1%XG	31	10 days	33	36	40
2%PAC + 1%XG	31	14 days	33	36	40,5
2%PAC + 1%XG	32	0 hrs	32	33	33
2%PAC + 1%XG	32	1 hr	33	34	35
2%PAC + 1%XG	32	2 hrs	34	34,5	35
2%PAC + 1%XG	32	10 hrs	32,5	33	34
2%PAC + 1%XG	32	24 hrs	29,5	30,5	32
2%PAC + 1%XG	32	48 hrs	28,5	31	35
2%PAC + 1%XG	32	72 hrs	31	34,5	38
2%PAC + 1%XG	32	96 hrs	32	36	40
2%PAC + 1%XG	32	7 days	33	36	40
2%PAC + 1%XG	32	10 days	33	36	40
2%PAC + 1%XG	32	14 days	33,5	36,5	41
2%PAC + 1%XG	33	0 hrs	33	34	34
2%PAC + 1%XG	33	1 hr	34	34	36
2%PAC + 1%XG	33	2 hrs	34	34,5	35,5
2%PAC + 1%XG	33	10 hrs	33	33,5	34,5
2%PAC + 1%XG	33	24 hrs	30	32	33,5
2%PAC + 1%XG	33	48 hrs	29	32	37
2%PAC + 1%XG	33	72 hrs	32	36	41
2%PAC + 1%XG	33	96 hrs	33	37	42
2%PAC + 1%XG	33	7 days	34	37,5	43
2%PAC + 1%XG	33	10 days	33	35	42

2%PAC + 1%XG	33	14 days	34	38,5	43
2%PAC + 1%XG	34	0 hrs	32	32,5	32,5
2%PAC + 1%XG	34	1 hr	33	34	34,5
2%PAC + 1%XG	34	2 hrs	33	33	34
2%PAC + 1%XG	34	10 hrs	32	32,5	33,5
2%PAC + 1%XG	34	24 hrs	30	30,5	31,5
2%PAC + 1%XG	34	48 hrs	28	30	34
2%PAC + 1%XG	34	72 hrs	31	34	37
2%PAC + 1%XG	34	96 hrs	32	36	41
2%PAC + 1%XG	34	7 days	34	36	40,5
2%PAC + 1%XG	34	10 days	34	36	40,5
2%PAC + 1%XG	34	14 days	33,5	37	41
2%PAC + 1%XG	35	0 hrs	30	32	32
2%PAC + 1%XG	35	1 hr	32,5	33	34
2%PAC + 1%XG	35	2 hrs	32,5	33	34
2%PAC + 1%XG	35	10 hrs	32	32,5	33,5
2%PAC + 1%XG	35	24 hrs	29,5	30,5	31
2%PAC + 1%XG	35	48 hrs	28	30	34
2%PAC + 1%XG	35	72 hrs	31,5	35	39
2%PAC + 1%XG	35	96 hrs	32	36	42
2%PAC + 1%XG	35	7 days	34	37	42,5
2%PAC + 1%XG	35	10 days	32	36	42
2%PAC + 1%XG	35	14 days	34	38	43

Table 29: 4 % PAC + 20 wt. % barite mixed at 25°C, gel strength

Mixing Parameters			Gel Strength		
Mixture	Sample #	rest time	10sec	1min	10min
4%PAC + 20%barite	38	0	8	8,5	6,5
4%PAC + 20%barite	38	1	8,5	9	6,5
4%PAC + 20%barite	38	2	9	9,5	7
4%PAC + 20%barite	38	24	9	9,5	7
4%PAC + 20%barite	38	48	7,5	8	6
4%PAC + 20%barite	38	144	3	3,5	3
4%PAC + 20%barite	38	168	3	2	2
4%PAC + 20%barite	38	216	2	1,5	2
4%PAC + 20%barite	38	336	1	1	0,5
4%PAC + 20%barite	39	0	8	9	6

4%PAC + 20%barite	39	1	8,5	8,5	6,5
4%PAC + 20%barite	39	2	8,5	9	6,5
4%PAC + 20%barite	39	24	9	9	6,5
4%PAC + 20%barite	39	48	7	9	6
4%PAC + 20%barite	39	144	3,5	3,5	3,5
4%PAC + 20%barite	39	168	2,5	2,5	2
4%PAC + 20%barite	39	216	2,5	2	2,5
4%PAC + 20%barite	39	336	1	1	1
4%PAC + 20%barite	40	0	9	9	7
4%PAC + 20%barite	40	1	9,5	10	7
4%PAC + 20%barite	40	2	10	10	7,5
4%PAC + 20%barite	40	24	10	10,5	8
4%PAC + 20%barite	40	48	8	9	6
4%PAC + 20%barite	40	144	8	8	5
4%PAC + 20%barite	40	168	6	6	4,5
4%PAC + 20%barite	40	216	3,5	3	2,5
4%PAC + 20%barite	40	336	2	1,5	2
4%PAC + 20%barite	41	0	9,5	9,5	7
4%PAC + 20%barite	41	1	10	10,5	7,5
4%PAC + 20%barite	41	2	10	10	7,5
4%PAC + 20%barite	41	24	10	10	7
4%PAC + 20%barite	41	48	8	9	6
4%PAC + 20%barite	41	144	6	5	3,5
4%PAC + 20%barite	41	168	4	4	3
4%PAC + 20%barite	41	216	2	2	2
4%PAC + 20%barite	41	336	1	1	0,5
4%PAC + 20%barite	44	0	8,5	9	6,5
4%PAC + 20%barite	44	1	9	9	6,5
4%PAC + 20%barite	44	2	9	9	6,5
4%PAC + 20%barite	44	24	9,5	10	7
4%PAC + 20%barite	44	48	7,5	8,5	6
4%PAC + 20%barite	44	144	4	4	3,5
4%PAC + 20%barite	44	168	3,5	3	3
4%PAC + 20%barite	44	216	2,5	2	2,5
4%PAC + 20%barite	44	336	1	1	1
4%PAC + 20%barite	45	0	9	9	7
4%PAC + 20%barite	45	1	9,5	10	7,5
4%PAC + 20%barite	45	2	9,5	10,5	7,5
4%PAC + 20%barite	45	24	10	10,5	8
4%PAC + 20%barite	45	48	8	9	6
4%PAC + 20%barite	45	144	6	7	4,5

4%PAC + 20%barite	45	168	6	6	4,5
4%PAC + 20%barite	45	216	3,5	3	2,5
4%PAC + 20%barite	45	336	1,5	1,5	1