

**STATISTICAL CHARACTERIZATION OF PERFORMANCE OF
BIOPOLYMER DRILL-IN FLUID FOR DIFFERENT RHEOLOGICAL
MODELS**

**A
THESIS**

Presented to the Faculty of the

African University of Science and Technology

in Partial Fulfilment of the requirements for the

Degree of

MASTER OF SCIENCE

By

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Abuja, Nigeria

November, 2010

**STATISTICAL CHARACTERIZATION OF PERFORMANCE OF
BIOPOLYMER DRILL-IN FLUID FOR DIFFERENT RHEOLOGICAL
MODELS**

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ABSTRACT

Appropriate selection of rheological models is important for hydraulic calculations of pressure loss prediction and hole cleaning efficiency of drilling fluids. Power law, Bingham-Plastic Herschel-Bulkley models are the conventional fluid models used in the oilfield. However, there are other models that have been proposed in literature which are under / or not utilized in the petroleum industry. The primary objective of this study is to recommend a rheological model that best-fits the rheological behaviour of xanthan gum based biopolymer drill-in fluids for hydraulic evaluations. Ten rheological models were evaluated in this study. These rheological models have been posed deterministically. Obviously this is unrealistic so these deterministic models are replaced by statistical models by adding an error (disturbance) term and making suitable assumptions about them. Rheological model parameters were estimated by least-square regression method. Models like Sisko and modified Sisko which are not conventional models in oil industry gave a good fit. Modified Sisko model which is a four parameter rheological model was selected as the best-fit model since it produced the least residual mean square. There is 95% certainty that the true best-fit curve lies within the confidence band of this function of interest.

TABLE OF CONTENT

	Page
Title Page	i
Signature Page	ii
ABSTRACT	iii
TABLE OF CONTENT	iv
LIST OF FIGURES	vi
LIST OF TABLES	vii
ACKNOWLEDGEMENT	viii
CHAPTER ONE INTRODUCTION	1
1.1 INTRODUCTION.....	1
1.1 PROBLEM DEFINITION	2
1.2 OBJECTIVES	3
1.3 METHODOLOGY	3
CHAPTER TWO LITERATURE REVIEW	4
2.0 INTRODUCTION.....	4
2.1 DRILL-IN FLUIDS.....	4
2.2 POLYMER STRUCTURE	5
2.3 APPLICATION OF POLYMERS	6
2.3.1 Hydroxymethylcellulose (HEC)	6
2.3.2 Carboxymethylcellulose (CMC).....	6
2.3.3 Starches	6
2.3.4 Polyacrylamides	7
2.3.5 Biopolymers.....	7
2.3.5.1 Rheology.....	7
2.3.5.2 Shear Stability	8
2.3.5.3 Temperature Stability.....	8
2.3.5.4 Salt Solubility	9
2.3.5.5 Acid Stability	9
2.4 PROPERTIES OF XANTHAN GUM.....	9
2.4.1 Shear Thinning.....	10
2.4.1 Low Shear Rate Viscosity, LSRV.....	11
2.4.3 Shear Degradation of Xanthan Gum.....	11
2.5 THEORY OF THE RHEOLOGICAL MODELS.....	12
2.5.1 Bingham Plastic Model.....	13

2.5.2 Power Law Model.....	14
2.5.3 Herschel-Bulkley Model.....	14
2.5.4 Robertson-Stiff Model	15
2.5.5 Prandtl-Eyring Model	15
2.5.6 Sisko Model.....	16
2.5.7 Modified-Sisko Model.....	16
2.5.8 Casson Model	17
2.6 REGRESSION ANALYSIS AND MODEL COMPARISON	18
CHAPTER THREE DEVELOPMENT OF STATISTICAL MODEL	20
3.0 INTRODUCTION.....	20
3.1 DATA COLLECTION	20
3.2 MODEL SPECIFICATION.....	21
3.3 CHOICE OF FITTING METHOD AND MODEL FITTING	22
3.4 STATISTICAL MEASURE	23
3.4.1 Model Comparison	23
3.4.2 Confidence Interval	23
CHAPTER FOUR APPLICATION OF MODEL EQUATION ON DATA.....	25
4.0 INTRODUCTION.....	25
4.1 EVALUATION OF BINGHAM PLASTIC MODEL	25
4.2 EVALUATION OF POWER LAW MODEL.....	26
4.3 EVALUATION OF HERSCHEL-BULKLEY MODEL	26
4.4 EVALUATION OF ROBERSTON-STIFF MODEL	27
4.5 EVALUATION OF MODIFIED ROBERTSON-STIFF MODEL.....	27
4.6 EVALUATION OF PRANDTL-EYRING MODEL.....	28
4.7 EVALUATION OF MODIFIED PRANDTL-EYRING MODEL	29
4.8 EVALUATION OF SSKO MODEL	29
4.9 EVALUATION OF MODIFIED SSKO MODEL	30
4.10 EVALUATION OF CASSON MODEL.....	30
CHAPTER FIVE ANALYSIS OF RESULT.....	32
5.0 INTRODUCTION.....	32
5.1 DETERMINATION OF STATISTICAL CORRELATION.....	32
5.2 FITTED CURVES AND RESIDUAL ANALYSIS.....	32
5.3 SUM-OF-SQUARES AND MEAN SQUARES	41
5.4 CONFIDENCE INTERVAL.....	43
CHAPTER SIX CONCLUSION(S) AND RECOMMENDATION(S).....	44
REFERENCES.....	45
NOMENCLATURE.....	50
APPENDIX A	52

LIST OF FIGURES

	Page
Figure 2.1: Basic structure of Xanthan gum.....	10
Figure 2.2: Comparison of Non-Newtonian and Newtonian fluid effective viscosity	12
Figure 2.3: Rheogram of fluids behaviour.....	13
Figure 5.1a: Experimented rheological data and fitted Bingham Plastic values.....	33
Figure 5.1b: Residual plot of fitted Bingham Plastic values	33
Figure 5.2a: Experimented rheological data and fitted Casson values.....	34
Figure 5.2b: Residual plot of fitted Casson values.....	34
Figure 5.3a: Experimented rheological data and fitted Power law values.....	35
Figure 5.3b: Experimented rheological data and fitted Herschel-Bulkley values.....	35
Figure 5.4a: Residual plot of fitted Power values.....	35
Figure 5.4b: Residual plot of fitted Herschel-Bulkley values.....	36
Figure 5.5a: Experimented rheological data and fitted Robertson-Stiff values	36
Figure 5.5b: Experimented rheological data and fitted Modified Robertson-Stiff values.....	37
Figure 5.6a: Residual plot of fitted Robertson-Stiff values values.....	37
Figure 5.6a: Residual plot of fitted Modified Robertson-Stiff values.....	37
Figure 5.7a: Experimented rheological data and fitted Prandt-Eyring values.....	38
Figure 5.7b: Experimented rheological data and fitted Modified Prandt-Eyring values.....	38
Figure 5.8a: Residual plot of fitted Prandt-Eyring values.....	39
Figure 5.8b: Residual plot of fitted Modified Prandt-Eyring values.....	39
Figure 5.9a: Experimented rheological data and fitted Sisko values.....	40
Figure 5.9b: Experimented rheological data and fitted Modified Sisko values.....	40
Figure 5.10a: Residual plot of fitted Sisko values.....	40
Figure 5.10b: Residual plot of fitted Modified Sisko values.....	41
Figure 5.11: Experimented rheological data, fitted Modified Sisko values and its confidence interval.....	43

LIST OF TABLES

	Page
Table 5.1: Summary Result from Least Square Regression Approximation.....	42
Table A1: Table A1: Results of Rheological Data from Viscometer Readings	52
Table A2 Parameter constraints and Initial Guess to Evaluate the Rheological Model Functions.....	52
Table A3: Experimented, Expected Modified Sisko and 95 % Confidence Interval Shear Stress Values.....	53

ACKNOWLEDGEMENT

I wish to first thank the almighty God, Jehovah for granting me wisdom, power and a sound mind in compiling these ideas together into this work.

Secondly, I wish to appreciate my supervisor, Professor Emeritus Godwin A. Chukwu, for his efforts, advice and correction which brought the best out of me. His immense contributions which cannot be overemphasized have made this thesis work a great success.

I am very thankful to Dr. Calin Gheorghiu for introducing me to the usage of MATLAB Software environment and assisting me to develop an iterative algorithm to solve systems of non-linear equations.

I kindly and respectfully appreciate all the contributions provided by Professor Olurinde Lafe of Lafe Research LLC, USA in this work.

I also thank all my friends who have contributed in one way or other towards the success of this study. Your encouragement and moral support are really appreciated.

CHAPTER ONE

INTRODUCTION

1.1 INTRODUCTION

The use of rheological models to approximate the behaviour of non-Newtonian fluids is very paramount in the oil and gas industry especially during drilling, well completion, workover and acidizing. In drilling operations, mathematically designed rheological models are used to describe the viscous forces to develop frictional pressure loss equations. Accurate prediction of pressure losses help in the determination of bit optimization hydraulics, estimation of equivalent circulating density (ECD) and drilling fluid compressibility. The benefits of a more accurate estimation of ECD is adequate hole cleaning efficiency to enhance total drilling rate which in turn reduces total drilling cost. Prevention of circulation loss, maintenance of under-balanced drilling conditions and detection of potential kick are achieved if ECD is rightfully predicted (Bailey and Peden, 2000). Estimated model parameters help to perform other hydraulics calculations.

Power Law and Bingham Plastic models are widely used for hydraulics evaluation. They are assumed for standard API hydraulics calculations. Herschel-Bulkley, Roberston-Stiff and Casson models have been accepted to some extent in the petroleum industry. These models and the corresponding hydraulic calculations do provide a way for fair estimates of hydraulics for conventional wells using simple drilling fluids as asserted by Guo and Hong in 2010. Power Law model predicts shear stress well at low shear rate (in the annulus) and Bingham Plastic model describes the characteristics of drilling fluid at high shear rate (in the drill pipe).

Biopolymer drill-in fluid is a complex fluid formulated with several compositions to desired properties for optimum performance particularly in unconventional wells. It is a water soluble 'rheology engineered' drilling fluid designed to optimize the performance of rotary drilling. It is a complex high molecular weight (MW) polymer with a strong bond between the chains of its molecules which is efficiently used in unconventional wells like onshore and offshore horizontal wells, coiled tubing drilling and slim holes. The elastic structures of biopolymers make them have a higher carrying capacity than the other polymers applied in the petroleum industry during drilling. Due to the complex nature of this type of fluid and its unusual behaviour, it is very prudent to use a more precise rheological model to characterize its behaviour over a full range of shear rate to achieve a proper hydraulics evaluation.

Drill-in fluids are specially designed fluid system for drilling through the reservoir interval of a wellbore. They are basically formulated to drill the reservoir zone successfully, often a long, horizontal drainhole, to minimize damage and optimize the production of the exposed zones and to enhance the well completion needed. It contains additives that can principally control filtration loss and facilitate optimum carrying capacity. Its composition may be brine with right aggregate size (salt crystals or calcium carbonates) and polymers (www.oilfield.slb.com). Brian et. al (1997) asserted that polymers typically used as drill-in fluids are xanthan gum, starch, cellulose and scleroglucans. Hemphill et al. in 1993 proposed that Herschel-Bulkley model which is a three-parameter model is more likely to approximate the non-Newtonian behaviour of polymeric fluids.

This study focuses on ten rheological models proposed in various literatures and come out with a statistical criterion to select the most likely model to predict the rheological characteristics of xanthan gum base biopolymer drill-in fluids.

1.2 PROBLEM DEFINITION

The use of rheological models in the characterization of the behaviour of non-Newtonian fluid aids in the evaluation of drilling fluid hydraulics. The Power Law and / or Bingham Plastic models are more often used in evaluating hydraulics of drilling fluid in the oilfield during drilling operations. These are used because their resultant flow equations are simple and it is also easier to estimate parameters of the models by explicit solutions. However, none of these is able to predict the behaviour of the fluids over the wide ranges of deformation rate during the circulating of drilling fluid system throughout the wellbore. The advent of computers makes it realistic to estimate parameters of more complicated models and thereby deriving expression of pressure drop as function of flow rate. Rig site computers are now readily available making the requirement for simple parameter estimation and easily manipulated flow functions redundant and provide a platform conducive to more rigorous analysis.

1.3 OBJECTIVE

The objective of this work is to be able to identify the fluid flow model that is more likely to predict the behaviour of xanthan gum base biopolymer drill-in fluids and estimate the confidence interval of the selected model function in order to quantify the degree of certainty. The Power law, Bingham Plastics, Herschel-Bulkley, Roberston-Stiff, Modified- Roberston-Stiff, Prandtl – Eyring, Modified Prandtl – Eyring, Sisko, Modified Sisko and Casson models were used for the purpose of this study.

1.4 METHODOLOGY

The work was conducted by collection of Fann viscometer readings on ‘rheology engineered’ solid free xanthan gum base biopolymer drill-in fluids used in coiled tubing drilling. The appropriate models were specified and statistical regression model was used. After data collection and model specification, the estimation of model parameters using Least-square regression approximation of functions method was made. Matlab code was developed to solve each non-linear model function using a quasi Newton’s numerical iterative approach. Results of the regression analysis were plotted and the residual sum of squares were analysed. Due to small sample size, residual mean squares were employed as a statistical tool to account for the error variance. The model with the minimum residual mean squares was selected. Confidence interval of the selected model function of fitted shear stress values was also estimated. Relevant graphs were plotted to make judicious engineering analysis and decision based on the results and literature knowledge.

CHAPTER TWO

LITERATURE REVIEW

2.0 INTRODUCTION

This chapter covers a comprehensive background study of the drill-in fluids and their characteristics. The properties of polymeric drilling fluids including biopolymers as reported by previous investigations are discussed. Rheology of a drilling fluid is one of the important properties that enhances effective and efficient drilling especially for hole cleaning. This property is characterized by various mathematical rheological models which assist in hydraulic analysis. This chapter discusses the theory and applications of rheological models studied in this work.

2.1 DRILL-IN FLUIDS

Drill-in fluids are specially designed fluid for drilling through the reservoir interval of a wellbore. They are basically formulated to drill the reservoir zone successfully, often a long, horizontal drainhole, to minimize damage and optimize the production of the exposed zones and to enhance the well completion needed. It contains additives that can principally control filtration loss and facilitate optimum carrying capacity (www.oilfield.slb.com). The 2001 drilling fluid engineering manual states that reservoir drill-in fluids are extremely important in horizontal wells, where low drawdown pressures make clean-ups more difficult. Its composition may be brine with right aggregate size (salt crystals or calcium carbonates) and polymers. Gerrit (2007) asserted that the commonly used bridging agents in formulating the drill-in fluids are calcium carbonate, sized salts and oil soluble resins. He suggested that the most probable important factor is the selection of the right size of this high speed zone cutting path solids, as any solids that manage to enter the formation prior to the formation of a filter cake are likely to be unreachable for stimulation fluids.

Polymers typically used as drill-in fluids are xanthan gum, starch, cellulose and scleroglucans. Brian et. al (1997) stated that the starch or cellulose polymers provide viscosity for friction reduction and lubrication. The xanthan gum facilitates cuttings transportation whereas the removable particulates (bridging agents) help in filtration loss control. They discovered that although drill-in fluids are constitutionally less damaging than conventional drilling fluid, there are some levels of leakage of fluid into the formation and deposits of impermeable filter cake at the wellbore wall which will eventually impede flow capacity if not clean effectively. Brian et. al (1997) asserted that this problem is pronounced in horizontal well drilling and the formation

damage can be minimized using brine-based drill-in fluid system with acid or water soluble weighting agents followed by the application acid or an oxidative breaker systems to dissolve filter-cake solids and polymers.

Ekeledirichukwu in 2010 assessed the characteristics of biopolymer drill-in fluid used for coiled tubing drilling on the Alaskan North Slope. He analysed some properties that affect the optimum functionality of xanthan gum based biopolymer drill-in in hole cleaning as drilling depth increases. He discovered that alteration of rheology and low shear rate viscosities affect the fluid performance with depth due to degradation of polymer structure. However, high shearing enormously caused the destruction of biopolymer chain and recommended periodic addition of the said drill-in fluid as drilling progresses.

2.2 POLYMER STRUCTURE

Water soluble polymers are basically classified into three types. These are polysaccharides (i.e biopolymers), modified polymers and synthetic polymers (polyacrylamides). Polysaccharides are formed as result of bacteria fermentation process whereby saccharide molecules are polymerized. The molecules are bonded together by glycosidic linkages and are comparatively non-ionic. Modified polymers are those treated chemically to achieve a particular desired properties like high solubility, less salt contamination and high resistance against the attack of bacteria. Synthetic polymers are polymers which molecular chains grow polymerization (Talabani et. al., 1993).

Generally, polymers show viscoelastic or plasticoviscous structure dynamically and exhibit either thixotropic or time independent non-Newtonian behaviour under static conditions (Talabani et. al., 1993).

Talabani et. al.(1993) asserted that polymers can be classified under the low solid system which has solid content less than 10 % by weight with a density of less than 9.5 ppg and are water base structure and hydrocolloid materials. There is a critical concentration of polymers to be used in drilling fluids based on which each polymer is characterized. This concentration is called critical polymer concentration (CPC) which is the concentration of the polymer at which the polymer fluid properties change drastically (Talabani et. al., 1993).

2.3 APPLICATION OF POLYMERS

Polymer solutions are predominantly used in the oilfield in several operations ranging from enhanced oil recovery to drilling operations as drilling, workover and completion fluids because they exhibit unique and excellent properties. Biopolymers for instance show high viscosifying power and excellent stability to salt and shear degradation making them applicable in thickening process (Ash et. al., 1983). In drilling, Polymers have been used as viscosifiers especially in applications requiring minimum solids loading, filtration loss control material, flocculation, and shale stabilization (Cerico and Bagshaw, 1978).

Polymers (biopolymers) have been used adequately in drilling in several oilfields including Prudhoe Bay, Alaska horizontal wells. In Prudhoe Bay, the biopolymer based drill-in fluids was successful and exhibited good suspension and optimum transport capacity (Zamora et. al, 1993). Some of the polymers used mainly as or combined with other polymers as viscosifiers are hydroxyethylcellulose (HEC), carboxymethylcellulose (CMC), polyacrylamide (PAA), starches, and biopolymers. These polymers are discussed below.

2.3.1 Hydroxyethylcellulose (HEC)

HEC is a synthetic polymer. It is mostly added in drilling fluid containing clayey materials as viscosifiers. In terms of structure HEC is similar to CMC. Both are cellulose based polymers but ethyl group replace methyl group in CMC. Ekeledirichchukwu in 2010 noted in his research work that HEC comes in several grades but exhibits plastic viscosity up to 12 cp.

2.3.2 Carboxymethylcellulose (CMC)

Carboxymethylcellulose is also a synthetic polymer like HEC. Arrangement of its structure is such that chains of carboxymethyl group are attached to cellulose backbone. It has several repetitive units of carboxymethylcellulose. The solubility of CMC is inversely proportional to the amount of salt in solution. Its solubility therefore decreases in highly saline solution. It is recommended to use CMC in the presence of low salinity (Ekeledirichchukwu, 2010). The main purpose of CMC addition in drilling fluid is to increase fluid viscosity particularly in clay drilling fluids. It possesses various grade of viscosity.

2.3.3 Starches

Starches of potato and corn are popularly applied in drilling fluids. They are added to basically control filtration loss. They can also be used to stabilize shale in some formations. Starches are

vulnerable to bacteria degradation so they are always preserved.

2.3.4 Polyacrylamides

This is also used predominantly in drilling fluids in the oilfield for shale stabilization and flocculation of clay materials. Like Starches, polyacrylamides are degradable by bacterial attack so they always come along with preservatives.

2.3.5 Biopolymers (BP)

Biopolymers are natural polymers produced by bacterial action on carbohydrates. The drilling fluid should be highly concentrated and maintained if only biopolymer is to be used (Talabani et. al, 1993). They have unique properties that have been exploited in operations in the oilfield ranging from EOR to drilling. Biopolymers are of high molecular weight (MW) and show a strong bond between the chains of its molecules. The MW can be as high as 5 million and the chain is extracellular microbial polysaccharides (Talabani et. al, 1993). The elastic structures of biopolymers make them have a higher carrying capacity than the other polymers applied in the petroleum industry during drilling. The stability of biopolymers depends on salinity of the solution at a particular temperature (Ash et. al., 1983). Ash et. al.(1983) concluded in their research that biopolymers exhibit good retention of viscosity in sea water at 90 °C for several months. The water soluble BP (e.g Xanthan gum and Sclerogulcan) produced by microbes are the ones widely used. Xanthan gum, scleroglucan, guar gum, and polysaccharides are the commonly used water-soluble biopolymer in drilling and completion fluids. Some of the unique characteristics exhibited by biopolymers according to literature are discussed below.

2.3.5.1 Rheology

Rheology by definition is the science of deformation and flow of materials in response to stress. According to Talabani et. al (1993), field experiments have shown that biopolymer loose a lot of water into formations and hence making them have higher rheology than any other polymer added to drilling fluid. Talabani et. al assert that the chains of biopolymer do not have similar length so the chain with low molecular weight will pass through 325 mesh paper. This gives an implication that if biopolymer is only used as drilling fluid, these chains can easily penetrate the pore spaces in the formation leaving behind thicker biopolymer with high rheology.

Most biopolymers exhibit a pseudoplastic (shear thinning) behavior in aqueous media (Lipton and Burnett, 1975; Katzbauer B., 1998). Thus their viscosities decrease with increase in shear rate. This behaviour exhibited by this type of polymer in fluid helps to optimize the pump pressure available

to circulate drilling fluid since maximum flow rate is achieved at minimum pump pressure. Adequate hole cleaning is obtained at the bit where shear rate is high resulting in high penetration rate.

They also show high low shear rate viscosity (LSRV) than any other viscosifier use in drilling fluid according to study conducted by Cerico and Bagshaw (Ekeledirichchukwu, 2010). This means in the annulus where low shear exists, the fluid will have high viscosity and will be able to keep the cuttings in suspension. Therefore, hole cleaning will be optimized if this viscosifier is used.

2.3.5.2 Shear Stability

Previous studies by Cerico and Bagshaw (1978) depict that biopolymers regain their rheological properties after being exposed to high shear rate. For instance, 80 % rheology is regained if xanthan gum is subjected to high shear rate. These can be attributed to the large molecular structure and the strong bond between the chains. Shear stability is very important in the overall cost effectiveness in a polymer system (Maerker, 1975). Drilling fluid system that can withstand realistic high shear stresses encountered during drilling and do not require continuous replacement is economically preferable.

2.3.5.3 Temperature Stability

Temperature is one of the factors that affect the rheological properties of most drilling fluid used in drilling operations in the oilfield. Factors that limit the stability of polymers at elevated temperatures include the nature and mechanism of degradation of these polymers. Understanding of these will help to apply polymers successfully. Most polymers long molecular chains are broken into short chains when they are subjected to high temperatures. Ash et. al.(1983) noted in their research that biopolymers exhibit good retention of viscosity in sea water at 90 °C for several months. Biopolymers are able to withstand high temperature because of their complex structure and strong bonds between the long chains. They start losing their rheological properties at temperatures between 250 °F to 300 °F. But they are able to regain their rheological properties when temperature starts reducing although they can degrade at elevated temperatures. Cerico and Bagshaw(1978) conducted an experiment by subjecting biopolymers to various elevated temperatures and later allowed them to cool down. They discovered that between 80 % and 100 % rheological properties of biopolymers were regained after they have been cooled down.

2.3.5.4 Salt Solubility

Polymers are mostly hydrophilic and this property can create problem in their application in the oilfield. Because of their hydrophilic nature they are not properly mix when powdered polymers are poured in water resulting in lump formations. The out surface quickly sticks to the water molecules which eventually prevents further entry of water into the inner layer of the polymers.

Biopolymers are exceptional; they are highly soluble in salt water like sea water, NaCl/KCl and high CaCl₂ solutions. Their stability depends on salinity. Unlike other polymers, biopolymers hydrate well in salt solution preventing lump from forming when used as viscosifier in drill-in fluids. When lumps are formed in drilling fluids the full potential of fluid properties will not be utilized. Talabani et. al. assert that in drilling shale formation, 5 % by weight of CaCl₂ should be mixed with biopolymer base drilling fluid solution. Field experience indicates that the gel structure and yield value will increase to certain level due to increase in salinity of the solution and becomes more sensitive to formations having other ions (Talabani et. al, 1993). In low solids drilling application, use of saline water to provide sufficient density is a common practice (Gantt et al. 1998; Garfield et al., 2008; Goodrich et al., 1995).

2.3.5.5 Acid Stability

Studies by Cerico and Bagshaw (1978) depicts that biopolymer can still retain their properties with an increase in acidity of solution. Other drilling fluid systems lose their properties in acidic medium although biopolymers would function effectively in an alkaline media.

2.4 PROPERTIES OF XANTHAN GUM

Xanthan gum exhibits distinctive properties from other polymers used in preparing drilling fluids. It is a high molecular-weight extracellular polysaccharide produced by the bacterium *Xanthomonas campestris* (Cheila and Denise, 2002). Katzbauer (1998) and Arendt et al. (1993) reported that the molecular weight of xanthan gum is approximately 2 million g/mol-3 (Ekeledirichukwu, 2010). Its complex structure is shown in figure 2.1(Ekeledirichukwu, 2010). It consists of 1,4 linked -D-glucose residues, having a trisaccharide side chain attached to alternate D-glucosyl residues. The side chains are one unit of D-glucuronic acid between two units of D-mannose. In order to adjust the desired flow behaviour, xanthan gum is used in combination with other hydrocolloids. Synergistic interactions between hydrocolloids have a special commercial interest, as they offer the possibility of novel functionalities and of using reduced levels of hydrocolloids, reducing costs (Cheila and Denise, 2002).

The secondary structure of Xanthan gum has been shown to have a five-fold helical structure although it is yet not clarified whether the structure is a double or single helix. This polymer undergoes a conformational change with increasing temperature (Arendt and Kulicke, 1998). It shows a unique change of physical properties by undergoing transitional change of conformation when its solution is heated. However, the helical structure is orderly arranged at low temperatures. The converse occurs at high temperatures as noted by Arendt and Kulicke (1998).

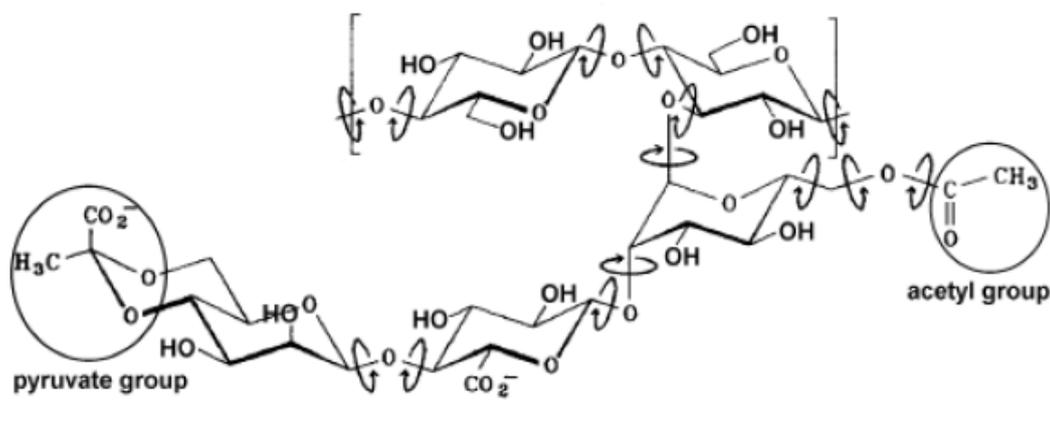


Figure 2.1: Basic structure of Xanthan gum (Ekeledirichukwu, 2010)

The ordered conformation has been shown to be responsible for the extraordinary stability of the polymer (Ekeledirichukwu, 2010). Pyruvate content, pH and particularly saline solution affect the stability of this type of polymer. They change charge density and the charge-shielding effects of the polymer. Arendt and Kulicke in 1998 asserted that salt content contributes enormously to the ordered conformation of Xanthan gum and it is important for optimum functionality of Xanthan gum (Ekeledirichukwu, 2010).

Katzbauer (1997) highlighted some of the unique properties of xanthan gum as high viscosity yield, a distinct shear -thinning behaviour and high shear stability.

2.4.1 Shear Thinning

An addition small amount of biopolymers can reduce frictional resistance of water significantly. Solutions of Xanthan gum exhibit a time independent non-Newtonian flow behavior and are extremely pseudoplastic. Sohn et. al. (2000) asserted that application of different magnitude of

shear stress on xanthan gum-based fluid has a substantial impact on its apparent viscosity (Ekeledirichukwu, 2010).

Katzbauer (1998) explains this behavior of Xanthan gum by the conformational status of the polymer molecules. Strong hydrogen bonds subsist between the molecules of the polymer when the fluid is static or exposed to low shear rates which therefore result to high viscosity. An increase in the shear (deformation) rate disintegrates the hydrogen bonds between the polymer molecules into aggregates resulting in a reduction of apparent viscosities. Arendt et al. (1998) suggest that the fast recovery of the primary structure implies the reformation of non-covalent intermolecular bonds (Ekeledirichukwu, 2010).

2.4.2 Low Shear Rate Viscosity, LSRV

Relative to other biopolymers, xanthan gum shows highly lower shear rate viscosities. In Alaska North Slope (ANS) field, xanthan gum is presently applied in coiled-tubing drilling (CTD) fluids as a viscosifier. The LSRV can be as high as 80000cp (Gantt et al., 1998). This property is also attributed to the conformational changes explained by (Katzbauer, 1998). Since hole cleaning relies on the LSRV, any factor that affects the conformational status of the biopolymer would alter the functionality of the fluid (Ekeledirichukwu, 2010).

2.4.3 Shear Degradation of Xanthan Gum

Sohn et al. (2000) performed an experiment to analyse the effect of xanthan gum solution on drag reduction by applying rotating disk apparatus. They discovered that as concentration of xanthan gum in solution increases so does drag reduction increases (decreasing apparent viscosity). Drag reduction continue to increase with increasing concentration of this polymer till a critical concentration is achieved where further increment in concentration will not have a substantial impact on drag reduction. However, if the molecular-weight of xanthan is high, lesser amount is required to increase drag reduction (Ekeledirichukwu, 2010).

Drag reduction experiments repeated on samples after one hour, indicated significant decreases in drag reduction coefficient. Increases in turbulence (high shear forces) led to decrease in drag reduction ability of the Xanthan gum. This indicates that there could be a distortion of the polymer structure as a result of exposure to high shear forces. Hunston and Zarkin (1980) noted that in turbulent flow, polymers in solution are exposed to elongational strain and to strong shear stresses. This mechanical energy causes a scission of the polymer chains. This leads to a decrease in the polymers' ability to enhance flow (Ekeledirichukwu, 2010).

2.5 THEORY OF THE RHEOLOGICAL MODELS

Rheology is the study of deformation and flow of matter including liquid and solid (Baker Hughes Drilling Fluids, 2006). Rheological models provide an approximate description of the behaviour of fluids by expressing a mathematical relationship between shear stress and shear rate. For Newtonian fluids the relationship between shear stress and shear rate (deformation rate) is linear and the constant of proportionality is effective viscosity. For Non-Newtonian fluids effective viscosity is not constant but shear rate dependent (see figure 2.2). Non-Newtonian fluids are generally characterized as pseudoplastic, dilatants, bingham plastics, thixotropic and so on. Figure 2.3 shows the behaviour

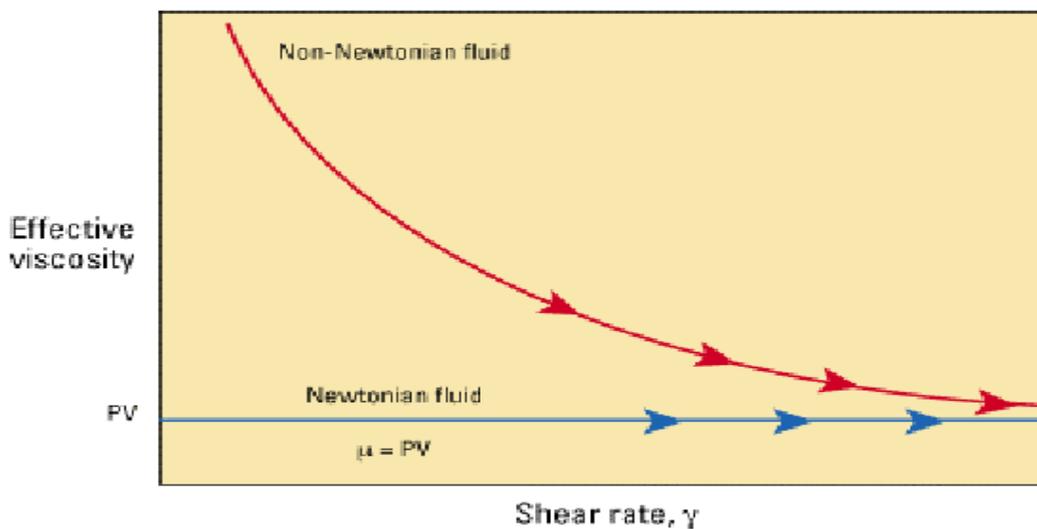


Figure 2.2: Comparison of Non-Newtonian and Newtonian fluid effective viscosity (Larsen, 2007)

of different types of fluids (Larsen, 2007). Most drilling fluid are fluid are non-Newtonian, and hence their viscosity is dependent on shear rate. A lot of rheological models have been used to describe Non-Newtonian fluids. The models applied in this work are discussed below.

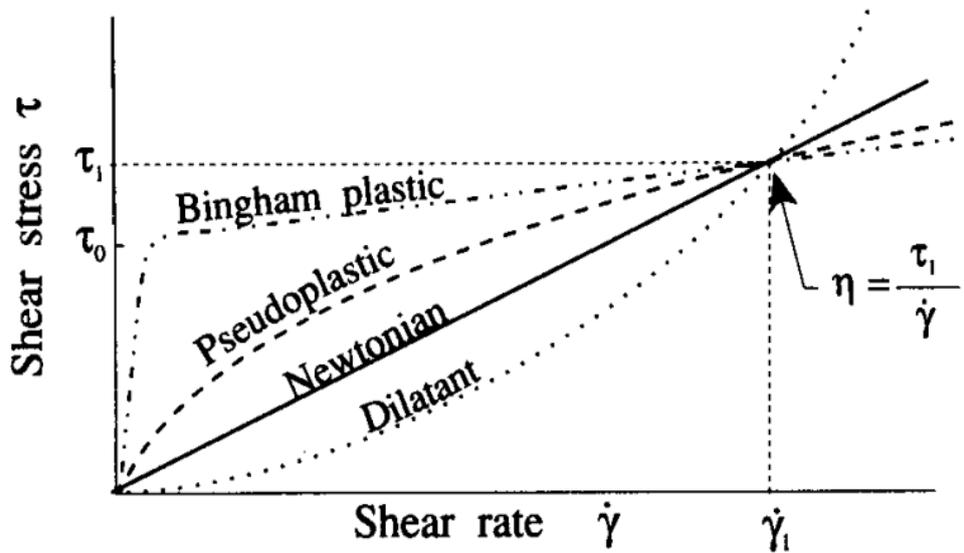


Figure 2.3: Rheogram of fluids behaviour (Larsen, 2007)

2.5.1 Bingham Plastic Model

This is a two-parameter rheological model that is applied to describe the behaviour of most drilling fluid. The mathematical expression of the model is shown in the equation below.

$$\tau = \tau_o + \mu_p \gamma \quad (2.1)$$

Where;

τ = Shear stress

τ_o = Yield point

μ_p = Plastic viscosity

γ = Shear rate

Fluids that depict the behaviour of Bingham Plastic are characterized by plastic viscosity and yield point which is not dependent on shear rate. The yield point is a critical stress that has to be exceeded before flow of fluid starts. It is a measure of attractive forces that exist within the fluid under dynamic conditions. Fluids that show the characteristics of this model are called Bingham plastic fluids. Most drilling fluids made up of clay suspension and some cement slurries exhibit this

behaviour. The plastic viscosity should be low to enhance fast drilling (increase in ROP). The plastic viscosity can be reduced by reducing the amount of colloidal solids in the drilling fluid. The yield point of the fluid should be high to ensure high carrying capacity but not too high to a point where pressure required to pump the fluid will be high (Larsen, 2007).

2.5.2 Power Law Model

This model is also a two-parameter rheological model to describe the characteristic of pseudoplastic fluids. This model is one of the widely used models in the oil and gas industry. The mathematical equation for the description of power law model is formed by replacing the viscosity term in Newtonian model by consistency index, K which is analogous with effective viscosity. Power law mathematical model is expressed as,

$$\tau = K \gamma^n \quad (2.2)$$

where consistency index, K and flow behavior index, n are the model parameters.

Fluids that behave according to this model are called power law fluids. The consistency index, K describes the thickness of the fluid and flow behaviour index also called power law index indicates the level of non-Newtonian behaviour. If $n=1$, the model becomes similar to Newtonian model. Moreover, $n>1$ indicates a dilatant type of fluid while pseudoplastic (shear thinning) fluid would exist if n becomes less than 1 (see figure 2.3). Power law model fits water base polymer mud especially those made up of XC Polymer better than Bingham Plastic model (Larsen, 2007). Power law model best describes the behaviour of most drilling fluid at a low shear rate not at high shear rate.

2.5.3 Herschel-Bulkley Model

The Herschel-Bulkley model is a three-parameter rheological model for describing the behaviour of non-Newtonian fluids. It is a power-law model with yield stress. It is a complex model which is recently used as it is notable to give accurate results than the simple Bingham and Power Law model when enough experimental data are available. The model is mathematically expressed as:

$$\tau = \tau_0 + K \gamma^n \quad (2.3)$$

where:

τ_0 = Yield point(stress)

K = Consistency index

n= flow behavior index

The main challenge in the use of this model and other non-linear curve model is curve fitting to evaluate model parameters. But the advent of computer and non-linear curve fitting has aided in solving this problem. Usually, the value of the yield stress used is the 3rpm readings from the Fann viscometer. 300 rpm or 600 rpm Fann viscometer readings are used to compute n and K or they can be estimated graphically. Although a certain level of stress is required to begin flow, this stress value becomes less with increasing shear (Larsen, 2007).

2.5.4 Robertson-Stiff Model

Robertson-Stiff model is a three-parameter model which is expressed in form of the equation shown below:

$$\tau = A(\gamma_0 + \gamma)^B \quad (2.4)$$

Where;

A, γ_0 and B are model parameters

$\tau_0 = A \gamma_0^B$ is the yield stress

The Robertson-Stiff Model includes the gel strength as a parameter and is used to a limited extent in the oil industry (Baker Hughes drilling fluids, 2006). It combines the Power law and Bingham Plastic Model. The model tends to Bingham plastic model if B=0; and behaves like power law model when γ_0 approaches zero. It has the advantages of Bingham and power law model, which can well describe the rheology of drill fluid under high or low shear rate.

Longlian and Jingxinyuan in 2010 selected Robertson-Stiff model as the best model to describe the rheological behaviour of non-zero drilling fluid in Managed Drilling Pressure (MPD) operation for Narrow Pressure Windows. Power law, Bingham plastic, Herschel-Bulkley, Casson and Robertson-Stiff model were analysed on the on-site rheological measurement data. Considering the well drilled is a horizontal well, the effect of eccentricity on simulation results must be take into account. Robertson model was then selected as best candidate for search situation.

2.5.5 Prandtl-Eyring Model

An alternative to the Power-Law model is the Prandtl-Eyring Model which tends to a constant

viscosity, A in the limit of γ going to zero from equation 2.5. However, the viscosity function tends to zero as γ tends to infinity. The constitutive equation of the behaviour of time-independent non-Newtonian fluids by Prandtl-Eyring is given as follows:

$$\tau = A \sinh^{-1}(\dot{\gamma}/B) \quad (2.5)$$

where A and B are material (fluid) constants which are required to describe the shear behaviour of matters.

This particular type of model has been used by Khan et. al (1984) to correlate the viscosity of gas-free Arhabasca bitumen.

2.5.6 Sisko Model

It is one of the rarely used rheological models to describe the behaviour of drilling fluids to perform hydraulic calculations in the oil and gas industry. This is because the form of this model makes the derivation of tractable expressions for pressure drop as a function of flow rate nontrivial or impossible (Bailey and Peden, 2000). The solution of their expression required rigorous computation. The constitutive law is expressed in equation 2.6 below. It is a three-parameter model where a, b and c are estimated for curve fitting.

$$\tau = a \dot{\gamma} + b \dot{\gamma}^c \quad (2.6)$$

Weir and Bailey (1996) statistically investigated twenty different rheological models on four different types of drilling fluid. After ranking of the models, Sisko model was selected as overall best fit for the selected fluids. They continued to derive a generalized consistent pressure loss equation which is independent on the type of rheological model for flow of fluids in a pipe and concentric annulus during laminar flow regime.

2.5.7 Modified Sisko Model

This is a current four-parameter rheological model based on the sisko model described above. The mathematical expression of this model is defined by equation (2.7). Guo and Hong (2010) asserted that this model is capable to represent accurately the rheogram of drilling fluids with its four parameters since it combines Bingham Plastic, Power Law and Sisko model.

$$\tau = \tau_o + a\gamma + b\gamma^c \quad (2.7)$$

From the above equation τ_o , a , b and c are the model parameters and the apparent viscosity μ_a is given by,

$$\mu_a = \tau / \dot{\gamma} = \tau_o \dot{\gamma}^{-1} + a + b\dot{\gamma}^{c-1} \quad (2.8)$$

where;

τ_o is the yield stress which stand for the shear stress of drilling fluid which need to be overcome flow before flow starts,

a is the coefficient of viscosity, which represents viscosity of the part of Newtonian fluid, that is, infinite shear rate viscosity,

b is the consistency coefficient ($\text{pa} \cdot \text{s}^c$), which represents viscosity of the part of non-Newtonian fluid, the lower shear rate correspond to the higher effect, and

c is the flowing behavior index (dimensionless). If $c < 1$, it represents the pseudoplastic fluid. If $c > 1$, it is the dilatant fluid. If $c = 1$, it may represents the Newtonian fluid.

Okafor and Evers (1992) and Weir and Bailey (1996) presented papers to discuss the elements of this model to in order to assess it.

2.5.8 Casson Model

The casson model is one of the rheological models used to describe the flow properties of non-Newtonian drilling fluid tough used in lesser extent in the oilfied. This model looks similar to Bingham Plastic model but it combines a yield stress with greater shear-thinning behaviour than the Bingham plastic model (Davison et. al., 1999). It is mathematically expressed as:

$$\tau = (\sqrt{\tau_o} + \sqrt{\mu\dot{\gamma}})^2 \quad (2.9)$$

The parameters τ_o and μ_p are needed to characterized the drilling fluid. The point at which the Casson curve intercepts the shear stress axis varies with the ratio of the yield point to the plastic viscosity (Baker Hughes drilling fluids, 2006).

Houwen and Geehan (1986) performed a rigorous study on the rheology of oil-based mud (OBM) using Bingham Plastic, Herschel-Bulkley and Casson taken into consideration the effects of temperature and pressure. They concluded that Herschel-Bulkley and Casson models fitted the data very well. However, Casson model was selected since it fitted better at high shear rate, which could be more precise if extrapolation is needed. It was also easier to associate the model to the drilling

fluid composition. Wanneng et. al (1986) studied the high shear rate behaviour of unweighted bentonite fluids and found that the Casson model gave the best fit.

2.6 REGRESSION ANALYSIS AND MODEL COMPARISON

Regression analysis is a conceptually simple method for investigating functional relationship among variables (Samprit and Ali, 2006). The function that relates dependent variables to independent variables can be linear and non-linear and hence, resulting in the name linear and non-linear regression analysis.

Some of the methods in regression analysis to estimate model parameters are the least-square method, the maximum likelihood method, the ridge method, Lagrangian Interpolation, piecewise linear polynomials or splines and the principal components method. Lagrangian Interpolation does not guarantee a better approximation of function when the polynomial degree gets large. This pitfall can be solved by composite interpolation (such as piecewise linear polynomials or splines). However, neither is desirable to extrapolate information from available data, that is, to generate new values at points lying outside the interval where interpolation nodes are given (Quarteroni and Saleri, 2006).

Least-square method is the commonly used method for function approximation. This is because standard non-linear (and linear) regression is always almost based on the assumption that the scatter follows a Gaussian (Normal) distribution. Given this particular assumption, it can be proved that the most likely model parameter values can be estimated by least-square method (Motulsky and Christopoulos, 2003). Maximum likelihood method tends to over-fit data and hence very bias.

Wier and Bailey (1996), (Bailey and Peden, 2000), Guo and Hong (2010), and Ali et. al (2001) used mean sum-of-squares criterion to select rheological models that best fit a given viscometer data for various types of drilling fluid used in the oilfield. Model with the minimum mean sum-of-squares was selected as the best fit to estimate model parameters.

Aswad and Saleh (1990) proposed that absolute percentage error (AAPE) should be employed as the most suitable statistical technique to select the rheological model that fit a given non-Newtonian fluid behaviour. They considered that the model which gives the lowest average absolute percentage error (AAPE) as the best rheological model. Their method was applied by Jawad and Akgun (2002) as a criterion to select best rheological model among six adopted rheological models for a non-

Newtonian drilling fluids.

Other authors also used value of coefficient of determination, R^2 to quantify goodness of fit. Kvalseth (1985) discussed the use of R^2 as a measure of goodness-of-fit in ordinary least-squares (OLS) regression. He identified that various alternative definitions that are suggested in statistical and data-analytic literature, are in general, not the same. However, Draper (1984) suggested that R^2 was misleading in data sets which replicate data points and Box-Wetz (1973) was more appropriate although modified this decision later. Healy (1984) asserted that R^2 is an unsuitable measure of OLS regression relationship and that an “absolute rather than relative is to be preferred”. He highlighted that this misleading is more pronounced in any non-linear model. Motulsky and Christopoulos (2003) in their paper cautioned that mistake should not be made to use R^2 as your main criterion for whether a fit is reasonable. A high R^2 indicates that the curve came very close to the points, but does not indicate that the fit is sensible in other ways. The best-fit values of the parameters may have values that make no sense or the confidence intervals may be very wide.

Motulsky and Christopoulos (2003) continued to recommend the use of statistical inference like hypothesis test (t-test, F-test, and Z-test) and confidence intervals to compare models after regression.

CHAPTER THREE

DEVELOPMENT OF STATISTICAL MODEL

3.0 INTRODUCTION

As stated in chapter one of this study, the main objective is to select a best-fit rheological model to describe the characteristics of biopolymer drill-in fluids from field data. This objective is achievable by developing a statistical regression model to fit the observed data points. The various rheological models have been posed deterministically. Obviously this is unrealistic and so these deterministic models are replaced with statistical models by adding an error (disturbance) term and making suitable assumptions about them. This results in a 'statistical regression model'. The rest of the chapter discuss how the model was developed.

3.1 DATA COLLECTION

Rheological data of xanthan based biopolymer drill-in fluid from rotational viscometer readings were collected. Equations 3.1a and 3.1b are used to determine the shear stress and shear rate values. Using equations 3.2a and 3.2b the dial readings were converted to shear stress (τ_i) in Ibf/100ft² and shear rate (γ_i) in second⁻¹ respectively. Table A1 in Appendix A shows the experimented Fann viscometer readings, shear stress and shear rate data.

$$\tau = \frac{360.5\theta}{2\pi r^2} \quad (3.1a)$$

Where,

τ = shear stress

θ = rotational viscometer dial readings in degrees

r = any radius between the bob radius r_1 and the rotor radius r_2 of the viscometer in cm

h = length of bob in cm

$$\gamma = \frac{5.066S}{r^2} \quad (3.1b)$$

A standard Fann viscometer with a bob radius of 1.7245 cm and bob length of 3.8 cm is used. If these values are substitute in equation 3.1a and shear stress is converted to Ibf/100ft², equation (3.1a) becomes:

$$\tau = 1.067 \theta \tag{3.2a}$$

Where τ is in Ibf/100ft²

By substituting $r=r_1= 1.7245$ cm into equation 3.1b:

$$\gamma = 1.703S \tag{3.2b}$$

Where,

γ = shear rate in sec⁻¹

S = speed of rotation of outer cylinder of the viscometer in rpm

3.2 MODEL SPECIFICATION

Models that relate shear stress to set of shear rates were selected. These models are specified as a function of form $f(\gamma_1, \gamma_2, \dots, \gamma_N)$ but still depend on unknown parameters $(\beta_1, \beta_2, \dots, \beta_q)$. The model function can be linear or non-linear. Ten popular rheological models were selected and analysed. For this work, apart from Bingham Plastic rheological model the rest of the model functions are non-linear. A list of rheological models employed is shown in Table A2 of Appendix A.

There is a functional relationship between the shear stress and shear rate in the models used. Therefore, the values of shear stress(τ) to be predicted by each model is a function of shear rate (γ) and q number of parameters ($\beta=\beta_1, \beta_2, \beta_3, \dots, \beta_q$) to be estimated in each model. But practically, readings of data are accompanied by some amount of errors (ϵ) which might result from poor measurements and instrument error. These errors are assumed to be random constituting the discrepancies in the models approximation. These errors are added to the model function to cater for the failure of the model to fit the experimental data exactly. Hence, a general statistical regression model is formed as shown in equation 3.3 below to approximate the relationship between shear stress and shear rate.

$$\tau = f(\gamma, \beta) + \epsilon \tag{3.3}$$

Where,

τ is shear stress in Ibf/100ft²

γ is shear rate in sec⁻¹

β is the value of model parameter

ϵ is random error in Ibf/100ft²

3.3 CHOICE OF FITTING METHOD AND MODEL FITTING

The next task is estimation of model parameters after collection of relevant data and defining the models to be used. Least-squares approximation method was used to performed regression analysis to estimate parameters in each model based on the given data sets. Least-square method was used due to the following assumption made about the data and the regression model:

- i. The scatter follows a Gaussian (normal) distribution
- ii. Errors are random errors that are independent and identically distributed with mean of zero and variance, σ^2 .

In Least-square we look for a function (model) that minimizes the sum-of-squares of vertical distances (residuals) between the fitted model regression line and the observed data points. Considering N number of data points (τ_i, γ_i) , least-square is expressed mathematically in equation 3.4 below.

$$\text{RSS}(\beta) = \sum_{i=1}^N (\tau_i - f(\gamma_i, \beta))^2 = \varepsilon^2 \quad (3.4)$$

Where;

RSS (β) is the residual sum of squares

β is the value(s) of model parameters that gives minimum RSS (also called least square estimators).

β has to be determined so that RSS (β) will be minimum. Therefore, for the sum of squares to be minimum,

$$\frac{\partial \text{RSS}(\beta)}{\partial \beta} = 0 \quad (3.5)$$

Equations 3.4 and 3.5 were used to estimate model parameters and sum of squares (RSS) of each model. For non-linear models, the aforementioned equations were solved using iterative estimation algorithm. Matlab code was developed to optimize the system of non-linear equation derived from each model equation by Newton optimization method. Detailed procedure of how each model was applied is as follows:

- i. A relationship (statistical correlation) between the shear rate and shear stress data points were determined before each model function is fitted to Fann viscometer data points
- ii. Functions for equation 3.5 for each model were created in Matlab.
- iii. A quasi Newton's iterative algorithm was created to solve each model function (equation

- 3.4) by calling each function defined in step (ii) above.
- iv. Appropriate initial values for each model parameters were chosen by looking at a graph of their model function behaviour and constraints set for each parameter. Parameters constraints were formed with idea that shear stress are positive and increase with shear rate. Table A2 of Appendix A depicts the initial guess and constraints for the models.
 - v. The algorithms developed were run to solve (converge) each model and relevant output results well tabulated and plotted.
 - vi. Residuals of the fitted models were plotted to assess for goodness-of-fit.

3.4 STATISTICAL MEASURE

3.4.1 Model Comparison

Residual mean squares (equation 3.6) were employed as a statistical tool to account for the error variance because of small sample size (8),

$$\text{RMS} = \frac{RSS}{N - q} \quad (3.6)$$

Where;

RMS= residual mean squares or residual variance

N = Number of data points

q = Number of parameters in a model

N-q = df = degree of freedom in a fitted model

RMS was used as a performance measure of each model. The model with a minimum RMS was selected as most likely model to describe the behaviour of biopolymer based drill-in fluids.

3.4.1 Confidence Interval

Confidence interval of the selected model function of fitted shear stress values was also estimated. To solve any of model function fitted to a measured viscometer data is dependent on estimation of rheological model parameter. However, these measured data are subjected to instrument measurement or reading error. It is therefore conceivable to quantify the degree of certainty attached to the fitted functions by calculating level of confidence interval and significant test. This is computed by statistical formula developed by Gallant (1985) to approximate the true confidence interval (100(1- α) %) of non-linear function of concern. This method is applied as follows;

Let $h(\beta)$ be the nonlinear function of interest that is obtained using the rheological model parameters, β . Then, using the results of Gallant (1985), an approximate $100 \times (1-\alpha)$ % confidence interval estimate of the true value of the nonlinear function is given by

$$h(\beta) \pm t_{|(N-q)\alpha|} \sqrt{\hat{H}(\hat{F}^T \hat{F})^{-1} \hat{H}^T s^2}, \quad (3.7)$$

where

$$\hat{H} = \left(\frac{\partial[h(\beta)]}{\partial\beta_1} \quad \frac{\partial[h(\beta)]}{\partial\beta_2} \quad \dots \quad \frac{\partial[h(\beta)]}{\partial\beta_q} \right) \quad (3.8)$$

is the row vector of partial derivatives of $h(\beta)$ with respect to the rheological model parameters

$$\hat{F} = \begin{bmatrix} \frac{\partial[f(\dot{\gamma}_1; \beta)]}{\partial\beta_1} & \frac{\partial[f(\dot{\gamma}_1; \beta)]}{\partial\beta_2} & \dots & \frac{\partial[f(\dot{\gamma}_1; \beta)]}{\partial\beta_q} \\ \frac{\partial[f(\dot{\gamma}_2; \beta)]}{\partial\beta_1} & \frac{\partial[f(\dot{\gamma}_2; \beta)]}{\partial\beta_2} & \dots & \frac{\partial[f(\dot{\gamma}_2; \beta)]}{\partial\beta_q} \\ \vdots & \vdots & \ddots & \vdots \\ \frac{\partial[f(\dot{\gamma}_N; \beta)]}{\partial\beta_1} & \frac{\partial[f(\dot{\gamma}_N; \beta)]}{\partial\beta_2} & \dots & \frac{\partial[f(\dot{\gamma}_N; \beta)]}{\partial\beta_q} \end{bmatrix} \quad (3.9)$$

F is the $N \times q$ matrix of partial derivative of $h(\beta)$ written in terms of the rheological model evaluated at β and N data points $\dot{\gamma}_1$. s^2 is the estimated error variance given by the RMS value and $t_{|(N-q)\alpha|}$ is the t-distribution value corresponding to the significance level α . It has $(N - q)$ degrees of freedom. The accuracy of above approximation will, of course, increase with small sample size. For small data sets (as exist with fitted rheological models where typically eight, or fewer, samples are available) close-to-linear model behaviour is necessary to ensure that the above formulas are valid.

CHAPTER FOUR

APPLICATION OF MODEL EQUATIONS ON DATA

4.1 INTRODUCTION

This section covers the detailed application of the general statistical regression model to each rheological model function using least-square approximation of function. Model parameters were estimated from the data shown in Table 1A of appendix A. In least-square regression we look for a function of form $f(\gamma, \beta)$ that relates shear stress (dependent variable) to shear rate (independent variable) such that the sum-of-squares of error is minimized. For the square of the error to be minimal, the partial derivative of the function with respect to each model parameters has to be asymptotically approximately equal to zero.

The partial derivatives with respect to the model parameters resulted in systems of equations which were solved simultaneously to estimate the parameters in each model. Most of the functions of the rheological models applied in this study are non-linear which can only be solved numerically by developing iterative algorithms. Matlab code was developed to optimize the system of non-linear equation of the partial derivatives with respect model parameters derived from each model equation by a quasi Newton's iterative optimization method. The system of non-linear equations was evaluated by choosing the right initial guess of parameters and selecting appropriate tolerance level for to allow the function of each equation to converge. Each model parameter estimated from the given data is shown in Table 5.1.

Extensive analysis of the results obtained from application of equations on the data is discussed in Chapter five.

Detailed equations regarding each model together with the system of partial differential equations evaluated in Matlab Software Environment is as follows:

4.1 EVALUATION OF BINGHAM PLASTIC MODEL

$$\tau = \tau_o + \mu_p \gamma \quad (4.1)$$

Parameters τ_o and μ_p were estimated

$$f(\gamma, \beta) = f(\gamma, \tau_o, \mu_p) = \tau_o + \mu_p \gamma \quad (4.2)$$

$$RSS = \sum_{i=1}^N (\tau_i - \tau_o - \mu_p \gamma_i)^2 \quad (4.3)$$

$$\left\{ \frac{\partial RSS}{\partial \tau_o} = \sum_{i=1}^N (\tau_i - \tau_o - \mu_p \gamma_i)^2 = 0 \right. \quad (4.4a)$$

$$\left. \frac{\partial RSS}{\partial \mu_p} = \sum_{i=1}^N (\tau_i - \tau_o - \mu_p \gamma_i) \gamma_i = 0 \right. \quad (4.4b)$$

The system of equation (4.4a and 4.4b) was solved in a linear least-square sense using Matlab to estimate the unknown model parameters from the given rheological data in Table A1 (Appendix A).

The parameters were used to evaluate the model function and RSS.

4.2 EVALUATION OF POWER LAW MODEL

$$\tau = K\gamma^n \quad (4.5)$$

Parameters K and n were estimated

$$f(\gamma, \beta) = f(\gamma, K, n) = K\gamma^n \quad (4.6)$$

$$RSS = \sum_{i=1}^N (\tau_i - K\gamma_i^n)^2 \quad (4.7)$$

$$\left\{ \frac{\partial RSS}{\partial K} = \sum_{i=1}^N (\tau_i - K\gamma_i^n) \gamma_i^n = 0 \right. \quad (4.8a)$$

$$\left. \frac{\partial RSS}{\partial n} = \sum_{i=1}^N (\tau_i - K\gamma_i^n) \gamma_i^n \ln \gamma_i = 0 \right. \quad (4.8b)$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the system of non-linear equation (4.8a through 4.8b) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

4.3 EVALUATION OF HERSCHEL – BULKLEY MODEL

$$\tau = \tau_o + K\gamma^n \quad (4.9)$$

Parameters estimated are τ_o, K and n

$$f(\gamma, \beta) = f(\gamma, \tau_o, K, n) = \tau_o + K\gamma^n \quad (4.10)$$

$$RSS = \sum_{i=1}^N (\tau_i - \tau_o - K\gamma_i^n)^2 \quad (4.11)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial \tau_o} = \sum_{i=1}^N (\tau_i - \tau_o - K\gamma_i^n) = 0 \\ \frac{\partial RSS}{\partial K} = \sum_{i=1}^N (\tau_i - \tau_o - K\gamma_i^n)\gamma_i^n = 0 \\ \frac{\partial RSS}{\partial n} \sum_{i=1}^N (\tau_i - \tau_o - K\gamma_i^n)\gamma_i^n \ln \gamma_i = 0 \end{array} \right. \quad \begin{array}{l} (4.12a) \\ (4.12b) \\ (4.12c) \end{array}$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the system of non-linear equation (4.12a through 4.12c) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

4.4 EVALUATION OF ROBERTSON - STIFF MODEL

$$\tau = A(\gamma_o + \gamma)^B \quad (4.13)$$

$$f(\gamma, \beta) = f(\gamma, \gamma_o, A, B) = A(\gamma_o + \gamma)^B \quad (4.14)$$

Parameters estimated are γ_o, A and B

$$RSS = \sum_{i=1}^N [\tau_i - A(\gamma_o + \gamma_i)^B]^2 \quad (4.15)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial A} = \sum_{i=1}^N (\tau_i - A(\gamma_o + \gamma_i)^B)(\gamma_o + \gamma_i)^B = 0 \\ \frac{\partial RSS}{\partial B} = \sum_{i=1}^N [\tau_i - A(\gamma_o + \gamma_i)^B](\gamma_o + \gamma_i)^B \ln(\gamma_o + \gamma_i) = 0 \\ \frac{\partial RSS}{\partial \gamma_o} = \sum_{i=1}^N [\tau_i - A(\gamma_o + \gamma_i)^B](\gamma_o + \gamma_i)^{B-1} = 0 \end{array} \right. \quad \begin{array}{l} (4.16a) \\ (4.16b) \\ (4.16c) \end{array}$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the system of non-linear equation (4.16a through 4.16c) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

4.5 EVALUATION OF MODIFIED ROBERTSON-STIFF MODEL

$$\tau = \tau_o + A(\gamma_o + \gamma)^B \quad (4.17)$$

Parameters τ_o, A, γ_o and B were estimated

$$f(\gamma, \beta) = f(\gamma, \tau_o, \gamma_o, A, B) = \tau_o + A(\gamma_o + \gamma)^B \quad (4.18)$$

$$RSS = \sum_{i=1}^N [\tau_i - \tau_o - A(\gamma_o + \gamma_i)^B]^2 \quad (4.19)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial A} = \sum_{i=1}^N [\tau_i - \tau_o - A(\gamma_o + \gamma_i)^B] (\gamma_o + \gamma_i)^B = 0 \end{array} \right. \quad (4.20a)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial B} = \sum_{i=1}^N [\tau_i - \tau_o - A(\gamma_o + \gamma_i)^B] (\gamma_o + \gamma_i)^B \ln(\gamma_o + \gamma_i) = 0 \end{array} \right. \quad (4.20b)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial \gamma_o} = \sum_{i=1}^N [\tau_i - \tau_o - A(\gamma_o + \gamma_i)^B] (\gamma_o + \gamma_i)^{B-1} = 0 \end{array} \right. \quad (4.20c)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial \tau_o} = \sum_{i=1}^N [\tau_i - \tau_o - A(\gamma_o + \gamma_i)^B] = 0 \end{array} \right. \quad (4.20d)$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the system of non-linear equation (4.20a through 4.2d) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

4.6 EVALUATION OF PRANDTL – EYRING MODEL

$$\tau = A \sinh^{-1}(\gamma/B) \quad (4.21)$$

Parameters A and B were estimated

$$f(\gamma, \beta) = f(\gamma, A, B) = A \sinh^{-1}(\gamma/B) \quad (4.22)$$

$$RSS = \sum_{i=1}^N [\tau_i - A \sinh^{-1}(\gamma_i/B)]^2 \quad (4.23)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial A} = \sum_{i=1}^N [\tau_i - A \sinh^{-1}(\gamma_i/B)] [\sinh^{-1}(\gamma_i/B)] = 0 \end{array} \right. \quad (4.24a)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial B} = \sum_{i=1}^N [\tau_i - A \sinh^{-1}(\gamma_i/B)] \frac{\gamma_i}{B^2 \left[\sqrt{1 + (\gamma_i/B)^2} \right]} = 0 \end{array} \right. \quad (4.24b)$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the system of non-linear equation (4.24a through 4.24b) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

4.7 EVALUATION OF MODIFIED PRANDTL – EYRING MODEL

$$\tau = \tau_o + A \sinh^{-1}(\gamma/B) \quad (4.25)$$

τ, A and B are model parameters estimated

$$f(\gamma, \beta) = f(\gamma, \tau_o, A, B) = \tau_o + A \sinh^{-1}(\gamma/B) \quad (4.26)$$

$$RSS = \sum_{i=1}^N [\tau_i - \tau_o - A \sinh^{-1}(\gamma_i/B)]^2 \quad (4.27)$$

$$\begin{cases} \frac{\partial RSS}{\partial \tau_o} = [\tau_i - \tau_o - A \sinh^{-1}(\gamma_i/B)] = 0 & (4.28a) \\ \frac{\partial RSS}{\partial A} = [\tau_i - \tau_o - A \sinh^{-1}(\gamma_i/B)] [\sinh^{-1}(\gamma_i/B)] = 0 & (4.28b) \\ \frac{\partial RSS}{\partial B} = [\tau_i - \tau_o - A \sinh^{-1}(\gamma_i/B)] \frac{\gamma_i}{B^2 \left[\sqrt{1 + (\gamma_i/B)^2} \right]} = 0 & (4.28c) \end{cases}$$

Given the rheological data (see Table A1, Appendix A), Matlab codes were developed to optimize the system of non-linear equation (4.28a through 4.28c) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

4.8 EVALUATION OF SISKI MODEL

$$\tau = a \gamma + b \gamma^c \quad (4.29)$$

Parameters to be estimated are a, b and c

$$f(\gamma, \beta) = f(\gamma, a, b, c) = a \gamma + b \gamma^c \quad (4.30)$$

$$RSS = \sum_{i=1}^N (\tau_i - a \gamma_i - b \gamma_i^c)^2 \quad (4.31)$$

$$\begin{cases} \frac{\partial RSS}{\partial a} = \sum_{i=1}^N [\tau_i - a \gamma_i - b \gamma_i^c] \gamma_i = 0 & (4.32a) \\ \frac{\partial RSS}{\partial b} = \sum_{i=1}^N [\tau_i - a \gamma_i - b \gamma_i^c] \gamma_i^c = 0 & (4.32b) \\ \frac{\partial RSS}{\partial c} = \sum_{i=1}^N [\tau_i - a \gamma_i - b \gamma_i^c] \gamma_i^c \ln \gamma_i = 0 & (4.32c) \end{cases}$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the system of non-linear equation (4.32a through 4.32c) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model

function and RSS.

4.9 EVALUATION OF MODIFIED SSKO

$$\tau = \tau_o + a \gamma + b \gamma^c \quad (4.33)$$

Parameters τ_o, a, b and c were estimated

$$f(\gamma, \beta) = f(\gamma, \tau_o, a, b, c) = \tau_o + a \gamma + b \gamma^c \quad (4.34)$$

$$RSS = \sum_{i=1}^N (\tau_i - \tau_o - a \gamma_i - b \gamma_i^c)^2 \quad (4.35)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial \tau_o} = \sum_{i=1}^N [\tau_i - \tau_o - a \gamma_i - b \gamma_i^c] = 0 \end{array} \right. \quad (4.36a)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial a} = \sum_{i=1}^N [\tau_i - \tau_o - a \gamma_i - b \gamma_i^c] \gamma_i = 0 \end{array} \right. \quad (4.36b)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial b} = \sum_{i=1}^N [\tau_i - \tau_o - a \gamma_i - b \gamma_i^c] \gamma_i^c = 0 \end{array} \right. \quad (4.36c)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial c} = \sum_{i=1}^N [\tau_i - \tau_o - a \gamma_i - b \gamma_i^c] \gamma_i^c \ln \gamma_i = 0 \end{array} \right. \quad (4.36d)$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the system of non-linear equation (4.36a through 4.36d) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

4.10 EVALUATION OF CASSON MODEL

$$\tau = (\sqrt{\tau_o} + \sqrt{\mu \gamma})^2 \quad (4.37)$$

Parameters τ_o and μ were estimated.

$$f(\gamma, \beta) = f(\gamma, \tau_o, \mu) = (\sqrt{\tau_o} + \sqrt{\mu \gamma})^2 \quad (4.38)$$

$$RSS = \sum_{i=1}^N (\tau_i - (\sqrt{\tau_o} + \sqrt{\mu \gamma_i})^2)^2 \quad (4.39)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial \tau_o} = \sum_{i=1}^N [\tau_i - (\sqrt{\tau_o} + \sqrt{\mu \gamma_i})^2] (\sqrt{\tau_o} + \sqrt{\mu \gamma_i}) \frac{1}{\sqrt{\tau_o}} = 0 \end{array} \right. \quad (4.40a)$$

$$\left\{ \begin{array}{l} \frac{\partial RSS}{\partial \mu} = \sum_{i=1}^N [\tau_i - (\sqrt{\tau_o} + \sqrt{\mu \gamma_i})^2] (\sqrt{\tau_o} + \sqrt{\mu \gamma_i}) \frac{\sqrt{\gamma_i}}{\sqrt{\tau_o}} = 0 \end{array} \right. \quad (4.40b)$$

Given the rheological data (see Table A1, Appendix A), Matlab code was developed to optimize the

system of non-linear equation (4.40a through 4.40b) using a quasi Newton's iterative numerical method to estimate the unknown parameters. The parameters were used to evaluate the model function and RSS.

CHAPTER FIVE

ANALYSIS OF RESULTS

5.0 INTRODUCTION

This chapter discusses the results obtained by applying the statistical regression model for each model function on the experimental rheological data of the aforementioned drill-in fluid as discussed in Chapter four. Plots of the fitted rheological models and their corresponding residuals are shown in this chapter and interpreted accordingly. Conclusions made basically depend on the interpretation of the relevant plots and tables and literature knowledge. The remaining results from this study are shown in Table A3 of Appendix A.

5.1 DETERMINATION OF STATISTICAL CORRELATION

Before the various functions were fitted to the data to model the relationship between the shear stress and shear rate, it was prudent to determine if a good statistical correlation relationship exists between these variables. The matrix in equation 5.1 shows the correlation-coefficient result. The diagonal matrix elements represent the perfect correlation of each variable with itself and are equal to 1. The off-diagonal elements are very close to 1, indicating that there is a strong statistical correlation between the variables shear stress and shear rate.

MATLAB calculated correlation-coefficient matrix is:

$$\begin{bmatrix} 1 & 0.909 \\ 0.909 & 1 \end{bmatrix} \quad (5.1)$$

The rheological data in Table A1 of Appendix A were used to perform the least-square regression analysis by applying the statistical model developed after the establishment of a strong statistical correlation between the variables shear stress and shear rate.

5.2 FITTED CURVES AND RESIDUAL ANALYSIS

Fitted plots of all the models are shown in figures 5.1a, 5.2a, 5.3a, 5.3b, 5.5a, 5.5b, 5.7a, 5.7b, 5.9a and 5.9b. The red dotted lines in the plots show the experimented rheological data points while the continuous green lines are for the fitted values of the rheological model functions. Residuals scatter plots of the models under study are also shown in figures 5.1b, 5.2b, 5.4a, 5.4b, 5.6a, 5.6b, 5.8a, 5.8b, 5.10a and 5.10b after performing least-square regression analysis on the given rheological

data. The residual plots are to some degree necessary to assess sufficiency of the functional part of the models especially when the number of data points is large. The reference line at 0 emphasizes that the residuals are split about 50-50 between positive and negative. Figure 5.1a and 5.1b show the Bingham Plastic model fitted to the rheological data and its residual plot, respectively. Deficiency in fitting is much clearly seen in the residual plot (figure 5.1b) since the ordinate axis

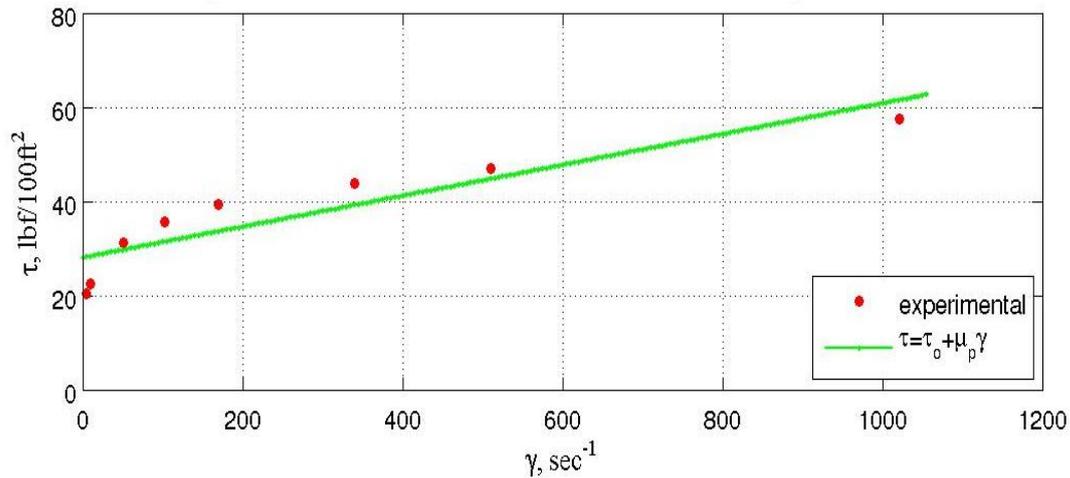


Figure 5.1a: Experimented rheological data and fitted Bingham Plastic values

is rescaled. If a critical look is made at the residual plot of the Bingham-Plastic model, it can be depicted that the data points are not randomly distributed above and below the curve. There are

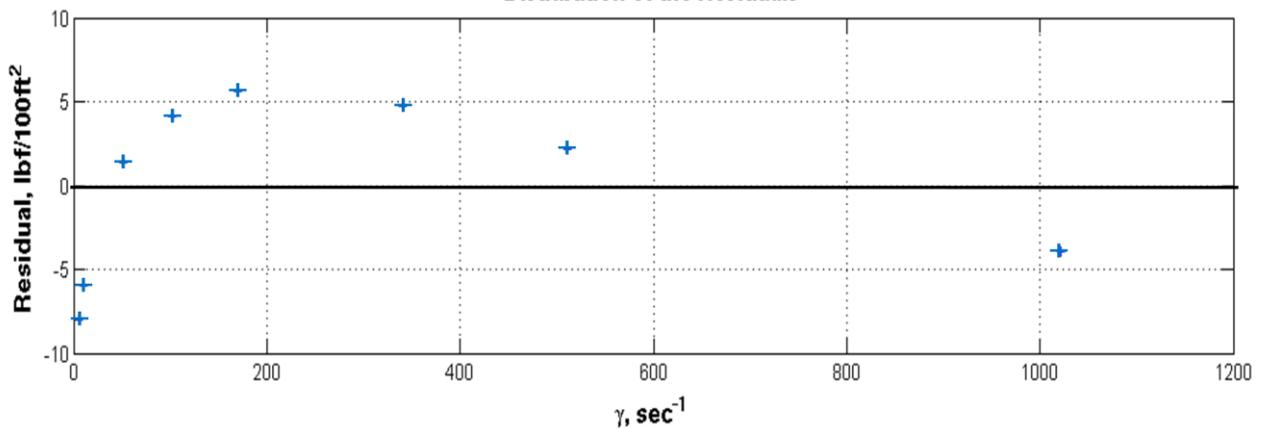


Figure 5.1b: Residual plot of fitted Bingham Plastic values.

clusters of points above the fitted regression line between the shear rate of 51.1 sec^{-1} and 510.9 sec^{-1} which are clearly observed in the residual plot. The fitted curve of Casson model (see figure 5.2a) is

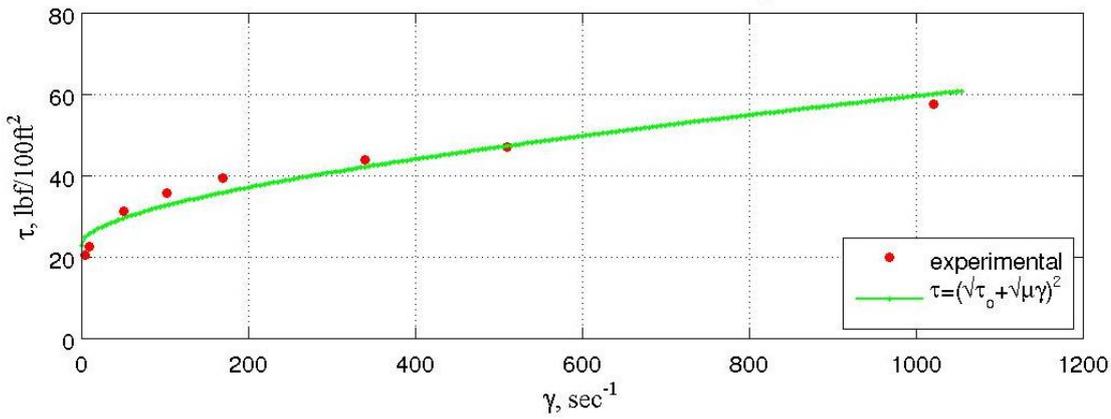


Figure 5.2a: Experimented rheological data and fitted Casson values

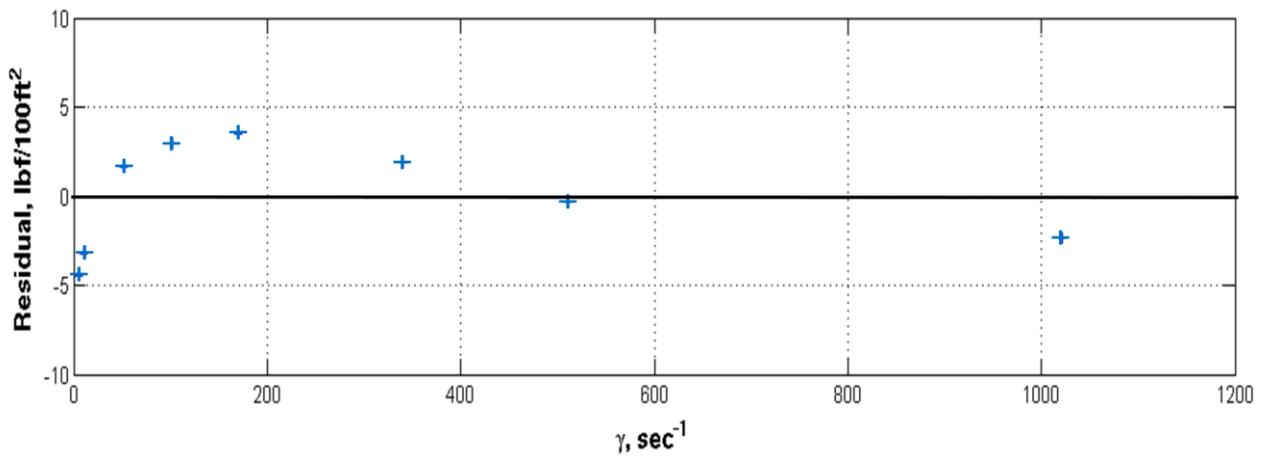


Figure 5.2b: Residual plot of fitted Casson values

similar to that of Bingham Plastic model but the former bends a bit at lower shear rates (0 to 10.2 sec^{-1}). Observations seen in the residual plot of the fitted Casson model shown in figure 5.2b were virtually indifferent from that of Bingham plastic model. It can be seen that these two models clearly does not fit the data very well.

The fitted flow curve for both Power Law model (figure 5.3a) and Herschel-Bulkley model(5.3b) looks similar but the curve of the latter passes through the origin while the former passes through an intercept (yield stress) initially which needs to be overcome before flow begins. A mere look at their fitted curves to determine how well they fit the rheological data will be quite difficult until their residual plots are made where the residual between the fitted values and raw data are rescaled on the ordinate axis for clear view.

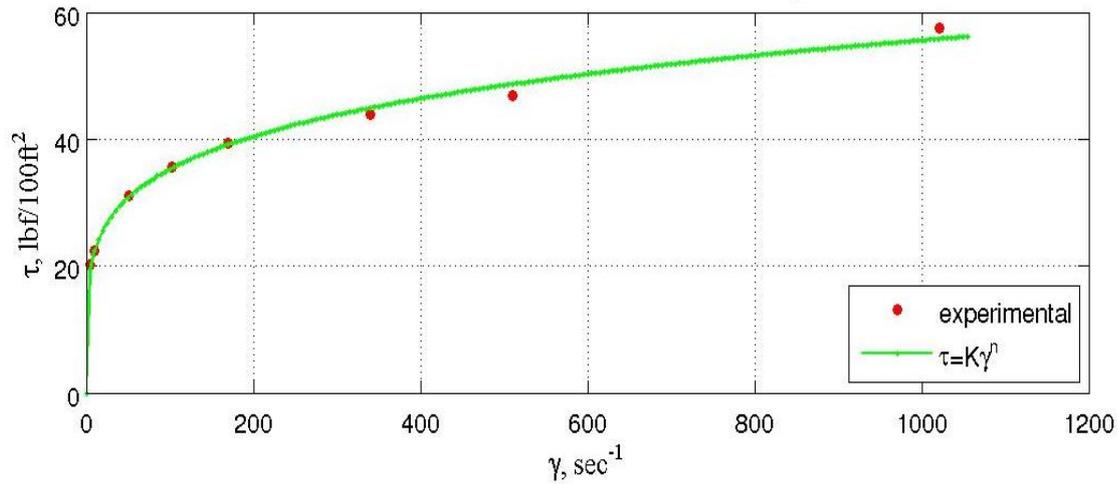


Figure 5.3a: Experimented rheological data and fitted Power law values

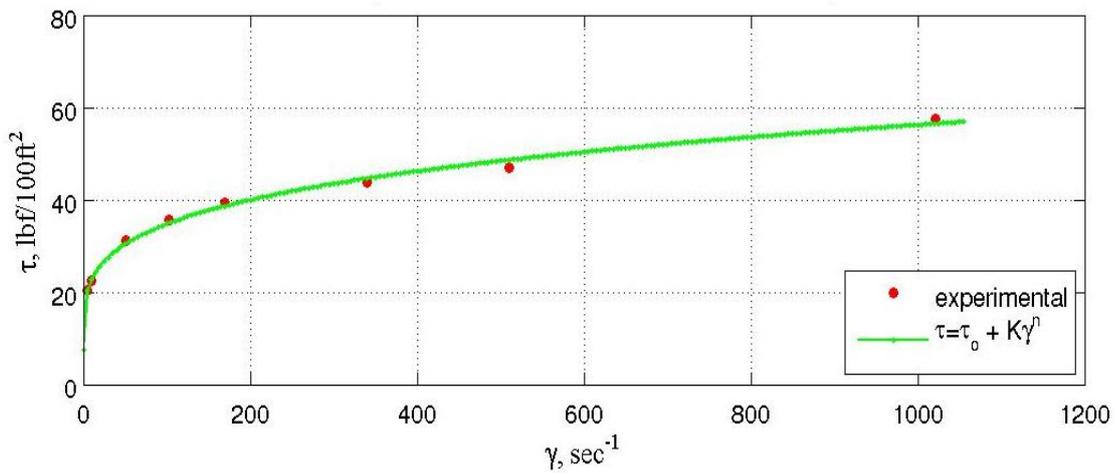


Figure 5.3b: Experimented rheological data and fitted Herschel-Bulkley values

The residual plots of Power law model (figure 5.4a) and Herschel-Bulkley model (figure 5.4 b) look quite similar according to their structural pattern formed. However, sectional analysis of the two

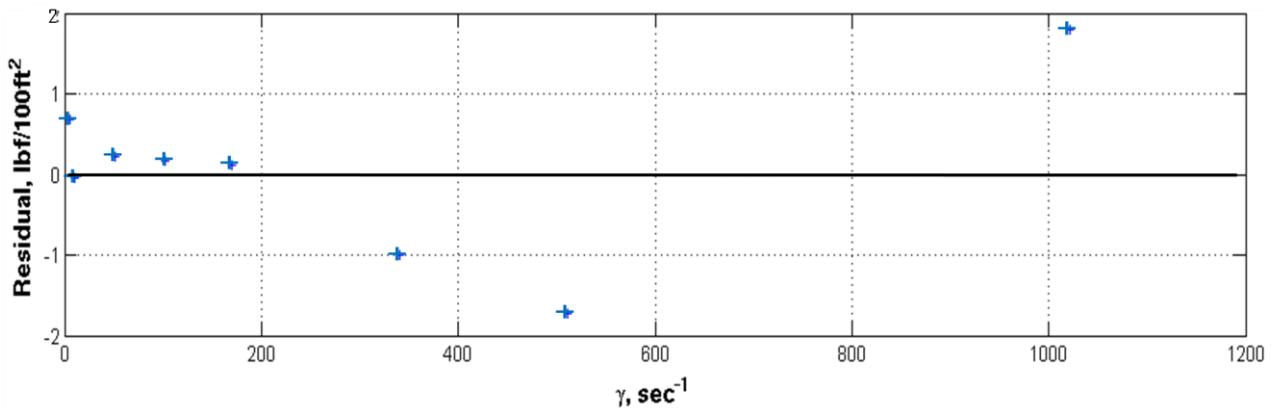


Figure 5.4a: Residual plot of fitted Power values

plots indicates that power law (PL) model describes data well at low shear rates (from 10.2 sec^{-1} to 170.3 sec^{-1}) than Herschel-Bulkley (HB) model. But at very low shear rate (less than 5.10 sec^{-1})

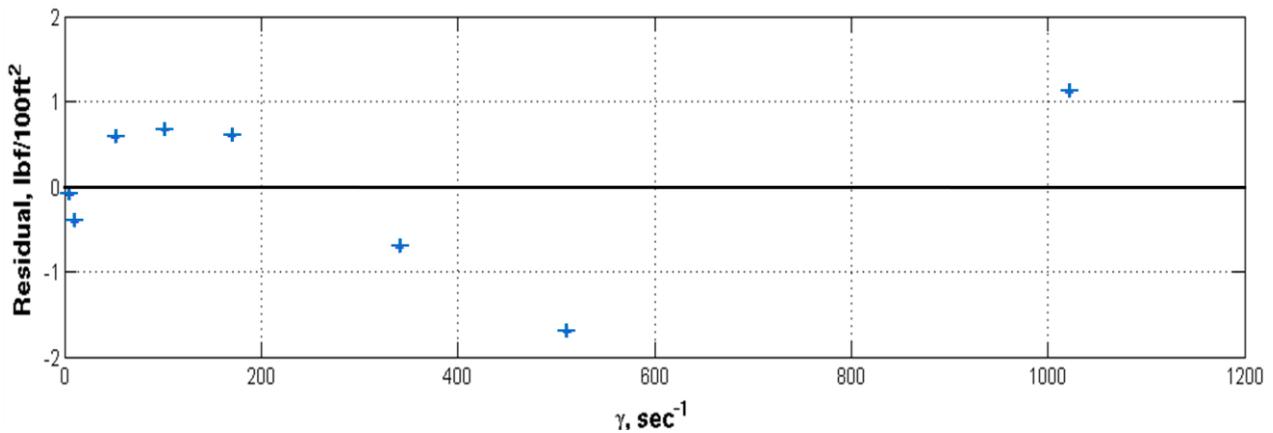


Figure 5.4b: Residual plot of fitted Herschel-Bulkley values

Herschel-Bulkley model captures the behaviour of the data very well. At very high shear rate within the range of 340.6 sec^{-1} to 1021.8 sec^{-1} HB model characterized the rheological behaviour of the biopolymer better than the PL model since their residuals are closer to the zero horizontal reference line in the residual plots. Hence, this model can be rely on to make accurate predictions at higher shear rates outside the range of readings that can be gotten from the Fann viscometer.

The rheogram of Robertson-Stiff and modified Robertson-Stiff are shown in figure 5.5a and 5.5b respectively. The shape of these curves look alike but their residuals plots reveal the insufficiency of their functional parts to the data.

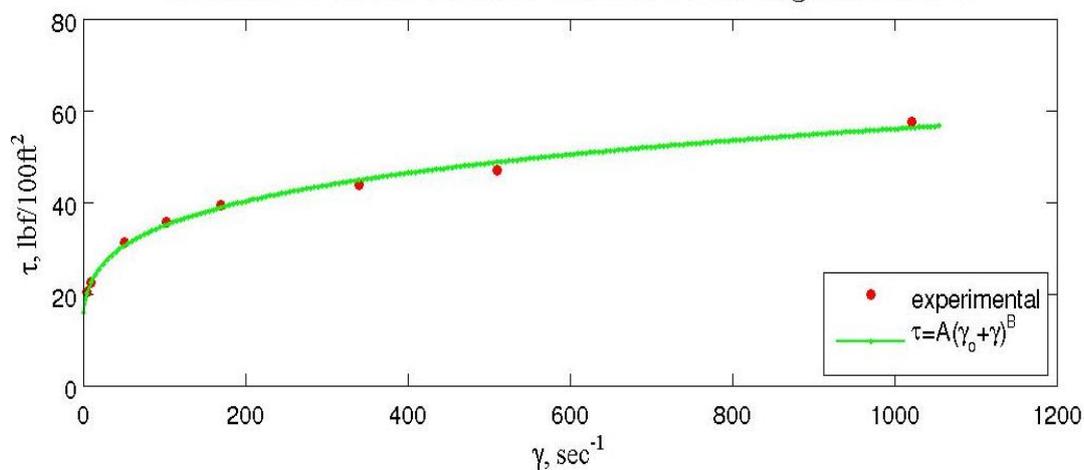


Figure 5.5a: Experimented rheological data and fitted Robertson-Stiff values

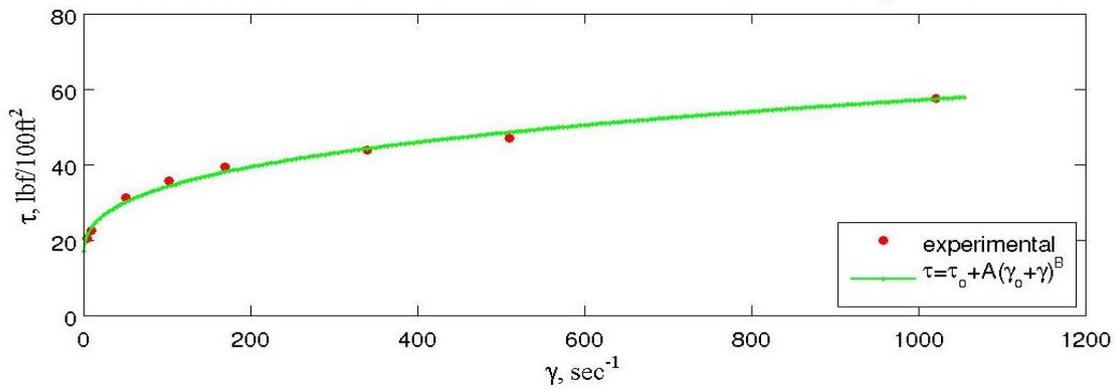


Figure 5.5b: Experimented rheological data and fitted Modified Robertson-Stiff values

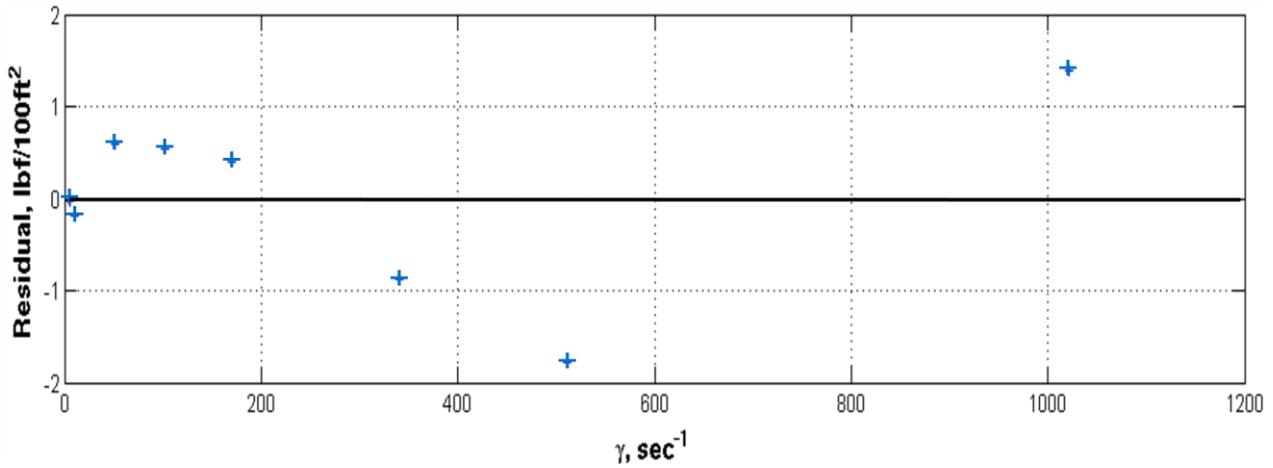


Figure 5.6a: Residual plot of fitted Robertson-Stiff values values

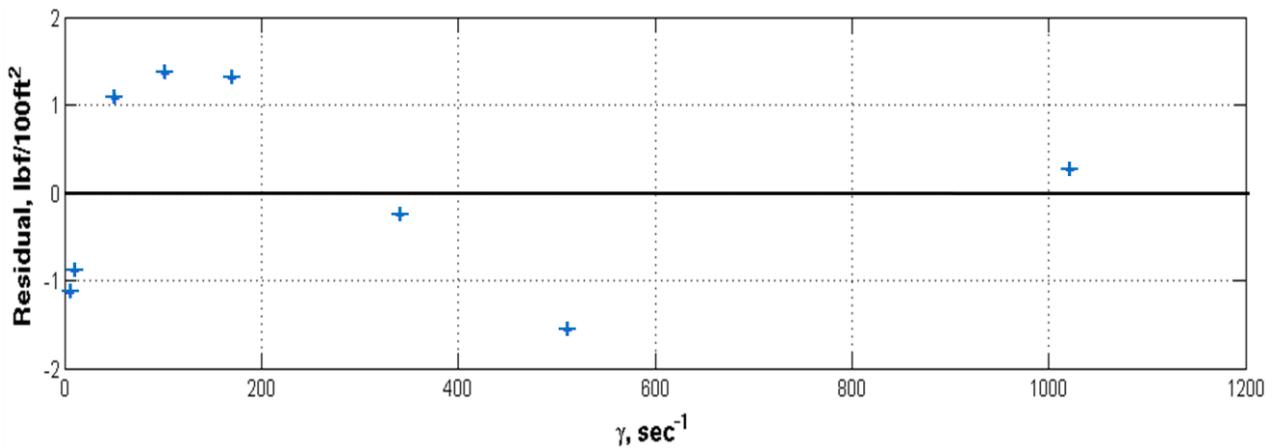


Figure 5.6a: Residual plot of fitted Modified Robertson-Stiff values

Robertson-Stiff (see figure 5.6a) and modified Robertson-Stiff (figure 5.6b) models residual plots almost gave the same scatter plots but the latter gives a better description as their fitted values at high shear rates (340.6sec^{-1} to 1021.8sec^{-1}) is closer to the horizontal reference line. The latter

however under-fits the data at low shear rates. These two models might not give a good fit compared to PL and HB since their residual plots follow a particular structured pattern.

Figure 5.7a and 5.7b show the flow curves of Prandt-Eyring(PE) and modified Prandt-Eyring(MPE) models respectively. But the MPE curve describes the rheological data far better than PE curve.

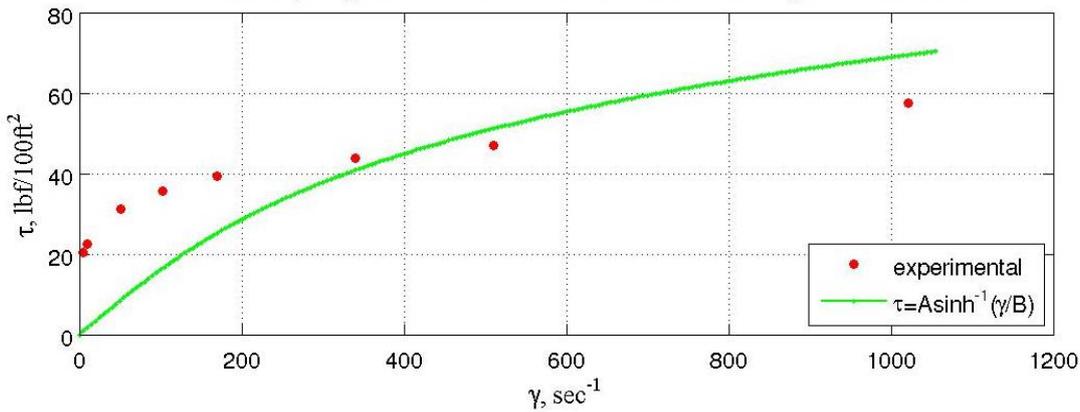


Figure 5.7a: Experimented rheological data and fitted Prandt-Eyring values

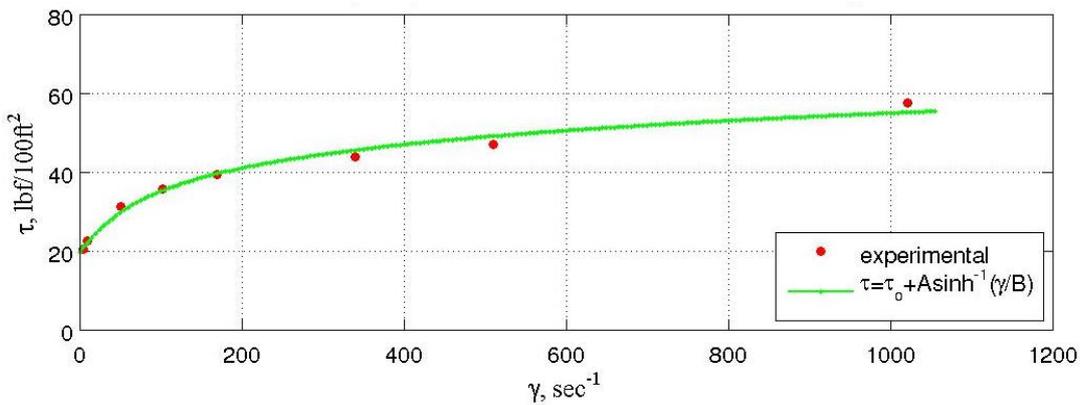


Figure 5.7b: Experimented rheological data and fitted Modified Prandt-Eyring values

This observation is clearer in their residual plots. PE (figure 5.8a) and MPE (figure 5.8b) models residuals follow a particular pattern which indicates that these models might not be the most-likely rheological models to describe the rheological behaviour of drill-in fluids under study. Moreover, most of the data points are above the reference line in the residual plots.

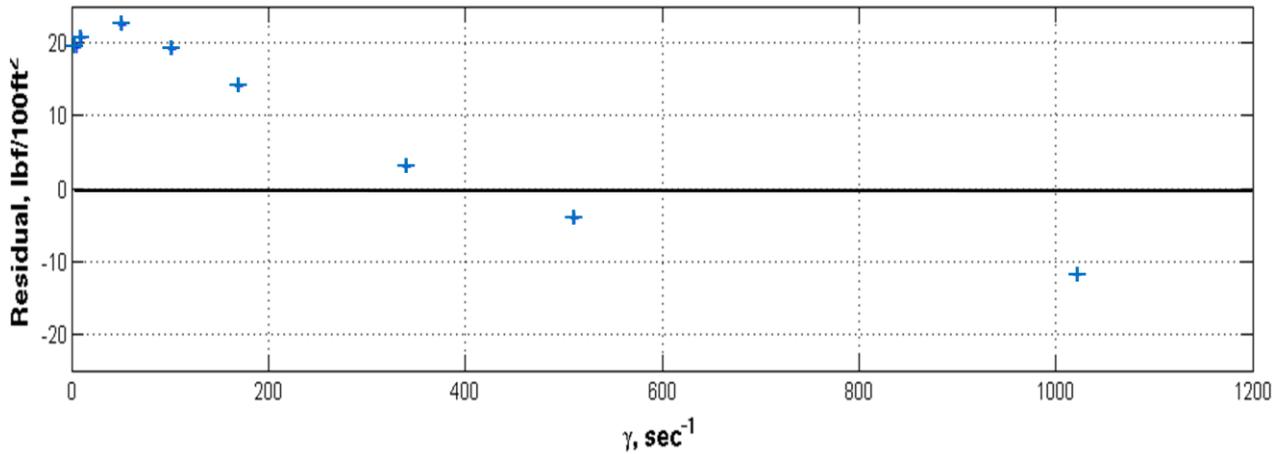


Figure 5.8a: Residual plot of fitted Prandtl-Eyring values

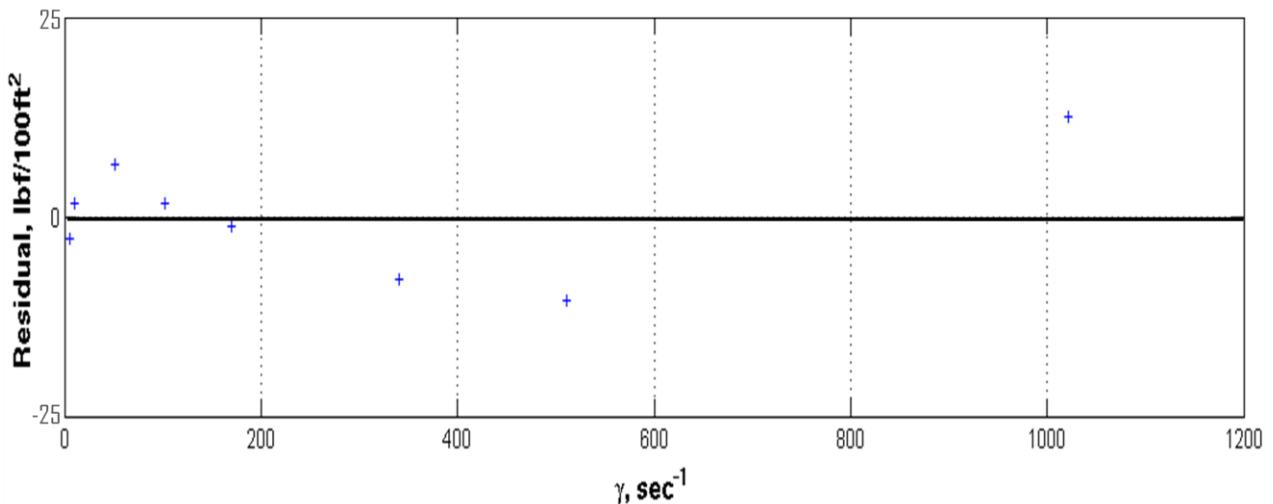


Figure 5.8b: Residual plot of fitted Modified Prandtl-Eyring values

Sisko (see figure 5.9a) and modified Sisko (see figure 5.9b) models rheogram describe the rheological behaviour of fluid in the same manner. They fit the data almost in all range of shear rates plotted. If a careful look is made at the residual plots of Sisko and modified Sisko in figure 5.10a and figure 5.10b respectively, you'll see that the data points are randomly distributed above and below the curve. These models look likely to give the best-fit but you cannot make a conclusive judgment to guarantee the goodness-of-fit only by analyzing the residuals. Residual mean squares (RMS) are further computed to compare models since it gives the total variance of the residuals. This RMS is very important to compare models especially when most models are non-linear and sample size is small (only eight).

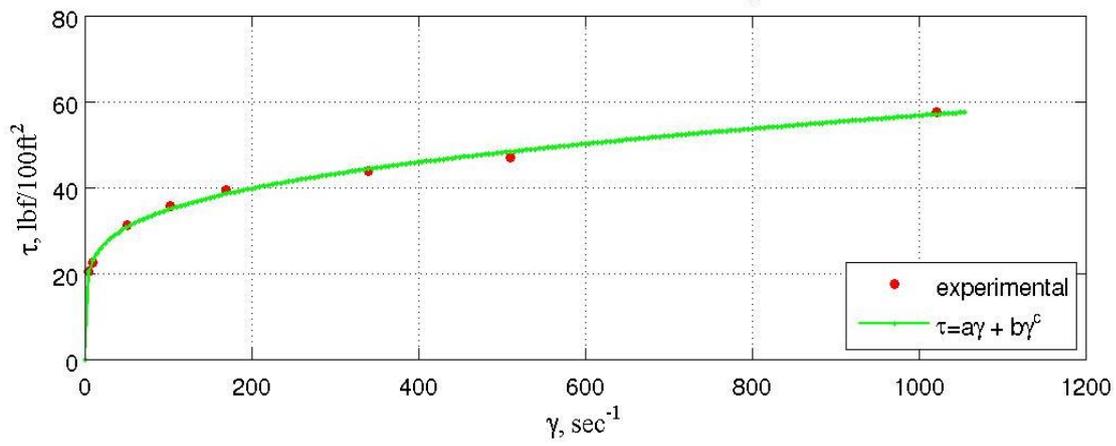


Figure 5.9a: Experimented rheological data and fitted Sisko values

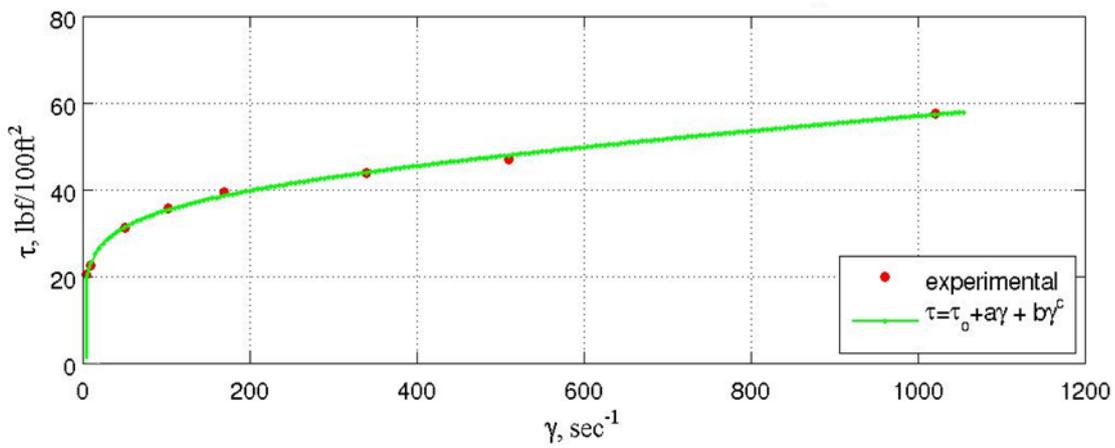


Figure 5.9b: Experimented rheological data and fitted Modified Sisko values

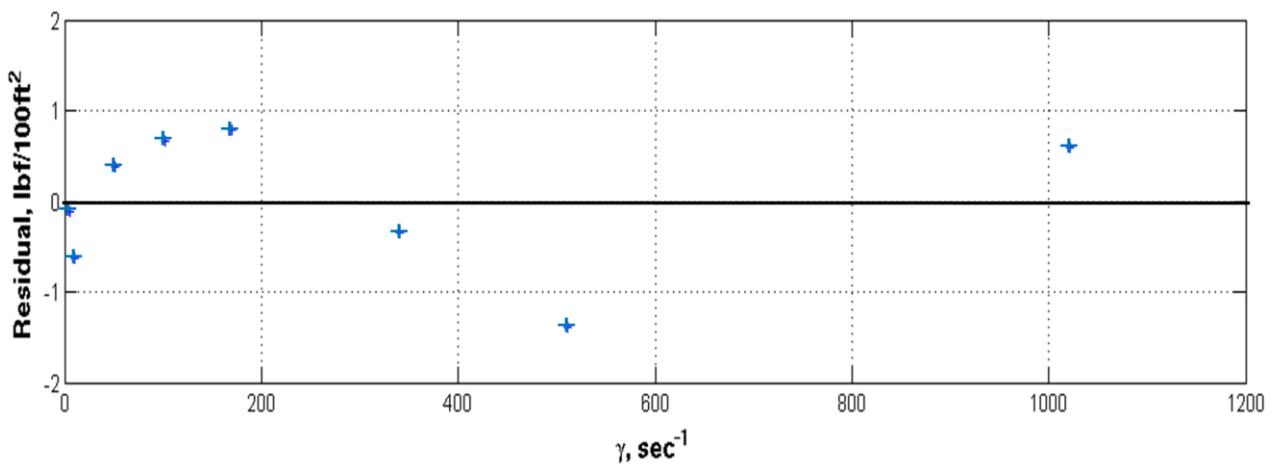


Figure 5.10a: Residual plot of fitted Sisko values

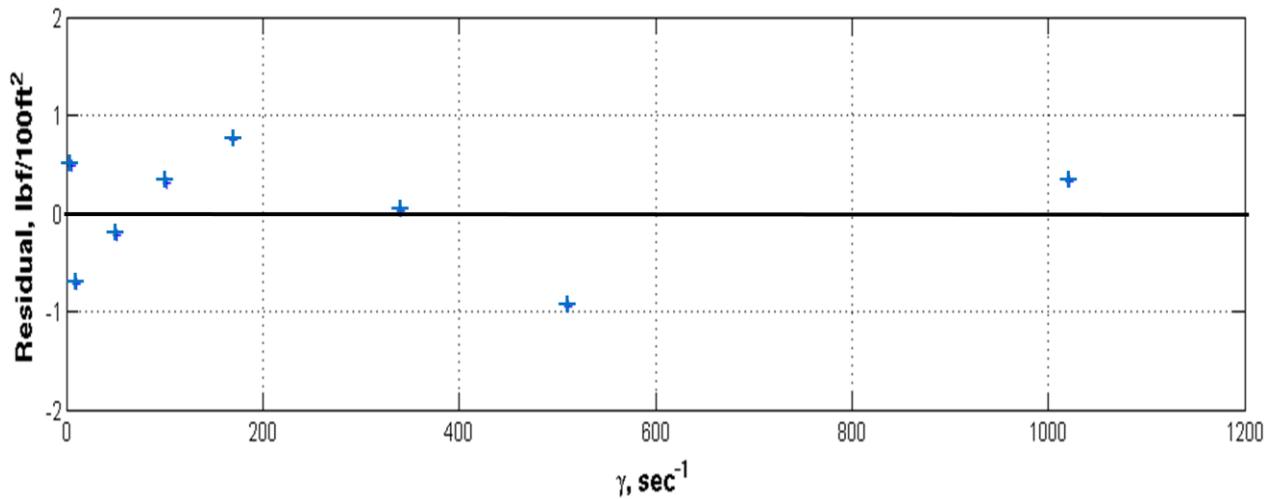


Figure 5.10b: Residual plot of fitted Modified Sisko values

5.3 SUM-OF-SQUARES AND MEAN SQUARES

Comparison of models to assess the goodness-of-fit by analyzing the residuals of functions is not sufficient especially when data points is small (<50). A Residual plot only cannot completely justify the adoption of a particular model. Total variability of model functions from the observed data is needed to make any plausible conclusions from the goodness-of-fit.

Based on the least-square regression analysis on the data, the parameters of the fitted models were calculated by minimizing the sum-of-squares of the residuals in order to produce a good fit. Figures 5.1a, 5.2a, 5.3a, 5.3b, 5.5a, 5.5b, 5.7a, 5.7b, 5.9a and 5.9b above show the comparison of the various fitted rheological models base on least-square regression analysis to the observed raw rheological data. A summary of result from the least square regression approximation using Matlab including the RMS values is shown in Table 5.1. It can be observed from Table 5.1 that Prantl-Eyring Model has the highest RSS and RMS values which are 2041.48 $\text{lbf}^2/100\text{ft}^4$ and 340.25 $\text{lbf}^2/100\text{ft}^4$, respectively. Modified Sisko model has the lowest RSS and RMS values of 2.47 $\text{lbf}^2/100\text{ft}^4$ and 0.61 $\text{lbf}^2/100\text{ft}^4$, respectively. In this study RMS is used as the main criterion to measure the performance of fit to select the model which is able to describe the rheogram of the biopolymer drill-in over all realistic range of shear it is exposed to. This is because of the small data sets of eight that can be produced by the viscometer readings. This criterion takes into consideration the varying number of parameters (degree of freedom) between models and produces an estimate of error variance.

Model Number	Model Name	q	RSS, Ibf²/100ft⁴	RMS, Ibf²/100ft⁴	Estimated Model Parameters
1	Bingham Plastic	2	192.576	32.096	$\mu= 0.032, \tau_o= 28.08$
2	Power Law	2	7.747	1.291	$K=14.19, n= 0.20$
3	Herschel-Bulkley	3	5.938	1.188	$\tau_o=7.47, K=8.56, n= 0.25$
4	Robertson-Stiff	3	6.732	1.346	$A=13.38, \check{Y}_o=2.38, B=0.21$
5	Modified Robertson-Stiff	4	9.362	2.341	$T_o 13.04, K=4.79, \check{Y}_o=0.60,$ $B=0.32$
6	Prandtl Eyring	2	2041.477	340.246	$A=30.67, B=205.97$
7	Modified Prandtl Eyring	3	15.556	3.111	$\tau_o=19.55, A= 8.73, B=34.89$
8	Sisko	3	3.982	0.796	$a= 0.006, b=15.33, c= 0.17$
9	Modified Sisko	4	2.468	0.617	$\tau_o=0.489, a= 0.012, b=501.35,$ $c= 0.009$
10	Casson	2	63.612	10.602	$\mu= 0.009, \tau_o= 22.71$

Table 5.1 shows the summary result from least square regression approximation. In ranking the RMS results in Table 5.1 it can be seen that some models perform (fit) better than others because of their low RMS values relative to other models. Some of these models are Sisko, modified Sisko and Herschel-Bulkley. This is because of the flexibility of these models to adapt to the rheogram the biopolymer drill-in fluid will exhibit. Prandtl-Eyring mathematical model should not be used since it gave the poorest fit and hence will result to wrong hydraulics predictions.

Most of the conventional industrially accepted models particularly Bingham Plastic model are not the best to model the pseudoplastic behaviour of the data as compared to some of the models base on their respective RMS values. Modified Sisko gave the best-fit because it gave the least RMS value followed by the Sisko model itself.

5.4 CONFIDENCE INTERVAL

Modified Sisko model is selected as a suitable model to describe the behaviour of biopolymer drill-in fluid because of the minimum RMS value. Once the suitability of the model is checked, it is possible to infer and create prediction intervals more reliably and hence to estimate shear stress with greater confidence. Within the range of experimental points, the prediction interval $100(1 - \alpha)\%$ for a particular shear stress is estimated by the Gallant(1986) formula in equations 3.7 through to 3.10. The 2-tailed t-value being taken at the required probability level, 0.05 and 4 degrees of freedom is 2.78. The confidence interval for the fitted modified Sisko is narrow enough as shown in figure 5.11. This means we obtain the smallest uncertainty near the centroid of the Modified Sisko function plot and can be 95% sure that the true best-fit curve (which could only be known if you had an infinite number of data points) lies within the confidence band.

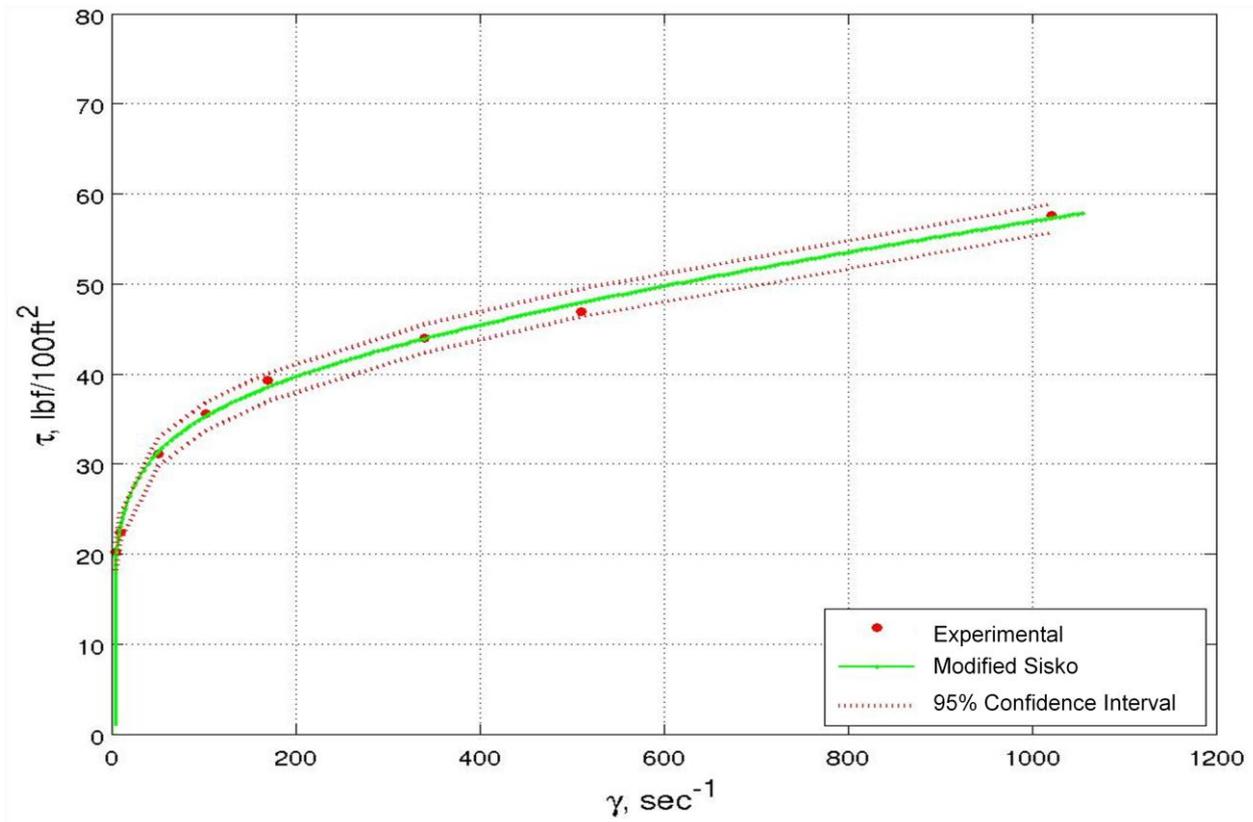


Figure 5.11: Experimented rheological data, fitted Modified Sisko values and its confidence interval

CHAPTER SIX

CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

Statistical evaluation of the biopolymer drill-in rheological data using least-square regression statistical method has revealed that there are suitable rheological models to approximate the behaviour of this fluid other than the conventional industry Power law and Bingham plastic models.

Conclusions drawn at end of this study are as follows:

1. The most likely rheological model to characterize the behaviour of xanthan based drill-in fluid is the Modified Sisko model. This model gave the minimum error variance (residual mean square) and there is 95% certainty that the true best-fit curve lies within the confidence band.
2. Prandtl-Eyring mathematical model gave the poorest fit and should not be applied since it will result in wrong hydraulic predictions as far as this drill-in fluid is concerned.

6.2 RECOMMENDATIONS

1. The rheological properties of this xanthan base biopolymer drill-in were measured at a nominal temperature of 120°F. Using the parameter obtained from this rheological model at this temperature conditions might result in inaccurate hydraulic calculation especially when drilling offshore because drilling fluids experience high temperatures downhole and very cold temperatures in risers, while both locations are associated with high pressures. It is recommended that future work should be done on temperature and pressure effects on the rheological behaviour of this xanthan based biopolymer drill-in in order to select the appropriate model.
2. Future studies should be carried out to make hydraulic calculations like frictional pressure loss predictions and ECD estimation base on the selected rheological model (Modified Sisko model).

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NOMENCLATURE

a = parameter in Sisko or Modified Sisko fluid model

A = parameter in Roberston-Stiff, Prandtl-Eyring or Modified Prandtl-Eyring fluid model

AAPE = absolute percentage error

b = parameter in Sisko or Modified Sisko fluid model

BP= Biopolymers

B= parameter in Roberston-Stiff, Prandtl-Eyring or Modified Prandtl-Eyring fluid model

c= parameter in Sisko or Modified Sisko fluid model

CMC=carboxymethylcellulose

CPC= critical polymer concentration

df= degree of freedom

ECD= equivalent circulating density

$f(\gamma, \beta)$ = A general function of the rheological models

F = N x q matrix of partial derivative of $h(\beta)$

h=length of viscometer bob in cm

$h(\beta)$ =nonlinear function of interest

H = the row vector of partial derivatives of $h(\beta)$

HEC = hydroxyethylcellulose

HTPT=High Temperature High Pressure

Matlab = Matrix Laboratory Software

MPD=Managed Drilling Pressure

MWW = mud weight window

N= Number of data points

OLS=ordinary least-squares

PAA=polyacrylamide

q=Number of parameters in a model

r_1 = Fann viscometer bob radius in cm

r_2 = Fann viscometer rotor radius in cm

R^2 = coefficient of determination

RMS= Residual Mean Square in $\text{Ibf}^2/100\text{ft}^4$

RSS= Residual Sum of Square in $\text{Ibf}^2/100\text{ft}^4$

s^2 =estimated error variance in $\text{Ibf}^2/100\text{ft}^4$

S = speed of rotation of outer cylinder of the viscometer in rpm

t = t-distribution value

α = significance level

β is the value of model parameter

ϵ = random error in lbf/100ft²

τ = shear stress in lbf/100ft²

τ_0 = yield stress in lbf/100ft²

θ = rotational viscometer dial readings in degrees

μ = apparent viscosity

μ_p = Plastic viscosity, cp

γ = shear rate in sec⁻¹

γ_0 = parameter in Roberston-Stiff fluid model

APPENDIX A

Table A1: Results of Rheological Data from Viscometer Readings

Speed, rpm	Readings, °	$\dot{\gamma}$, sec ⁻¹	τ , lbf/100ft ²
600	54.0	1021.8	57.62
300	44.0	510.9	46.95
200	41.2	340.6	43.94
100	36.8	170.3	39.29
60	33.3	102.2	35.58
30	29.1	51.1	31.10
6	21.0	10.2	22.41
3	19.0	5.1	20.27

Table A2 Parameter constraints and Initial Guess to Evaluate the Rheological Model Functions			
Model Name	Model Equation	Parameter Constraints	Initial Guess
Bingham Plastic	$\tau = \tau_o + \mu_p \dot{\gamma}$	$\mu_p > 0, \tau_o \geq 0$	$\mu_p = 0.02, \tau_o = 1$
Power Law	$\tau = K \dot{\gamma}^n$	$K > 0, 0 < n < 1$	$K = 2, n = 0.4$
Herschel-Bulkley	$\tau = \tau_o + K \dot{\gamma}^n$	$\tau_o \geq 0, K > 0, 0 < n < 1$	$\tau_o = 1, K = 2, n = 0.4$
Robertson-Stiff	$\tau = A(\dot{\gamma}_o + \dot{\gamma})^B$	$A > 0, 0 < B < 1, \dot{\gamma}_o \geq 0$	$A = 2, B = 0.4, \dot{\gamma}_o = 1$
Modified Robertson-Stiff	$\tau = \tau_o + A(\dot{\gamma}_o + \dot{\gamma})^B$	$\tau_o \geq 0, A > 0, 0 < B < 1, \dot{\gamma}_o \geq 0$	$\tau_o = 0, A = 2, B = 0.4, \dot{\gamma}_o = 1$
Prandtl-Eyring	$\tau = A \sinh^{-1}(\dot{\gamma}/B)$	$A > 0, B > 0$	$A = 16, B = 30$
Modified Prandtl-Eyring	$\tau = \tau_o + A \sinh^{-1}(\dot{\gamma}/B)$	$A > 0, \tau_o \geq 0, B > 0$	$\tau_o = 0, A = 10, B = 50$
Sisko	$\tau = a \dot{\gamma} + b \dot{\gamma}^c$	$a \geq 0, b \geq 0, 0 < c < 1$	$a = 0, b = 2, c = 0.4$
Modified Sisko	$\tau = \tau_o + a \dot{\gamma} + b \dot{\gamma}^c$	$a \geq 0, b \geq 0, 0 < c < 1, \tau_o \geq 0$	$\tau_o = 0, a = 0, b = 2, c = 0.4$
Casson	$\tau = (\sqrt{\tau_o} + \sqrt{\mu \dot{\gamma}})^2$	$\mu > 0, \tau_o \geq 0$	$\mu = 1, \tau_o = 1$

Table A3: Experimented, Expected Modified Sisko and 95 % Confidence Interval Shear Stress Values

Experimental Value	Expected Modified Sisko Value	95 % Confidence Interval
$\tau, \text{Ibf}/100\text{ft}^2$	$\tau, \text{Ibf}/100\text{ft}^2$	$\tau, \text{Ibf}/100\text{ft}^2$
20.27	19.79	21.50 to 18.08
22.41	23.13	24.84 to 21.42
31.10	31.317	33.03 to 29.60
35.58	35.27	36.98 to 33.56
39.29	38.54	40.25 to 36.83
43.94	43.92	45.63 to 42.21
46.95	47.9	49.61 to 46.19
57.62	57.29	59.00 to 55.58