EVALUATION OF SOME PLANT EXTRACTS AS SURFACE-ACTIVE AGENTS IN DRILLING MUD FORMULATIONS

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A Thesis submitted to the School of Graduate Studies, University of Port Harcourt in partial fulfillment of the requirements for the award of the degree of Doctor of Philosophy (Ph.D) in Analytical Chemistry in the Department of Pure and Industrial Chemistry, Faculty of Science, University of Port Harcourt.

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MARCH, 2020

DECLARATION

I **BIWOTT, TECLA CHEPTOO** with Registration number **G2017/PhD/ACE-CEFOR/FT/072** declare that the work in this Thesis on the "**EVALUATION OF SOME PLANT EXTRACTS AS SURFACE-ACTIVE AGENTS IN DRILLING MUD FORMULATIONS"** was carried out by me; that it is my original work and that it has not been submitted wholly or in part for the award of a degree in this or any other institution.

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CERTIFICATION

UNIVERSITY OF PORT HARCOURT

SCHOOL OF GRADUATE STUDIES

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The Board of Examiners certifies that this Thesis is accepted in partial fulfillment of the requirements for the award of the degree of Doctor of Philosophy (Ph. D) in Analytical Chemistry.

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DEDICATION

This work is dedicated to my family; George, Alvin, Ivy and Yvonne

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ABSTRACT

This research was aimed at: evaluating physicochemical properties of *Carica papaya* -PL, *Vernonia amygdalina* - BL, *Terminalia mantaly* - TL and *Moringa oleifera* – ML; establishing rheological properties of the drilling mud formulated with the plant leaves; evaluating thermal stability between 30 - 93˚C and fluid loss control ability. From standard experimental procedures, BL had the highest length of foam (56mL) and the longest vanishing time (89.33minutes). ML had the least foam length (10mL) and the least time (28.33minutes). Qualitatively, all plants had saponins as FTIR confirmed their presence through characteristic absorptions of OH, C = O, C-H, C = C and C-O-C for glycosidic linkages. Plastic viscosity (PV) of control mud with commercial surfactant (C2) and mud with TL leaves (MTL) were stable at high temperatures of 70˚C by recording 38cP and 37cP respectively. MPL gave the least PV (31cP) at 30˚C. Yield point (YP) of the mud showed that the mud with PL leaves (MPL) was stable at 70˚C by recording 24 $1b/100ft^2$. Mud with BL leaves (MBL) had good gel strengths at 10 seconds and 10 minutes by recording 5 and $31.11b/100ft^2$ respectively. Fluid loss decreased by 72% for mud with ML leaves (MML) and MTL mud. Test mud was more stable thermally compared to control sample: MBL PV was 31cP at 93°C, MPL recorded YP of (16 $1b/100ft^2$) at 93°C, gel strengths of mud with plant leaves showed flat gel (4 -7.2 $1b/100ft^2$) at all aging temperatures while control mud had progressive gel strength (26-56 $1b/100ft^2$) with increase in aging temperatures. MML and MTL had good filtration control properties by losing 5mL of the filtrate. The selected plants improved mud properties which could be attributed to the presence of surface-active compound(s) in the extracts, hence it can be used as mud additive once toxicity test and overall cost benefits are done.

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LIST OF ABBREVIATIONS

ADM	Air drilling mud
API	American Petroleum Institute
BL	*Veronia amygdalina* leaves
EDM	Emulsion drilling mud
FT-IR	Fourier transform infrared
HV	High Viscosity
IEDM	Invert emulsion drilling mud
LV	Low viscosity
MBL	Drilling mud with *Veronia amygdalina* leaves
ML	*Moringa oleifera* leaves
MML	Drilling mud with *Moringa oleifera* leaves
MPL	Drilling mud with *Carica papaya* leaves
MTL	Drilling mud with *Terminalia mantaly* leaves
OBM	Oil-based mud
PL	*Carica papaya* leaves
Ppg	pounds per gallon
PV	Plastic viscosity
RPM	Rotation per minute
SBM	Synthetic based mud
SDM	Synthesized drilling mud
TL	*Terminalia mantaly* leaves
WBM	Water-based mud
YP	Yield point

CHAPTER ONE

INTRODUCTION

1.1 Background to the study

An increase in demand for crude oil as an energy source has been steered by the rise in population and industrialization which is steady. Consequently, the urge to increase oil production through well-drilling enhanced oil recovery has gone up (Abduo *et al.*, 2016). Well-drilling operations are faced with many challenges such as damaging effects on rheological properties of the drilling mud in extreme conditions like high temperatures and high pressures (HTHP). These challenges have occasioned continuous demands in fluids development with specific physical, chemical and rheological properties (He *et al.*, 2016). For the desired properties of drilling muds to be achieved, all the additives to be introduced must give the drilling mud outstanding properties (rheology, gel strength, viscosity, and filtration properties) (Dardir *et al.*, 2017). In oil production industry, drilling mud is among the main fluid used in well-drilling which helps to suspend and release well cuttings, add power to the bit, seal permeable formations, maintain wellbore stability, reduce damage in the formation, cool and lubricate the drilling bit and bottom hole assembly (Fereydouni *et al.*, 2012)

Among additives of the drilling mud are surfactants that play very important roles such as; corrosion preventers, deformers, dispersants, and wetting agents. Surfactants are also used as shale swelling inhibitors to prevent wellbore instabilities and also prevent cuttings from sticking to drill bit (Galindo *et al.*, 2015). Surfactants are utilized in the production of high gas/water ratio (HGWR) foam in drilling fluids to generate low-pressure reservoirs and hard rock drilling. Also, surfactants used to eliminate undesirable foam in water-based fluids (Ghosh and Mohanty, 2019). Most of the surface-active agents used in drilling mud formulation are synthesized, giving rise to high cost in overall oil production. Besides, some synthesized surface-active agents are highly toxic and environmentally unfriendly. Naturally occurring surfactants are less toxic and eco- friendly hence find use in petroleum, agricultural, medical and cosmetics industry since they are biodegradable, have better physiological properties and are relatively readily available (Khan and Butt, 2016).

Synthesized surfactants and other drilling mud additives can lead to environmental risk in various ways e.g. contamination of soil, marine environment and effects on human life (Negm *et al.*, 2015). To reduce environmental challenges associated with drilling mud additives used for oil production, it is paramount to balance its performance while retaining its properties (Hossain and Islam, 2018). The environmental concern and high cost of synthesized or conventional mud additives have led to the search for safer and cheaper additives from natural products e.g. plants. This is because plants possess the ability to produce an almost limitless array of substances that are environmentally friendly, economically feasible and easy to use (Mohammadinejad *et al.*, 2019).

The drilling process can be upgraded by the use of drilling mud with appropriate additives. Three major types of drilling muds are; water-based drilling mud (WBM), oil-based drilling mud (OBM), and synthetic-based drilling mud (SBM) (Epikhin *et al.*, 2015). Drilling mud type is important because it is chosen depending on the behavior of the formation which is to be drilled (Al-Yasiri and Al-Sallami, 2015). Water-based drilling mud has advantages of higher shear thinning, high yield strength, have better bit hydraulics, reduces pressure losses in circulation compared to other mud systems and it increases borehole steadiness too (Elkatatny, 2019).

In WBM, the major component is water and other ingredients are considered as additives and they have various functions like viscosifiers, fluid loss controllers, corrosion inhibitors and weighting agents (Samavati *et al.*, 2014).

Plant derivatives have gained much interest since they have versatile uses such as bio-resources for the invention of drugs (Farhat, 2018). Studies have been carried out on drilling mud formulations using various mud additives. It is necessary to carry out studies on the effects of plant constituents on the properties of WBM (Ahmad *et al.*, 2018) which not much have been done on them.

Viability of some plant extracts needs to be tested for use as drilling mud additives to produce drilling mud additives that have the capacity for improving performance, sustainable and affordable.

1.2 Statement of the problem

Drilling mud with enhanced mud properties are essential to optimize the performance of oil well drilling. For ideal drilling mud to be formulated, mud additives have to be chosen considerably. Among the factors considered in proper drilling mud, the selection is drilling performance, expected well conditions, mud cutting disposals and personnel safety. Some of the properties to be improved in drilling fluid are rheological properties, pH, mud weight, fluid loss, and thermal stability. Mostly, surface-active agents used are synthesized and pose a negative impact on the environment. These negative impact leads to the death of flora and fauna and thus poses a threat to biodiversity and sustainable development. Plants are acknowledged to have many metabolites that can be utilized in chemical industries.

However, the drilling mud has to be improved because it is facing the following challenges:

i) Conventional mud additives in drilling mud can be toxic and expensive hence there is need to search for locally available materials which would be affordable and non-toxic and help actualize local content.

ii) When drilling into deeper formation, drilling mud has to endure elevated pressures and temperatures. Temperature and pressure influence on the properties of drilling mud is uncertain and leads to difficult challenges and mechanical concerns.

iii) Surface-active agents have found a major part in the industry of petroleum as additives in drilling mud where they play a part as corrosive preventers, deformers, and dispersants, wetting agents and also they are used to improve the properties of the drilling mud. It is good to test the capability of surface – active agents to improve the rheology of the mud.

1.3. Aim and objectives

The study aimed at evaluating some plant extracts as surface-active agents in drilling mud formulations that are safer, eco-friendly and sustainable.

The specific objectives of this study were to:

1. Determine the physicochemical properties of powdered leaves of the selected plants (*Carica papaya*, *Vernonia amygdalina*, *Terminalia mantaly,* and *Moringa oleifera*),
2. Establish the rheological properties of the drilling mud formulated with the selected plants,
3. Evaluate the thermal stability of the formulated drilling mud at different temperatures (30˚C- 93˚C)
4. Investigate the fluid loss of the formulated drilling mud.

1.4 Significance of the study

1. This research has the capability of steering the production of *Carica papaya*, *Vernonia amygdalina*, *Terminalia mantaly*, and *Moringa oleifera*, hence making it accessible for use in the petroleum industry and will result to economic base for local farmers which will result to economic sustainability.
2. The use of locally available materials will help in actualizing the local content policy in the oil and gas business regarding local raw materials utilization leading to sustainable growth in the oil and gas production.
3. With the minimal treatment of cuttings from drilling process, and reduced cost of importation of surface-active agent materials, leading to overall reduction of cost in production of oil and gas.

1.5 Scope of the study

The study applied leaves from four different plants, namely: *Carica papaya* (pawpaw), *Vernonia amygdalina* (bitter leaf), *Terminalia mantly (*Madagascar almond) and *Moringa oleifera* (Moringa). The physicochemical and properties of the formulated drilling mud were investigated. Crude extracts from the selected plants were characterized using FT-IR.

CHAPTER TWO

LITEREATURE REVIEW

2.1 Background

Africa has been undergoing trade and industrial growth since 2000 and this has resulted to the rise in energy consumption by 45%. Main aim of African countries is to manage the growing energy demand of its population and ensuring that there is collective way in to recent energy services in respect to the environment (Nadia, 2017). Energy accesses are fundamental to development and as trade and industry advance, incomes and growth in populations require more energy. Sustainable Development aims at obtaining accessibility to affordable, reliable, sustainable and modern energy hence require an increase to electricity accessibility and the take up of clean fuels (Shonali *et al.,* 2013).

Lasting forecast of energy supply and demand is most important in Africa due to the stable increase in energy requirements (Spalding-Fecher *et al.*, 2017). There is decline in availability of adequate resources, soaring dependence on fossil- fuels to meet the necessities, and the universal concerns over energy induced environmental issues (Jefferson, 2016).

Natural gas and oil are the leading source of the world's energy. Its consumption is more due to maintenance of present high living standard and most of this energy comes due to the technology of oil production. Current industrial activities consume huge amounts of energy to catch up with high demand and among societies' concerns is the accessibility of abundant and relatively cheap source of oil (Al-Maamary *et al.*, 2017).

The extent of field expansion cannot be overemphasized, mostly in the perspective of full-grown petroleum provinces due to decline in domestic production and increase in importation (Hoffmann *et al.*, 2015). Field growth is increasing in existing fields through time because of many factors including enhanced oil recovery, discovery of new pools or extensions of known pools. Also, there is improvement of reservoir characterization and development of more sophisticated and efficient reservoir simulators and data processing capabilities (Anifowose *et al.*, 2016). Global average recovery factor for oil field is within 40% hence large amount of oil identified which was left despite an existing production infrastructure (Jafarnezhad *et al.*, 2017).

2.2 Drilling mud

Drilling mud usage is part of drilling procedure and there are varieties of chemicals used in designing a drilling mud system so as to meet its purpose like improved mud rheology, good density, and fluid loss control property (Yunita *et al.*, 2015). In the process of searching these mud properties, preservation of environment is a global concern and many organizations are advised to use nontoxic drilling mud additives. Environment pollution is a serious hazard while drilling wells that are complex with high temperatures. These complex wells are managed incorporating high performance water based mud and oil based mud which use many chemicals to produce the desired mud properties (Haddad *et al.*, 2017).

Drilling mud is mainly designed to reduce damage effect of reservoir rock and ensure that drilling into the formation is successful and economical (Majid *et al.*, 2019). Drilling muds are also made to generate a filter cake that decreases the filtration loss into the formation, and less thick in order to hold the drilling mud within the wellbore (Agwu and Akpabio, 2018). In drilling process, mud is passed through the drilling bit, taken back to the surface by passing through the annular passage in amid the drill stem and well's wall (Perry *et al.*, 2019). To control viscosity, gel strength, filtration loss and yield point, mud additives are used (Zha and Zhou, 2019). Mostly, drilling mud should comply with key requirements like, easy to use, inexpensive and environmentally friendly (Hossain and Apaleke, 2015). Additives in drilling fluid can undergo degradation that can lead to fluid loss resulting to problem in real-time drilling (Pakdaman *et al.*, 2019). Biocides used in drilling mud can threaten the environment and could cause harm if not handled properly (Pakdaman *et al.*, 2019). Some studies found out that cationic surfactant (CTAB) used with sorbitan monooleate can be used to control fluid loss for preventing the fluid movement into the porous pristine formation and it can avoid the collapse of well wall in oil-drilling excavation (Shettigar *et al.*, 2018).

Surfactants could decrease the friction factor without help of the wetting materials used and formulation has higher performance in coefficient of friction than diesel –based formulation without affecting the rheology of the mud (Song *et al.*, 2016). For the drilling mud to be effective, there must be three conditions present; a permeable medium, liquid and liquid/solid slurry fluid (Talukdar *et al.*, 2018). Drilling mud performance is mainly affected by drilling fluid density, drilling fluid viscosity, drilling mud pH and gel strength (Avci and Mert, 2019).

2.3 Types of drilling muds

Drilling mud is selected for specific role and it must have favorable properties for the intended purpose. A drilling mud task is guided by its rheological properties and is carefully chosen according to the type of formation to be drilled, range of temperatures and strength, permeability and pore fluids pressure exhibited by the formation (Song *et al.*, 2016). In addition, drilling mud can be chosen according to safety and logistics, productions concerns, environmental impact and overall well cost. The drilling muds are chosen depending on the drilling conditions faced in the field and are categorized based on the continuous phase they are using (Wagle *et al.*, 2016).

2.3.1. Oil based mud (OBM)

Oil based mud uses oil as its continuous phase and mostly used when WBM is considered insufficient. Generally they are composed as reverse – emulsion of brine in a continuous oil phase which is stabilized by surfactants (Ma *et al.*, 2018). However, OBM are among the least used drilling mud because of its costly nature, though they do give better results when used (Shahmirzaee *et al.*, 2019).

2.3.2 Synthesized drilling mud (SDM)

Synthesized drilling mud is similar as oil based drilling mud but less destructive to the environment. It is composed of esters, olefins and occasionally paraffin. It has been identified to have low kinetic viscosity and could work under low pressures (Ma *et al.*, 2019).

2.3.3 Emulsion drilling mud (EDM)

Emulsion drilling mud has emulsion based phases. It has two phases, the outer and inner phases. Outer phase has water while the inner phase is made of oil (Tan *et al.*, 2015). Surfactants are mostly used to enable the two phases to be miscible because of the opposite emulsion effect. They are cheap and eco-friendly when compared with OBM, but costly when compared with WBM (Olaitan *et al.*, 2017).

2.3.4 Invert emulsion drilling mud (IEDM)

It resembles EDM in all aspects but dissimilar in how phase is being distributed. It is an inverse form of EDM and it has two phases but the outer one has oil while the inner one has salt water. It can be degraded faster by micro-organism, has light viscosity and it suits high temperature environment (Ihenacho, 2016).

2.3.4 Air drilling mud (ADM)

Air drilling mud is mainly aerosols and is utilized mostly for underbalanced pressure drilling where there is no water or oil in the reservoir. It is advantageous in that it has little formation damage, zero circulation loss, less contaminated and high penetration rate (Zhu *et al.*, 2015).

2.3.5 Water-based mud (WBM)

Water-based mud uses water as its continuous phase. The composition of water-based mud comprises mostly of clay, brine, and polymers (Negm *et al.*, 2015). It is one of the prime drilling fluid used since it is advantageous because it has high shear thinning, good bit hydraulic, high true yield and reduced circulating pressure losses. Also, known to improve borehole stability (Galindo *et al.*, 2015). This research used water-based mud formulations in the study.

2.4 Properties of drilling mud

It is significant to monitor and maintain the rheological properties and other parameters of drilling mud because failure to monitor can lead to financial and time loss and occasionally it can result in abandonment of the well (Rakshith *et al.*, 2017).

2.4.1 Density

The drilling mud density is defined as mass per unit volume of drilling mud and it is presented as parts per gallon (ppg) (Fattah and Lashin, 2016). The density of the drilling mud must match the density necessary to check formation pressure without fracturing or damaging the formation (Ahmadi *et al.*, 2018). Encountered problems in rotary drilling are associated with drilling mud weight. Mud weight is among the foremost parameter to consider during drilling mud formulation (Ahmed *et al.*, 2018). Drilling mud's densities decrease with rising temperature. The capability of the drilling mud to suspend the drill cuttings determines the impact of hole cleaning (Ebikapaye, 2018), thus needs attention. Mud weight (density), controls the pressure in the formation and help wellbore stability. Less mud density will result in underbalanced conditions, so any formation of fluids – gas, water, and oil will enter into the wellbore. Mud weight provides pressure to the hole back-formation, hence less mud weight may result to collapse of the wellbore (Feng *et al.*, 2016). Drilling mud density should match the estimated density necessary to check formation pressure without fracturing or damaging the formation (Lambert *et al.*, 2018).

High mud density can lead to lost circulation (Hosseini, 2017) caused by hydrostatic pressure from the mud column which exceeds the formation strength hence causing the formation to break (Feng *et al.*, 2016). Once the formation is cracked, drilling fluids will be lost into the induced formation fractures. Heavier drilling mud densities will lead to a slower penetration rate (ROP) and mitigate well control (Al-Sabagh *et al.*, 2016). High mud weight can cause differentially stuck pipe since there are changes between formation pressure and hydrostatic pressure, the chances that a drill string will get differentially stuck across the permeable rocks will be high (Ebikapaye, 2018).

Formation damage can occur if more mud weight is present in the well because more mud filtration will invade into porous formations (Feng and Gray, 2017). The invaded mud will cause damage to formation of rocks. Drilling in exhausted reservoirs are challenged by many issues, like formation damage due to increase of pressure, differential sticking in the wellbore and different pressure zone among others. The mud weight is of essence in such zones (Olise *et al.*, 2017).

2.4.2 Velocity

The pump rate must be kept low to maintain laminar flow, but not slow as to jeopardize efficient cutting removals; Critical velocity can be obtained from mud density, viscosity, speed and size of the annulus (Roustaei *et al.*, 2015).

2.4.3 Viscosity

Viscosity of drilling mud is its resistance to flow. In drilling fluid, the desired viscosity is affected by many factors including mud density, hole size, drilling rate, pumping rate and hold problems (Al-Mahdawi and Saad, 2018). Viscosity measured by an instrument is valid for the sheer rate at that specific time and shall not be the same when measured as a different rate of sheer (Azar, 2015). Viscosity is measured by many methods that include; marsh funnel, Fann V-G meter and rotational multi-speed instrument as a standard for plastic viscosity, gel strength and yield point. Increase in fluid viscosity increases amount of cuttings transport capability (Werner *et al.*, 2017).

2. 4.4 Plastic viscosity

Drilling fluid resistance to flow is termed as plastic viscosity which indicates the extent of physical and chemical relations between solids and fluid particles that are applied into the drilling mud (Ghahfarokhi *et al.*, 2015). A low plastic viscosity implies that the mud has the ability of drilling rapidly since the low viscosity leaving the bit (Bloys *et al.*, 1994). High plastic viscosity is caused by a viscous fluid and by excess colloid solids (Prince *et al.*, 2019). Any increment of solid content in drilling mud like barite and fluid loss materials results in higher plastic viscosity (Zhuang *et al.*, 2015).The occurrence of shale particles in drilling mud system when drilling shale zone can increase plastic viscosity too. High plastic viscosity generates higher resistance in mud hence will affect cutting lifting performance (CLP) (Yunita *et al.*, 2015).

2.4.5 Apparent viscosity

Viscosity of a fluid that is displayed in an instrument at a particular shear rate and is a function of Bingham properties of yield point plastic and viscosity is called apparent viscosity (Elkatatny *et al.*, 2018). It also shows the association between shear stress and shear rate (Christensen, 2017).

2.4.6 Shear rate

Shear rate is the gradient velocity measured across the diameter of a fluid –flow channel, annulus or shape (Patel *et al.*, 2017). It imitates the rate of alteration of velocity at which one layer of fluid passes over an adjacent layer. The velocity gradient is the degree of change of velocity with distance from the plates (Fakoya and Ahmed, 2018).

2. 4. 7 Filtration loss

Filtration of drilling fluid is its liquid phase being forced into a permeable formation by differential pressure (Patel *et al.*, 2017). It is the amount of filtrate (free water) passing from the drilling mud into a porous permeable formation. In this process, solid particles are filtered out and in the process, form a filter cake (Ezeakacha *et al.*, 2017).

2.4.8 Mud cake

A layer of particles formed by drilling mud as it exerts pressure on the wall of the wellbore is called mud cake. It provides a physical barrier that prevents further loss of the drilling mud (Ezeakacha *et al.*, 2017) . While drilling, the hydraulic head exerts some pressure into the fluid and some filtrate separates and form a filter cake on wellbore hence stabilizing the wellbore. Drilling mud should create a thin filter cake on the walls of the borehole. Low filtration loss is a characteristic of good drilling mud and the key bore hole integrity (Al-Ajmi *et al.*, 2018).

2. 4. 9 pH

pH of the drilling mud is the acidity or alkalinity of the mud (Inemugha *et al.*, 2019) . Drilling mud's pH should be maintained between 8 and 9 (Sulaimon *et al.*, 2017). When pH is lower (acidic), soda ash may be used to raise its pH level. Lower or high pH of drilling mud will lead to more use of additives hence making it costly (Alkamil *et al.*, 2018).

2.5 Water based drilling mud additives

Water based mud has a mixture of solids, chemicals and liquids where water is a continuous phase. The solids can be inactive or active in the mud. Solids that are hydrophilic like clay that is hydratable, can result to reaction with water phase and in the process dissolve the chemicals rendering the mud viscous (Negm *et al.*, 2015). The inert hydrophobic solids like sand or shale, are unreactive in water and other compounds and differ in specific gravity hence hinder the examination of solids in the drilling mud because they produce undesirable effects (Kania *et al.*, 2015). In WBM, many additives are used currently and they include; polymers, clays, weighting agents, fluid loss agent, lost-circulation materials, thinners, dispersants, inorganic chemicals, and surface-active agents (Gogoi and Tamuli, 2019).

Surface-active agents are used commonly in petroleum industry as additive in water based mud so as to alter colloidal form of the clay from spreading to one controlled flocculation (Negm *et al.*, 2015). Surface-active agents can be anionic or cationic and can act as fluid emulsifiers, dispersants, wetting agents, foamers and defoarmers. Surface-active agents are also used to decrease the hydration of the clay surface. Structural groups of the molecules affects the surfactant's behavior (Rieger, 2017). The additives that are mainly used in water based mud are: Barite, Bentonite, Hydro PAC, Caustic soda, potassium chloride and xantum gum.

2.5.1 Barite

This is a naturally occurring rock and it is used to increase density of water and oil based drilling mud (Ibrahim *et al.*, 2017). When mud density is increased, there is increase in rate of penetration (ROP), however, too high density leads to differential sticking of the drill string (Hilfiger *et al.*, 2017). Barite is insoluble and inert chemically and a dense mineral containing barium sulphate ($BaSO_4$) hence widely used as weighting agent. It has specific gravity between 4.20 - 4.40 g/cm^3 which is capable of meeting API recommends mud densities to be between 9 to 19 lbm/gal (Abdou *et al.*, 2018). Siderite, hematite, calcium carbonate, ilmetite and galena are also used in the industry as weighting agent (Martel *et al.*, 2018) depending on the type of the formation.

2. 5. 2 Bentonite

Bentonite clay is among the additives used as a fluid loss control and mud viscosifier in drilling mud (Needaa *et al.*, 2016). Bentonite has been used in petroleum industry since 1929 (Choudhary *et al.*, 2018). Bentonite is composed of smectite and contains less than 85% of clay mineral mentmorillonite. Bentonite is mostly classified as calcium bentonite or sodium bentonite (Oriji *et al.*, 2014), based on the main exchangeable cation present.

For fluid loss control, some clays, dispersants and polymers are extensively used. Bentonite (montmorillonite) is used as the primary fluid loss-control additive in water based fluids. It has a very thin and sheet like particles with large surface area that are able to produce a flexible filter cake. The drilling mud systems that are inhibitive obstruct the hydration of bentonite which can reduce its efficiency (Shadizadeh *et al.*, 2015) . It is important to prehydrate bentonite in clean water before mixing it with other additives (Oriji *et al.*, 2014).

2.5.3 Hydro Poly Anionic Cellulose LV /HV

Hydro PAC major use is as fluid loss control material and viscosifier. Thinners or dispersants serve the function of controlling fluid loss and decreasing filter cake thickness. Sometimes, dispersants act as fluid loss regulator by bridging small pores in the filter cake (Al Yami *et al.*, 2017). In drilling well operations, circulation may be lost through permeable sandstones, induced or natural formation fractures which are generally induced by excessive drilling –fluid pressures (Feng *et al.*, 2016) and in this way, there is absence of mud returning to the surface after it has been driven down a well (Scrivener *et al.*, 2018). To control the lost circulations, most chemicals used include fibrous materials like wood, cotton and mineral fiber (Liu *et al.*, 2018).

2.5.4 Caustic soda

Caustic soda in water based mud produces hydroxyl ion that control pH. Caustic soda is a strong base and its extreme solubility in water gives rise to Na^+ and OH^- in solution. Conversely, its corrosive nature makes it one of the hazardous chemical to the handlers (Gokul *et al.*, 2017).

2.5. 5 Potassium chloride

Potassium chloride in drilling mud provide potassium ions that stabilizes shales and control the swelling of clays (Aziz *et al.*, 2018).

2. 5.6 Xantum gum

Xantum gum is used predominantly in petroleum industries to accelerate the viscosity of the aqueous based drilling mud. Xantum gum is good in high temperatures, salinity makes xantum polymer the most used as a viscosifying material in drilling mud formulations(Talukdar *et al.*, 2018).

2.6 Use of plants to improve water based drilling mud properties

A lot of research efforts have been directed to formulating water based mud with mud additives aimed to handle problems associated with them. Substances originating from plants have handy application thus are very important. Since they are bio-based source, plants have found use in drug industry as well as variety of chemical development (Lee *et al.*, 2019).

Grass which is usually a fodder crop was used to formulate drilling mud (Hossain and Wajheeuddin, 2016). The mud formulated was of three different concentrations and varied particle sizes. Their investigation revealed that varied particle sizes and concentration of dried and grounded grass, when added to bentonite, improved rheological properties of the drilling fluid. They further gathered that the fluid was enhanced when grass was used because of the reduction of filtration loss. Their observations recommend using grass to control pH and filtration loss and also as rheological modifier to substitute toxic materials from drilling fluids. However the research did not test thermal stability of the water based mud formulated with grass.

In a study, it was found out that fluid loss reduced by 49.5% when rice husks and plantain peals were in cooperated to the drilling fluid, this was better than the standard mud additives, while filter cake increased by 38.9%. The rheological properties of the mud also increased. The results also showed that at 75g, plantain peeling concentration, the apparent and plastic viscosities increased by 11.3% and 16.1% respectively. The yield point reduced by 5.66%. From their results, it is evident that the use of plantain peelings and rice husks improves rheology and can be filtration control additives at substantial concentration. The research recommended establishing thermal stability of rice husks as a filtration control additive in dynamic conditions in WBM and OBM (Seteyeobot *et al.*, 2017).

The research on cassava brought forth the conclusion that cassava starch is good at advancing the rheological properties of water-based mud with an optimum concentration of 4% (Omotioma *et al.*, 2015).

Another research was also done and it was found out that extracts from mango and cashew nut leaves improved rheological properties in water-based mud. This is because it showed suitable results as mud additives. When the two plants are compared, mango leave extracts had higher improvement than cashew nut leave extracts (Oko *et al.*, 2015).

Dadge and Nmegbu (2014), on the other hand in his findings stated that cellulose from ground nuts shells could do better than polyanionic cellulose (PAC) when used to formulate the drilling mud. The results concluded that mud density and specific gravity of that mud were higher compared to that of standard mud. From the results the cellulose from ground nuts husk considerably reduce fluid loss hence it can be used as an agent for fluid loss control (Nmegbu and Bekee, 2014) .

A study using corn cobs and coconut shell in drilling mud formulations. The additives were in varied concentrations. They compared the mud containing corn cobs and the one containing coconut shell separately then mixed one corn cobs with coconut shell. The result showed that the mixture of coconut shell and corn cobs reduced filtration loss volume than the ones that were done separately. They also found out that corn cobs have better fluid loss control when compared to coconut shell (Student, 2017).

A study on lemon grass conducted found out that lemon grass performs as a good fluid loss control agent in oil-based mud. The filter cake obtained ranged between 2-25mm which is a requirement by API. Another finding was that 150 microns is a good size of lemon grass and therefore recommended its use in place of commercialized fluid control agent (Ghazali *et al.*, 2018)..

Majority of these studies choose their plant based mud additives based on their constituents that could enable the drilling mud to function better compared to the standard muds. For example, rice husks have 20% opaline silica and lignin. Combination of silica and lignin makes rice husks to be impervious to water and fungal penetration and also protects it from thermal decomposition (Khaleghian *et al.*, 2017). Rice husks also have good compressive strength when its concentration is increased (Kassim and Yong, 2017).

Plantain peelings on the other hand contain protein, ash, lipids, starch and dietary fibre (Khoozani *et al.*, 2019). These properties makes plantain peelings good for use as a rheological control and as a fluid loss control additive (Seteyeobot *et al.*, 2017)**.** Amylose is a component of starch and it controls the gelling behaviour. Amylopectin is large in size and it has branched nature which can reduce the movement of the polymer and their alignment in an aqueous environment. Starch molecules also have many hydroxyl groups which convey hydrophilic properties to the polymer giving it ability to disperse water. It is believed that these properties that made cassava to act as a filtration loss controller in drilling mud are due to the presence of hydroxyl groups (Rana *et al.*, 2019).

Studies were done on Canola, Moringa and algae seeds oils used as continuous phase of oil based mud and their results compared with diesel oil (Agwu *et al.*, 2015). Results indicated that when plot was done of dial readings against speed, they all exhibited Bingham plastic character. On pressure loss, Canola had best results and followed by Jatropha mud. Algae gave the best results on reduced friction followed by Jatropha then moringa oil based mud. Lower viscosities leads to low resistance (Parizad *et al.*, 2018) hence it implies that there will be less wear in drilling string. Filtration loss test showed that Moringa, Algae, Jatropha and Canola had better filtration properties when compared to Diesel oil based mud (Adesina *et al.*, 2018).

2.7 Importance of surface-active agents in drilling fluid

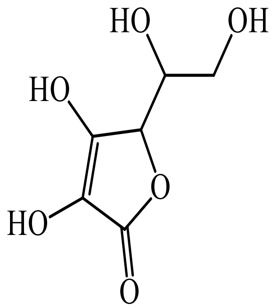
Surface-active agents are increasingly used in variety of applications in drilling fluids. It is used as emulsifiers and wetting agents in oil based mud. In water based mud, some of its many application includes; acting as an inhibitor in shale swelling, preventing wellbore uncertainties, lubricate drill bit, preventing cuttings from sticking to it and as dispersant helping the inhibition of flocculation in clay particles (Yunita *et al.*, 2015). It functions as a foaming additive to generate high gas/water ratio in reservoirs with low pressure and in drilling of hard rock. Presently, industries reduce chemical agents use and in return scientist explore natural substitute's e.g. natural surface-active agents (Tmáková *et al.*, 2016).

2.8.Selected plants in the study

2.8.1 *Carica papaya* (Paw paw)

Carica papaya belongs to family *caricaceae*. Its stem is usually weak and unbranched and crowned with long large cluster of leaves. Plate 2.1 shows *Carica papaya* (*C. papaya*) plant. The parts of various plants especially leaves have been used in treatment of diseases like malaria, dangue fever, jaundice, arthritis e.t.c. Various research have shown that the leaves of *C. papaya* have many compounds e.g. phenolic compounds, carotenoids, steroids and alkaloids. Thin layer chromatography examination revealed that leaves of *C. papaya* has polar compounds (Vij and Prashar, 2015). Agricultural activities and different climatic conditions, seasons and sites influence the content of *C. papaya* (Campostrini *et al.,* 2018). Most of these constituents exhibit efficacy against inflammations. The components of *C. papaya* leaves have demonstrated antibacterial, antitumor and immune modulatory activity. *C. papaya* leaves can be used in treatment of warts, corns, sinuses, eczema, cutaneous tubercles, and constipation and to expel worms etc. All these effects makes *C. papaya* to be considered as neutraceutical (Vij and Prashar, 2015). Studies on different parts of pawpaw has revealed that the plant is highly nutritive and the leaf is considered orally active and non-toxic as its lethal dose is $> 15g/kg$ body weight (Madjos and Luceño, 2019). Milk and juice of *C. papaya* leaves are extracted and used as chewing gum for digestive problems, toothpaste and tenderizers (Okunola *et al.*, 2012). Figure 2.1 shows some of the identified compounds of leaves from *C. papaya.* The leaves contain compounds such as ascorbic acid (**1**), carpaine (**2**), kaempferol-3 (**3**), papain (**4**) and tocopherol (**5**), amygdalin (**6**), halocalin (**7**) and prunasin (**8**). Papain from *C. papaya* is used in toothpaste, cosmetics and detergents. The reason why in the past leaves of *Carica papaya* were used to substitute soap could be attributed to the finding that *C. papaya* has saponins (Bisong *et al.*, 2018).

Plate 2.1: *Carica papaya* (pawpaw) plant



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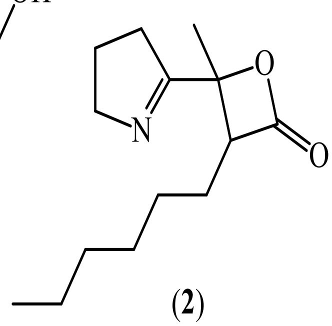

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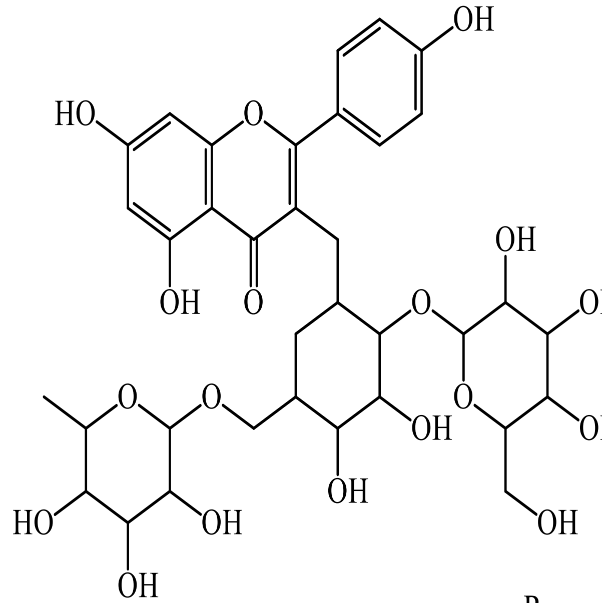

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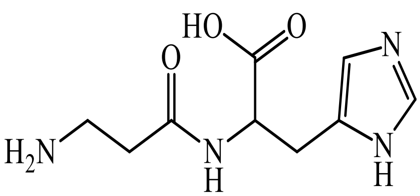

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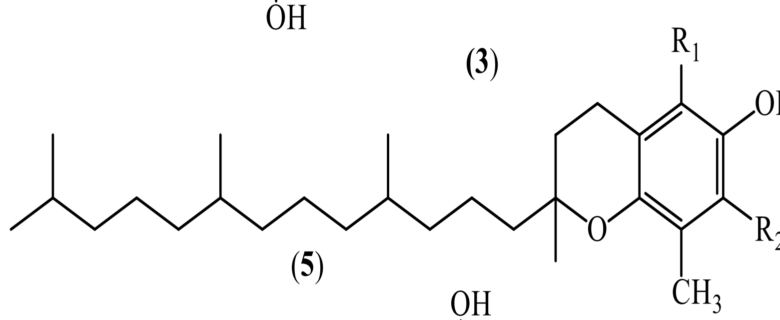

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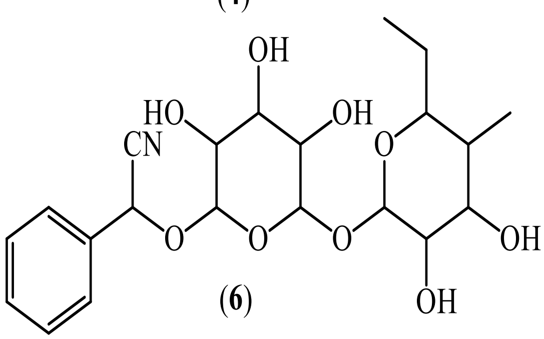

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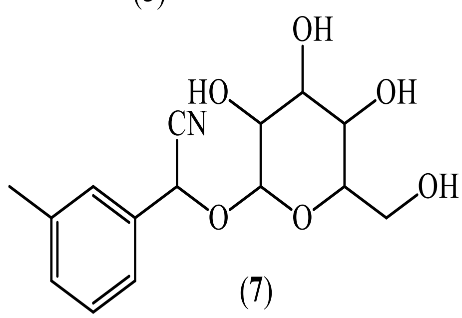

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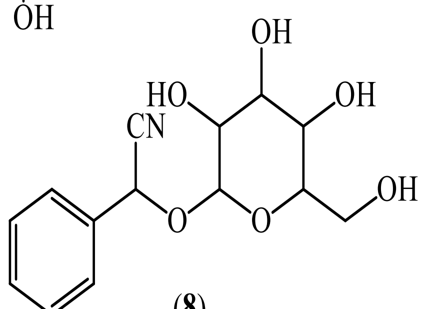


(**8**)

Figure 2.1: Some of the compounds found in the leaves of *C. papaya*

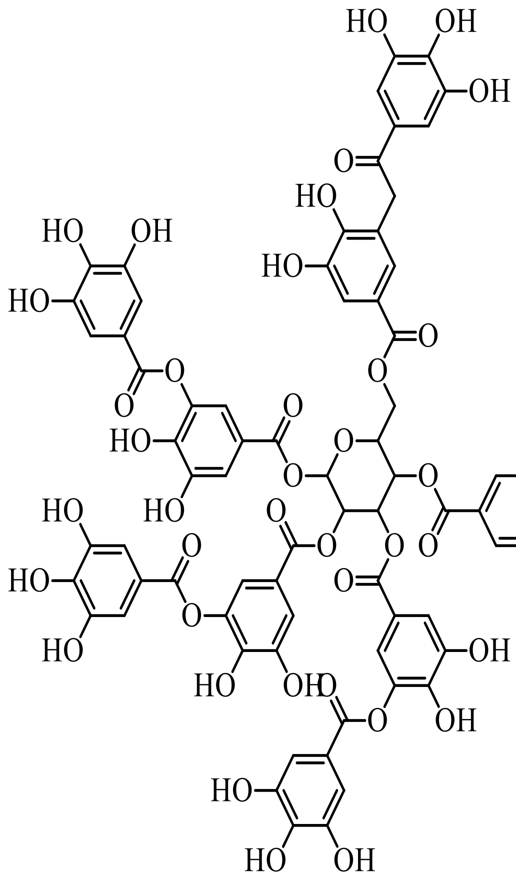
Papain (**4**) is known to hydrolyze proteins, amines and esters of amino acids moreso bonds involving the amino acids. Under prolonged incubation, bonds are divided more. Papain has esterase and transminase activity that are used in peptide synthesis (Kim, 2017). Tacopherol is a key component of vitamin E and their un-methylated carbons at position 7 and 5 act as electrophilic centers which can trap the reactive oxygen and nitrogen species (Hess, 2017). Cynogenic – glycosides are harmful to health because the cyanide hinders the cellular respiration hence oxygen cannot be available of the transportation of electron in the cytochrome (Fawole *et al.*, 2017). It is therefore good to investigate *C. papaya* activity in the drilling mud since much has not been reported on it as it can act as a source of surface-active agents (Soares *et al.*, 2018).

2.8.2 *Veronia amygdalina* (Bitter leaf)

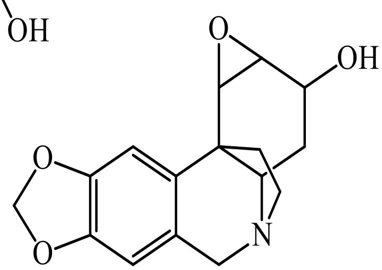
Veronia amygdalina belongs to the *Asteraceae* family and it grows throughout tropical Africa. It is known of its bitter taste and it is usually processed before cooking to remove the bitterness. It is reported that bitter leaf has saponins and saponins are usually known for their bitter taste and unpleasantness (Okunlola *et al.*, 2019). Plate 2.2 is an illustration of the *V. amygdalina* plant. Leaves from bitter leaf can be used to treat constipation, jaundice, stomach disorders, cough, wounds, venereal diseases and many more. Test on plasmodium proved that it is possible to use it as anti- malarial and has spermatogenic effect (Pelser, 2018). Documentations show that it can improve sperm concentration and mortality and toxicity test has revealed that it is non-toxic at doses of 500mg/kg per day (Kanyanga *et al.*, 2018; Tijjani *et al.*, 2017). Saponins in *V. amygdalina* also helps in reducing fever and pain (Ogidi *et al.*, 2019). Some of the studies revealed that there were twenty alkaloids, four tannins in the leaves of *V. amygdalina*. Figure 2.2 shows some of the compounds that have been identified from the leaves of *V. amygdalina*. The efficacy of *V. amygdalina* could be due to presence of tannins like tannic acid (**9**), alkaloids like crinamidine (**10**) and Lactupicrin (**11**) .(Ogidi *et al.*, 2019). It is during processing before cooking that the *V. amygdalina* produces foam and since more has been done on medicinal value, it is worth trying it in the drilling mud formulation because little has been done in it.

Plate 2.2: (a) *Veronia amygdalina* young leaves

Plate 2.2: (b) *Veronia amygdalina* old plant



(**9**)



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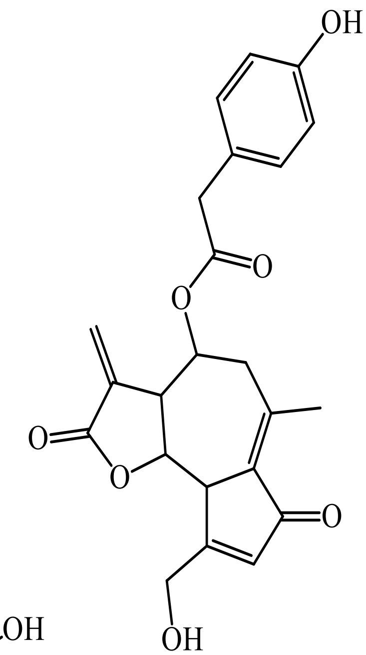


(**11**)

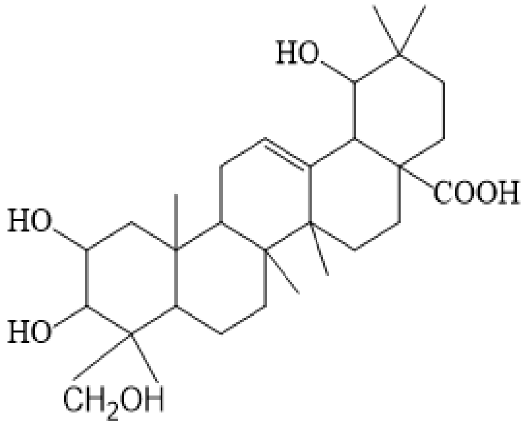
Figure 2.2: Some of the compounds found in leaves of *V. amygdalina* plant

2.8.3 *Terminalia mantaly* (Madagascar almond)

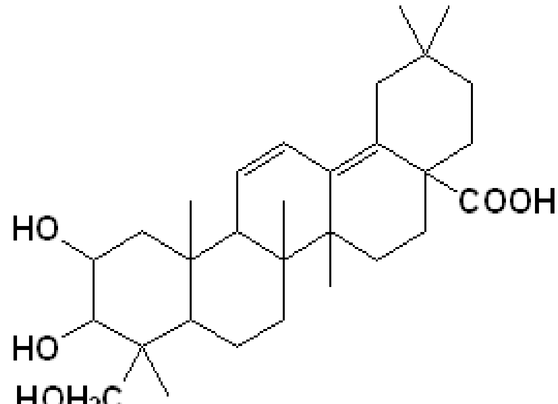
Terminalia mantaly is one of the deciduous trees with horizontal branches, sometimes termed as an umbrella tree. Most of the leaves turn brown before dropping during the winter or sometimes before the dry season in the tropics. Plate 2.3 gives an illustration of the *T. mantaly* plant. Its leaves are replaced instantly when the season changes (Mbouna *et al.*, 2018). People utilize the plant as shade or to beautify the surrounding. Because it is drought resistant, it is used mainly for reforestation especially in sandy areas (Lierop *et al.*, 2015). Its leaves drop frequently and so they provide good mulch hence leading to soil and small plant protection. As an umbrella-shaped, it looks attractive (Szwagrzyk *et al.*, 2017). The trunk of *T. mantaly* acts as a source of gum and the plant parts like leaves produce tannin and dye which is used in leather preparation (Gichora *et al.*, 2017). Many findings reveal that *T. mantaly* has the potential to produce many secondary metabolites. Since leaves of *T. mantaly* falls easily, and are replaceable, they are worthy to use in the petroleum industry where not much has been done on it. Figure 2.3 shows some of the compounds that have been identified from the leaves of *T. mantaly*. The following are some of the compounds of *T. mantaly* leaves: coarjungenin (**12**), arnjuglucoside (**13**), ellagic acid (**14**), 3, 3´ di-o methylellagic acid (**15**) and 2,3,24-trihydroxyolean (**16**) (Toghueo *et al.*, 2017).

Plate 2.3: (a) *Terminalia mantaly* used to beautify a place

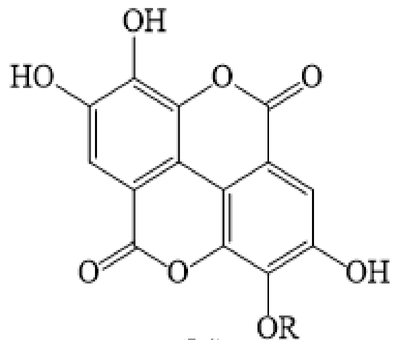
Plate 2.3: (b) *Terminalia mantaly* leaves



(**12**)



(**13**)



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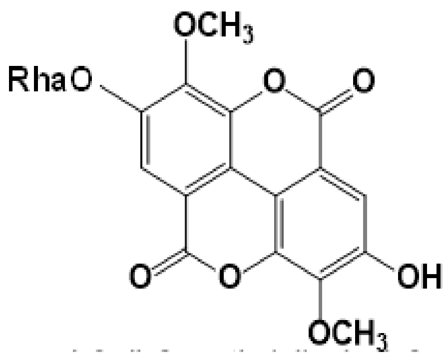
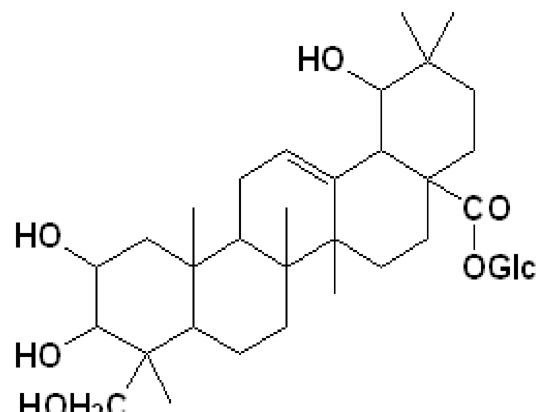


(**15**)



(**16**)

Figure 2.3: Some of the compounds that have been identified in the leaves of *T. mantaly*

2.8.4 *Moringa oleifera* (Moringa)

Moringa oleifera is a drought-resistant plant growing mostly in the tropical and subtropical areas and it is widely known for its medicinal values. Figure 2.7 shows the *M. oleifera* plant. *M. oleifera* leaves are the most nutritious unlike other parts of the plant. The leaves are known to have vitamins (B, C, A and K), beta carotene as well as proteins. The leaves are crushed and cooked with soups (Dhimmar *et al.*, 2015) and used in Africa to fight malnutrition. It is also used to treat degenerative diseases like cancer, asthma, and rheumatism (Padayachee and Baijnath, 2019). Other diseases that *M. oleifera* treats are epilepsy, parasitic infections, fungal and bacterial infections. Apart from these uses as medicine, *M. oleifera* powder is used for hand wash as a soap and as an antiseptic. Its seeds are known to have oil (Rashid *et al.*, 2008), which is utilized to cook food, hair care products and as machine lubricant (Ramasubramania *et al.*, 2016).

The *M. oleifera* tree is usually cut yearly to about one meter and allowed to regrow. Studies show that *M. oleifera* is rich in nutrients, anti-oxidants and can cure many diseases since it contains the majority of vitamins found in fruits and vegetables. *M. oleifera* is considered the most important plant e.g. it can be used as a vegetable, medicine, food security, sustaining land care and nurturing rural development (Martin, 2007).

Figure 2.4 shows some identified compounds from the leaves of *M. oleifera.* Among major compounds found in *M. oleifera* are Beta- 1- amnofuranoside (**17**), cyclopentatriol (**18**), 3,4- dichlorobenzonitrile(**19**), L- galactose (**20**), n-Hexadecanoic acid (**21**), palmitoyl chloride (**22**), and gamma –sitosterol (**23**), 7-diene-3-of-20-one (**24**), stigmasterol (**25**) and stigmasterol 3-OB-D- gluo pyranoside (**26**). Acids in *M. oleifera* releases hydroxyl ions in water hence making the solution alkaline (Yuliastri *et al.*, 2016). Further reports indicate that *M. oleifera* seeds are a useful source of plant oil for diesel substitute production (M.

Amanullah and Ramasamy, 2019) but more work needs to be done on *Moringa oleifera* leaves in the petroleum industry because little has been reported on it.

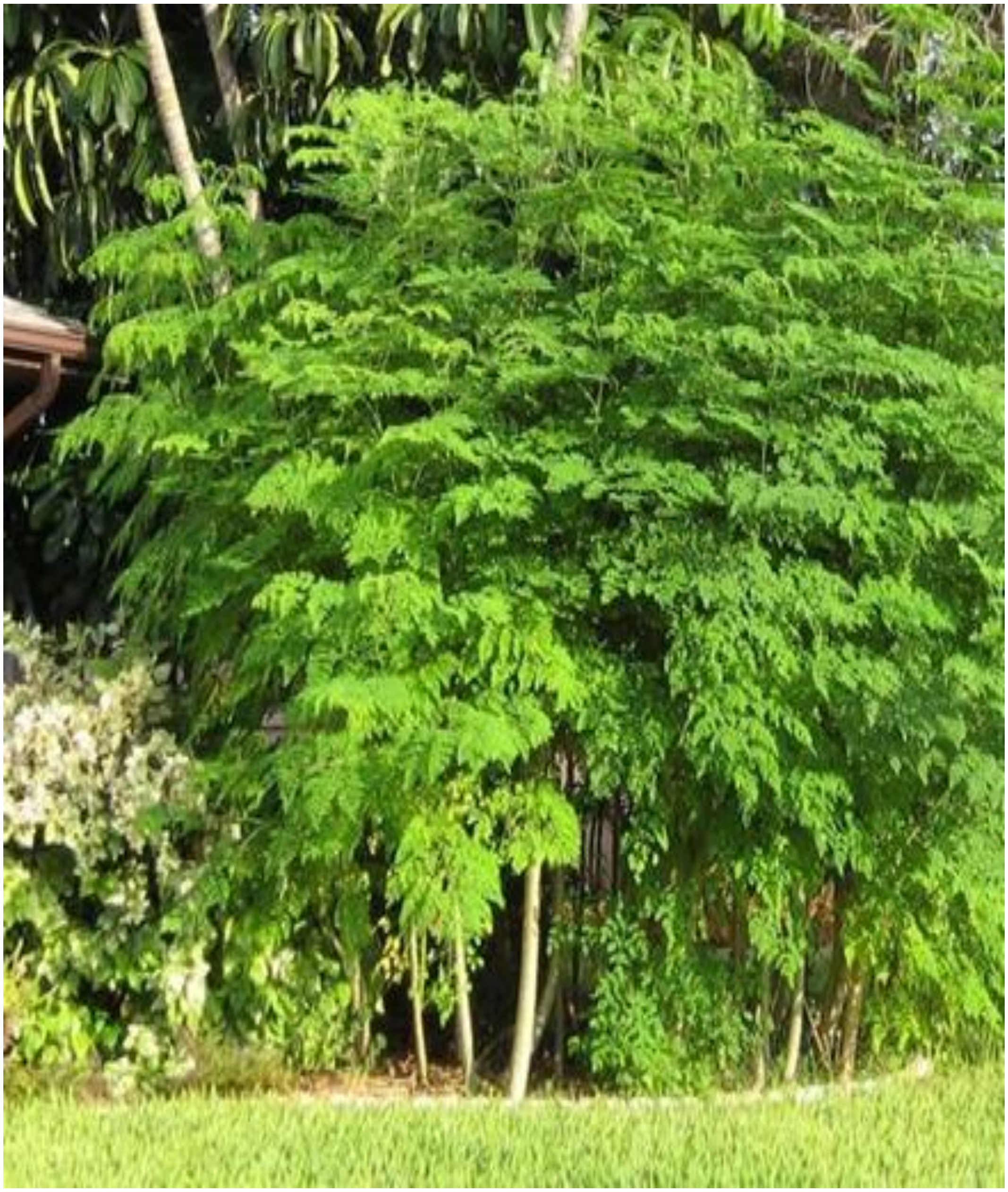
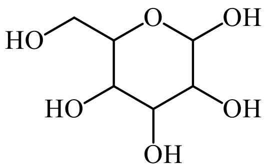
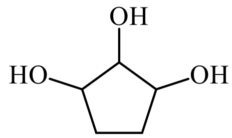


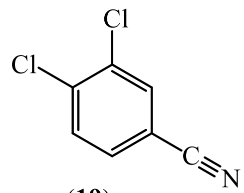
Plate 2.4: *Moringa oleifera* plant pictures



(**17**)



(**18**)



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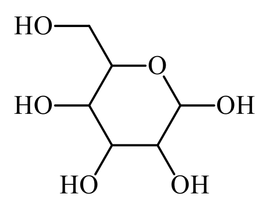

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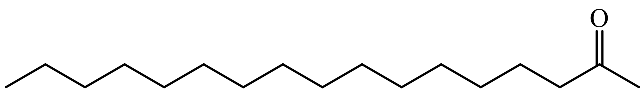

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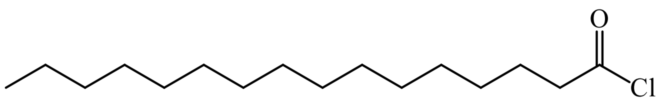

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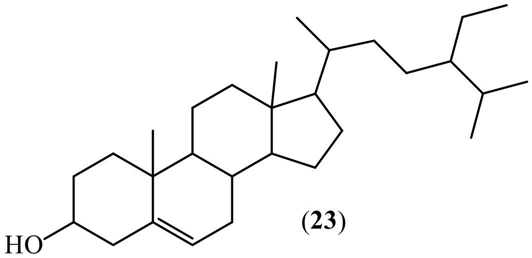

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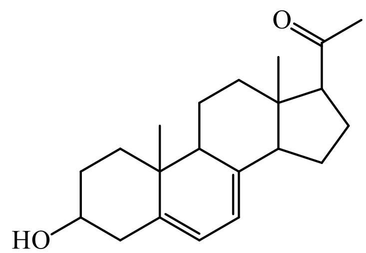

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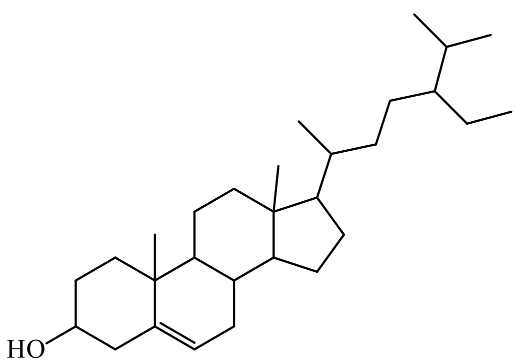

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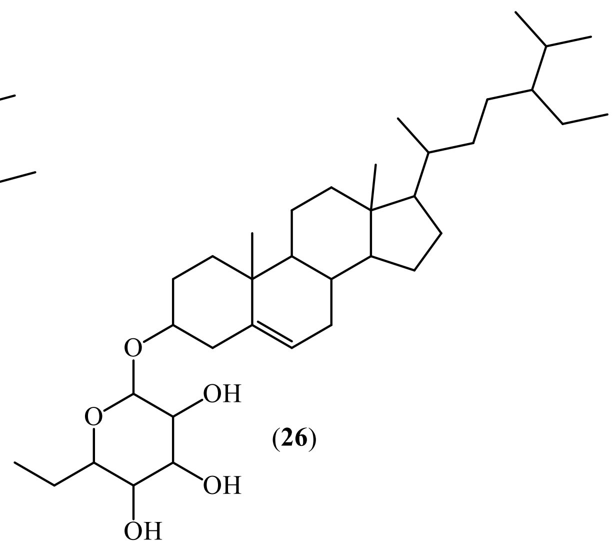


(**26**)

Figure 2.4: Compounds found in the leaves of *M. oleifera*

2.9 Surface-active compounds from plants in drilling mud formulation

Natural surface-active agents are derived either from plants or animals. Non-ionic bio-surfactants like saponins originate from plants. Structurally, surface-active agents have diversified due to their emulsifying and foaming agents' characteristics which are caused by hydrophobic and hydrophilic nature (Muntaha and Khan, 2015).

The physicochemical properties of surface-active agents give them relevance for use in industrial applications. For instance, surface-active agents have the ability to form supramolecular structures like liquid crystals, vesicles, mono-layers, bilayers, multi-layers and micelles by adsorption and aggregation. Surface-active agents are applicable both in edible and non-edible uses (Sharma *et al.*, 2018).

Surface-active agents also have a sweet and bitter taste. Surface-active agents have hydrophobic and hydrophilic molecules that give them foaming and emulsifying property (El-Sayed and Ahmed, 2016). Their biological properties like antimicrobial, insecticides, etc. are useful in food, pharmaceutical, and cosmetic industries. The application of surface-active agents in industries leads to negative environmental impacts because large quantities are discharged in diverse environments. Compounds in synthetic surface-active agents can lead to health risks like respiratory irritation, dermatitis and eye problems (Muntaha and Khan, 2015). Owing to their natural origin, natural surface-active agents have low negative impacts on the environment, good bio-compatibility and are available in large quantities. They can work effectively in extreme conditions like pH, temperatures, and salinity (Tmáková *et al.*, 2016). In industries that produce gas and oil, surface-active agents are used in the formulation of oil-based and water-based drilling muds. Surface-active agents are also used in enhanced oil recovery, oil-water gas separation, corrosion inhibitors and dispersing agents. Surface-active agents also have been utilized to reduce rheology, as a lubricant and lower the risk of water blockage (Okoro *et al.*, 2015). Some researchers have found out that the use of surface-active agents decreased the amount of lost filtration and it improved the overall performance of water-based mud (Oseh *et al.*, 2019).

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials

3.1 Materials

Purchase of all the reagents and chemicals used was done from Scientific Laboratory Suppliers, the U.K. They were all of the analytical grade (AG). Solvents used were hexane, acetone, methanol, and chloroform. Chemicals used were, Bentonite clay, Barite, Potassium Chloride (KCl), hydro polyanionic cellulose low viscosity (Hydro PAC LV), caustic soda (NaOH) and ARBREAK (commercially available surfactant).

Among the equipment and apparatus used for sample analysis were; FT-IR (Perkin- Elmer, Beaconsfield, (UK Spectrum one FT-IR spectrometer), Fann HPHT filter press 387 model (Fann instrument company, Huston, Texas, U.S.A), Hamilton Beach mixer (commercial, U.S.A), Roller oven MM 8601 model (Baroid, U.S.A), OFITE viscometer 900model (OFI testing equipment, U.S.A patent 6,776,028), pH meter 081439 model (Thermo electron corporation, U.S.A) and Baroid Mud balance 140 model (U.S.A).

3.2 Methods

3.2.1 Sample collection, identification, and preparation

Sample plant leaves were obtained from the Choba area in Port Harcourt, Rivers states, Nigeria. The geographical coordinates of Port Harcourt are 4° 54’ 10.4724” North, 7° 0’4.5468” East. Taxonomic identification of fresh green leaves was carried out at the University of Port Harcourt in the department of Plant Science and Biotechnology. The leaves were shade-dried for one month before grinding into a fine powder. The powdered plant’s samples were kept in airtight containers, labeled and kept in a cool dry place.

3.2.2 pH of the plant sample

Fine powder of 4 g each from the plants were mixed with 10mL of distilled water and shaken slightly for 30 seconds and left to settle down for 10 minutes before taking the pH measurement.

3.2.3 Extraction of plant material

The ground powder of 200 g each was soaked in three different solvents: hexane, acetone, and methanol of 500 ml each at room temperature of (25°C) for 48 hours with occasional shaking. The next process was filtering with Whatman No. 1 filter paper and rotary evaporation was done to eliminate the solvent. The crude extracts were then transferred into airtight containers and reserved for more analysis.

3.3 Characterization of extracts

3. 3.1 Phytochemical screening

Phytochemical screening of saponins in crude extracts was done using standard procedures (Ajuru *et al.*, 2017)

Saponin test: To test for the presence of saponins, 2 g of plant extract was treated with 1% lead acetate. The presence of saponins is indicated by the formation of white precipitate.

Foam test: "Robot Marie" Model was used to determine foam characteristics of the plant powder.

Foaming properties of the extract were determined using 2 g powder in 100 ml of distilled water. The content in every measuring cylinder was covered with hand palm both top and the bottom. Thorough Shaking of the mixture was done 50 times then it was allowed to settle in the cylinder for 1 minute. Measurements of the foam volume was taken immediately and at the initial time. Measurements were also recorded during the time of the complete disappearance of the last trace of the foam. The presence of saponins is indicated when a stable foam is formed (Eka *et al.*, 2017). The experiment was conducted in triplicates.

3. 3. 2 FTIR characterization

All the crude extracts from the four plants were subjected to FT- IR spectroscopy. FT-IR results were recorded in the solid-state as KBr pellet, the dispersion was done using the FT-IR spectrometer.

The crude extract was mixed with KBr (IR- grade) at a ratio of 1:100 and pressed to a pellet. The pellet was put into the sample holder of Perkin Elmer Spectrophotometer and the scanning range was between 4000-500cm^{-1}. Functional groups present in the crude extracts of the plants were obtained through the spectral data.

3.4 Formulation of water-based mud

3.4.1 Formulation of standard water-based muds (Control mud)

It was done through negative and positive control and mud formulation which encompassed the following ingredients: Water, bentonite, caustic soda, and potassium chloride, hydro PAC (LV), barite and ARBREAK (industrial surfactant). After five minutes, the samples were added in a mixer and then as per set standards left to mix for 45 minutes (Yunita *et al.*, 2015).

3.4.2 Formulation of water-based mud using plant extracts

Plant extract was added in water-based mud with the other additives and mixed the same way as the control drilling muds were formulated (Yunita *et al.*, 2015)but with little modification, as shown in Table 3.1.

Table 3.1: Drilling mud formulations

ADDITIVES	FORMULATION 1 (C1)	FORMULATION 2 (C2)	FORMULATION 3 (Test mud)	FUNCTION
Water	318.74ml	318.74	318.74	Base fluid
Bentonite	17.50g	17.50	17.50	Viscosifier & Fluid loss control
Caustic Soda	0.25	0.25	0.25	pH control
KCl	10.70	10.70	10.70	Inhibitor
Hydro PAC LV	3.00	3.00	3.00	Fluid loss control & Viscosifier
Barite	68.72	68.72	68.72	Weighting agent
Plant extract	-	-	1%, 2%, 3% and 4%	Rheology modifier
Commercial surfactant	-	1%, 2%, 3%, and 4%		

Key: C1- Mud without plant sample or any surface-active agent

C2- Mud with commercial surfactant (ARBREAK)

NB: *Plant extract varied depending on the investigated plant type.

Selected plants were: *Terminalia mantaly, Carica papaya, Madagascar almond, Veronia amygdalina*

3.5 Characterization of drilling muds

3.5.1 Mud weight

Mud density was measured using Baroid mud balance and adjusted to account for friction pressure drop in an annulus giving equivalent circulating density. The total volume was given by the sum of the component after assuming that the resulting mud mixture from all the

additives and water were ideal; (Werner *et al.*, 2017).

3.5.2 pH of the formulated mud

The pH of formulated muds and the plants' powder without formulation were determined using pH meter. Readings were done at room temperature of 25°C (Mansoor *et al.,* 2018). After drilling mud was formulated, they were then left for 30 minutes and the pH tested. pH was also tested after each aging temperature (Brian, 2016).

3.5.3 Rheological properties

The procedure that was used in measuring the rheological properties was the standard of the American Petroleum Institute (API).

3.5.3.1 Plastic viscosity and yield point

A FANN VG viscometer was used to measure plastic viscosity (PV) and yield point (YP). The viscometer has a heating jacket to control the temperature using the API standard method. The relationship between shear rate and share stress was obtained through the readings. Shear rate PV and YP were calculated by using Equation 3.1 and 3.2 (Elkatatny, 2019).

$$\text{PV (cP)} = 600\text{ rpm} - 300\text{ rpm} \quad 3.1$$

$$\text{YP (lb/100 ft2)} = (\text{plastic viscosity}) - (300\text{ rpm}) \quad 3.2$$

The dial readings were recorded at different rotations per minute (RPM) i.e 600, 300, 200, 100, 6 and 3. All the readings were taken at different temperatures of 30°C, 49°C, 50°C and 70°C. The difference in temperature may demonstrate the raised temperature at the bottom hole (Mohammed, 2017).

3.5.3.2 Gel strength

The gel strengths of the fluids were measured to know how temperature and plant concentration affect the fluid's gel strength. Two readings were taken, after agitation of the mud in the cup for 10 seconds and 10 more minutes to allow the mud in the cup to settle. A comparison of the two readings was done to see the strength of the mud as either Progressive or flat in different mud formulations. The viscometer can test the properties of the drilling fluid such as viscosity and gel strength, under different temperatures (Neshat and Shadizadeh, 2016).

3.5.4 Effect of temperature (Aging)

A thermal stability test was conducted. This was done through heating the drilling mud at room temperature (25°C) to a testing temperature (35,65 and 93°C) using roller oven for 16 hours and then cooling it to ambient temperature according to (He *et al.*, 2016). Formulated mud with 2% concentration was chosen. Hence, the rheological properties of the mud were taken after exposure to three different aging temperatures. This was because the viscosity of the mud is a role of temperature than it is of pressure. Three temperatures were chosen for aging 35°C (believed to be subsurface temperature), 65°C and 93°C is the temperature between surface temperature and bottom whole temperature (Ahmadi *et al.*, 2017; E. Hosseini *et al.*, 2017). Tested properties after aging were: pH, mud weight, rheological properties, filtration loss and filter cake.

For the aged sample, the rheology was tested at 49°C which is a standard temperature to perform rheology tests according to American Petroleum Institute (API) and it is mostly used in an oil field test on rheological parameters (Dias *et al.*, 2015). The results were compared with that of un-aged samples data at 49°C.

3.5.5 Filtration loss

For the filtration characteristic of the mud to be determined, the filtrate volume was measured. API fluid loss apparatus Fann HPHT filter press was used at 93°C and 500psi and the filtrate obtained after 30 minutes (ΔP = 500 psi, no. 5 Whatman filter paper). The volume of filtrate loss was recorded from the graduated cylinder at the end of 30 minutes. Filtration loss test was also done for the aged samples (Agwu *et al.*, 2015).

3.5.6 Filter Cake

At the end of the filtration loss test, the filter paper in the fluid loss cup was removed and the mud cake was measured using a tape measure according to (Agwu *et al.*, 2015).

3.6. Data analysis

All the data were obtained in triplicates and data were expressed as mean ± SEM. The data were subjected to multivariate analysis of variance (MANOVA) using SPSS and statistical significance was obtained at $P < 0.05$.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Results

4.1.1 Sample collection and preparation

Table 4.1 shows how the samples were labeled. The selected particle size for plant leaves was 250 µm.

Table 4.1: Table showing the plants' names and labels used in the research.

Plant name	Plant powder Labelling	Mud labeling
***Carica papaya* (Pawpaw)**	PL	MPL
***Moringa oleifera* (Moringa)**	ML	MML
***Terminalia mantaly* (Madagascar almond)**	TL	MTL
***Veronia amygdalina* (Bitter leaf)**	BL	MBL

4. 1. 2 Physicochemical characterization

Foaming tests results and pH of the powdered sample solution are shown in Table 4.2

Table 4.2: Foam forming ability and pH of the plant leaves solution

Plant sample	***Foam length (mL)***	***Time taken for the foam to disappear (minutes)***	***The pH of the plants leaves solution***
ML	10	28.33	5.56
TL	20	65.66	4.61
BL	56	89.33	6.57
PL	25	89.66	6.37

Qualitative test on saponins are in Table 4.3

The plants' sample pH was examined at ambient temperatures (25°C) and the results are displayed in Table 4.3

Table 4.3: Qualitative test for saponins with different crude extracts

Solvent	ML	TL	BL	PL
Hexane	+	+	+	+
Chloroform	+	+	+	+
Methanol	+	+	+	+

Key: + (positive) = presence of saponin

4.1.3 Spectroscopic studies

Plants' crude extracts from different solvents were subjected to Fourier Transform Infrared (FT-IR) to check the presence of functional groups that surface-active agents have. Figures 4.1 to 4.12 shows the major peaks that were detected in the spectra and their absorption frequencies.

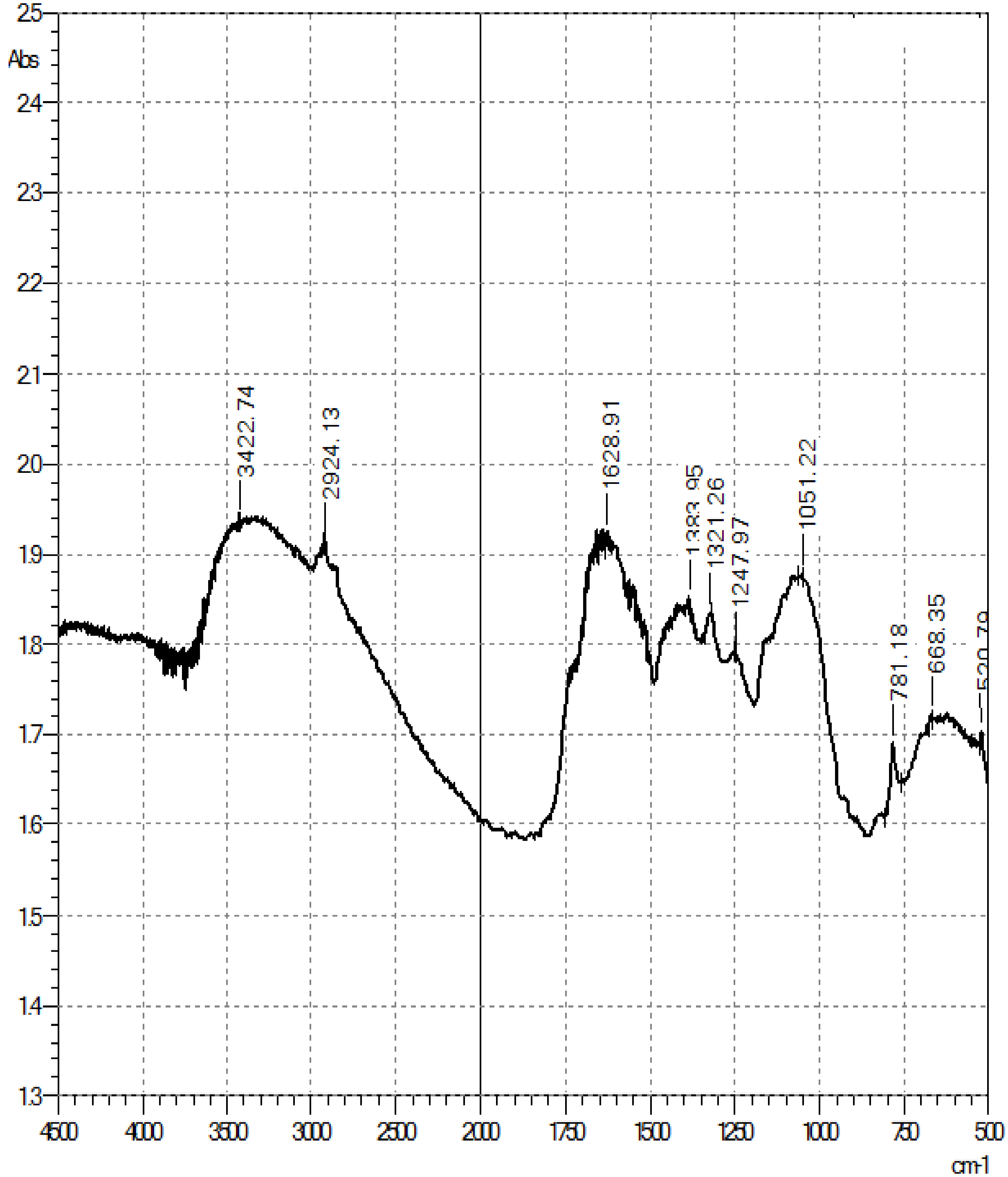


Figure 4.1: FT-IR spectrum for PL powder

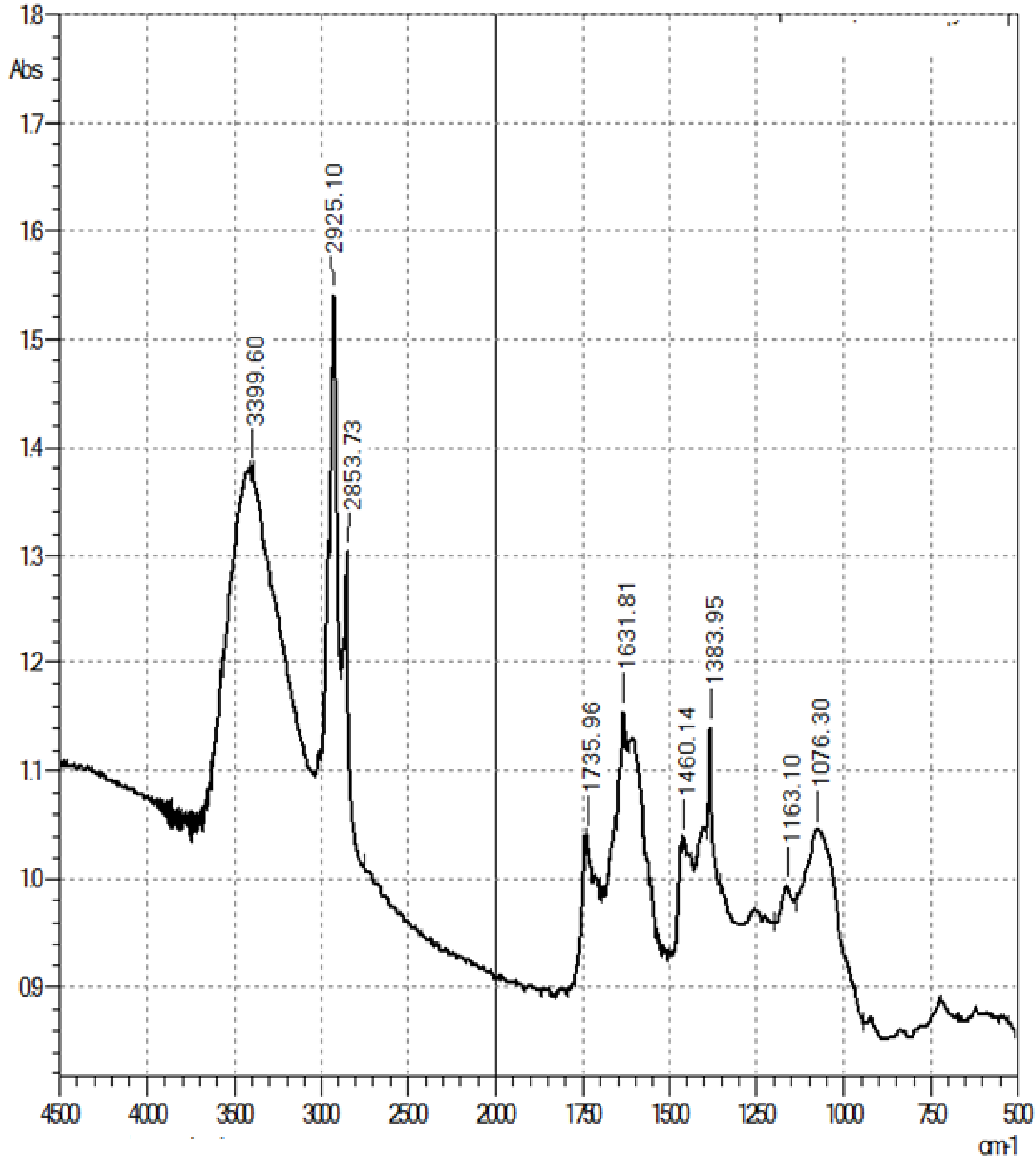


Figure 4.2: FT-IR spectrum for PL from hexane extract

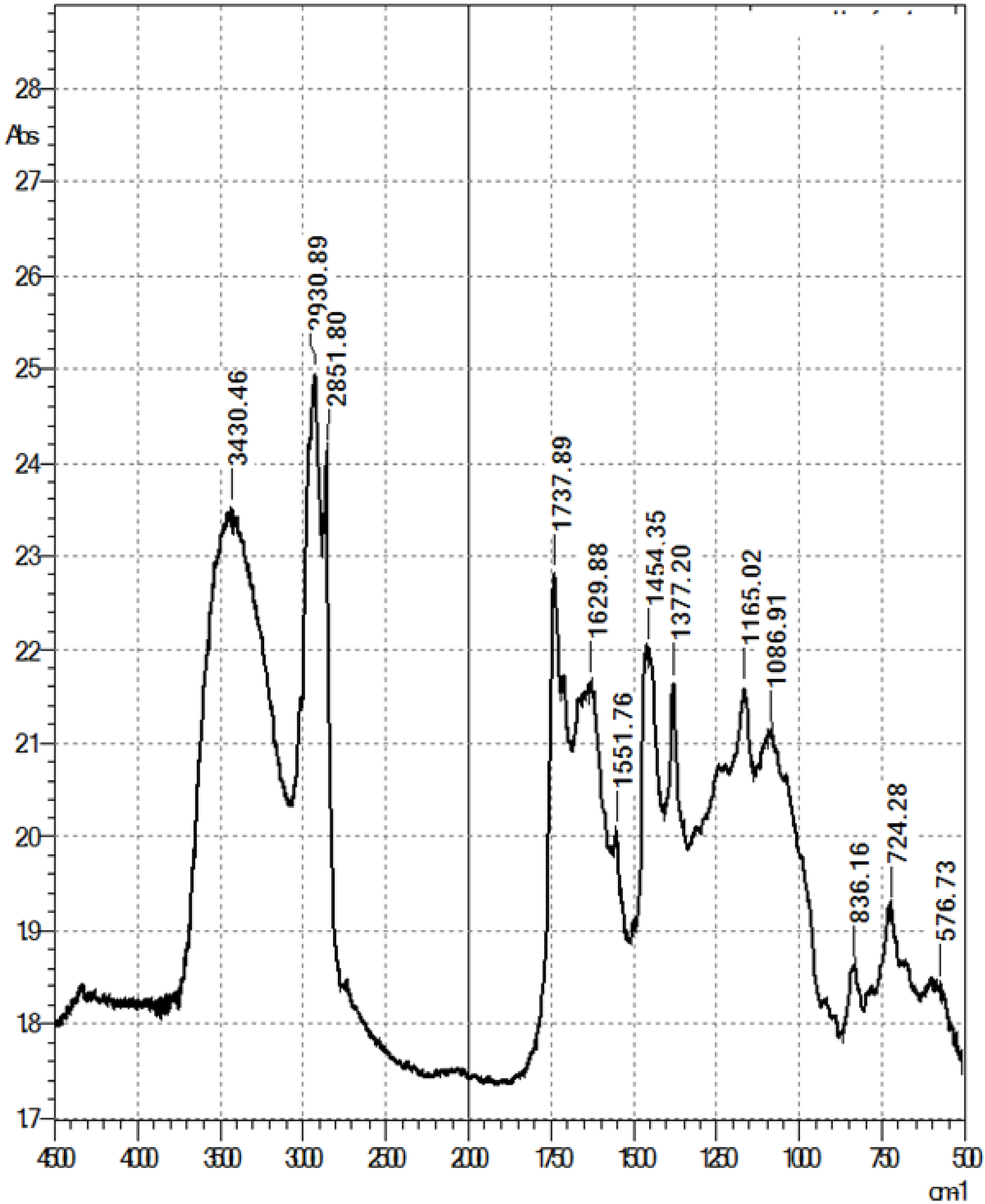


Figure 4.3: FT-IR spectrum for PL from the chloroform extract

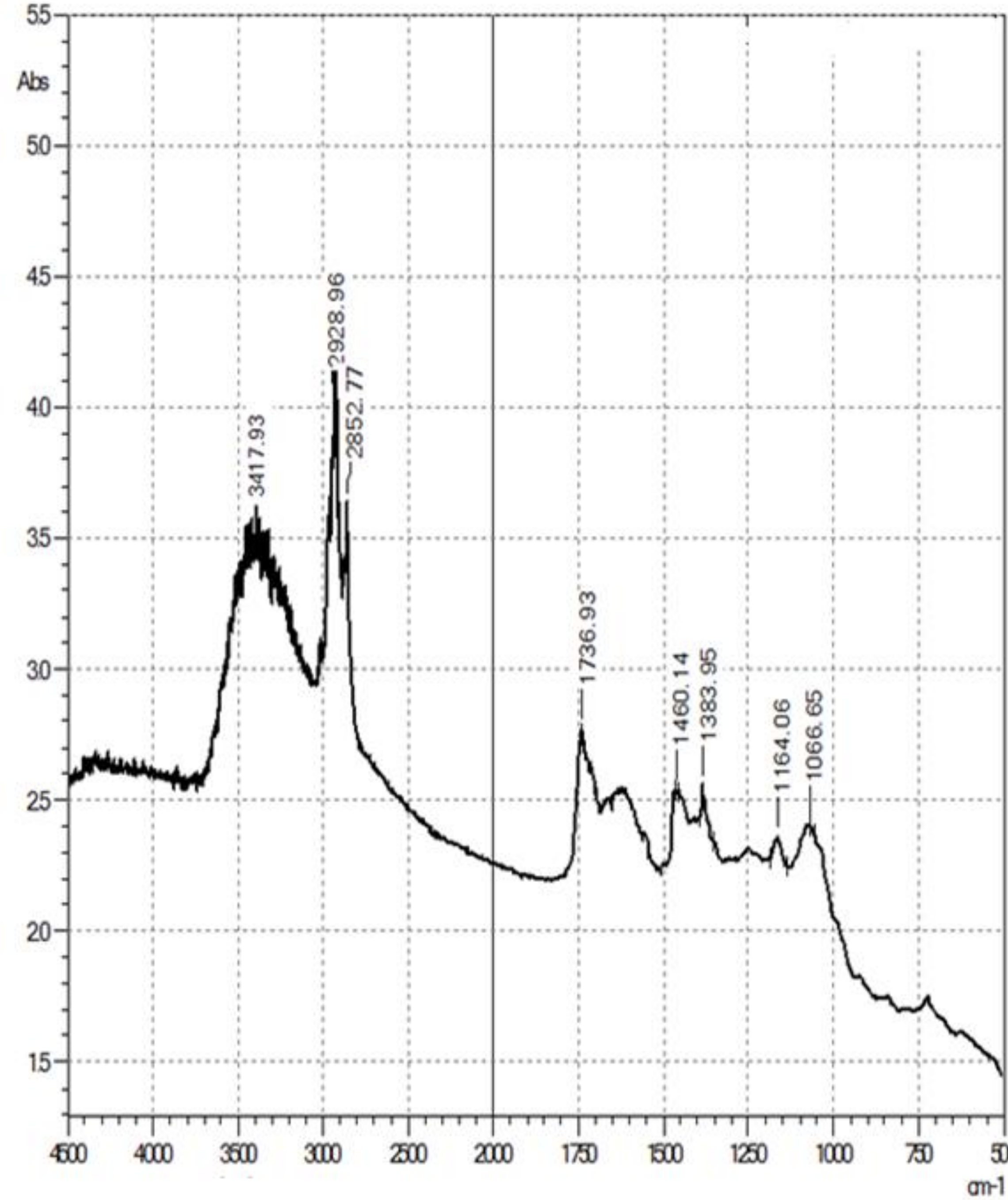


Figure 4.4: FT-IR spectrum for PL from methanol extract

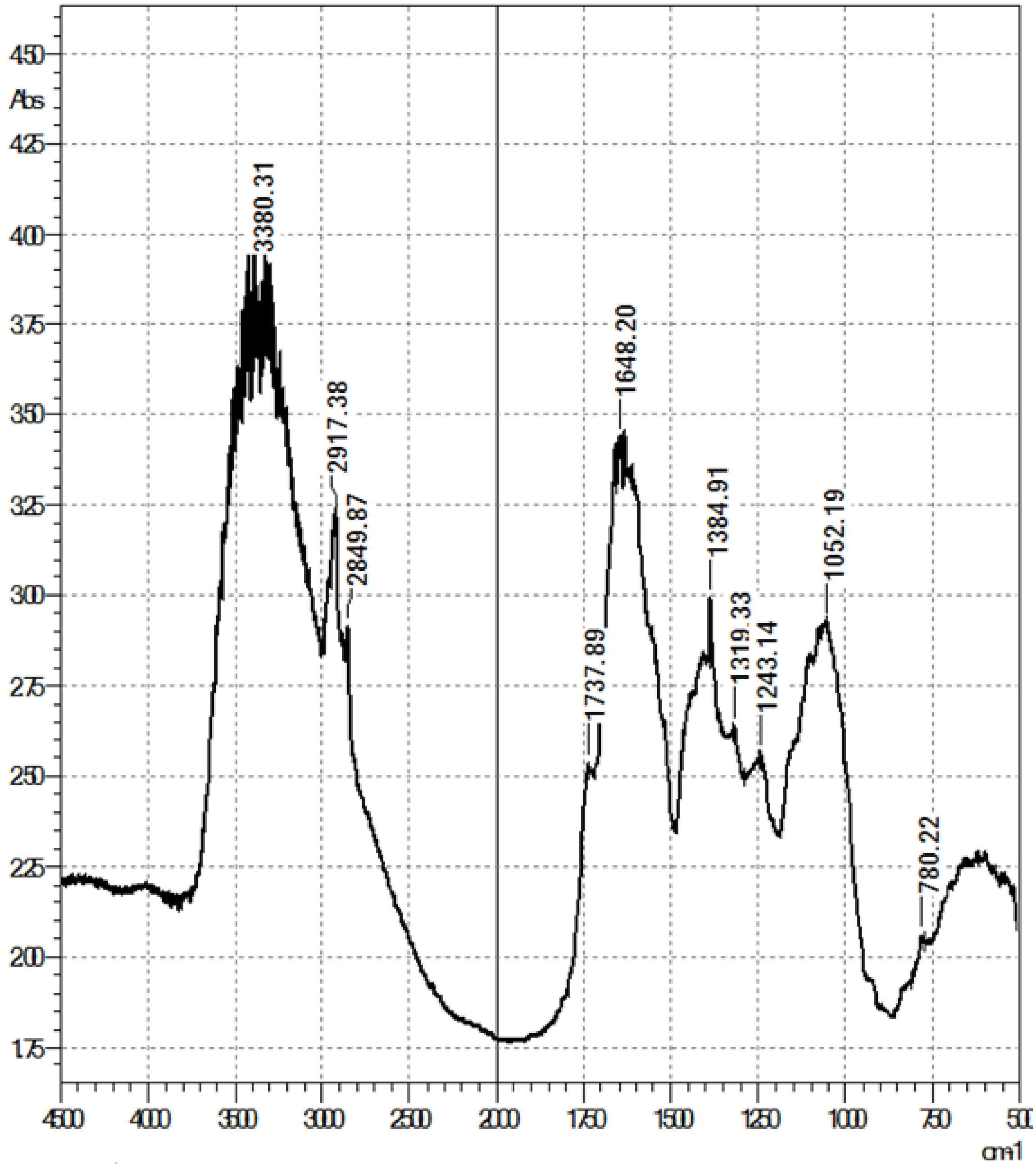


Figure 4.5: FT-IR spectrum for ML powder

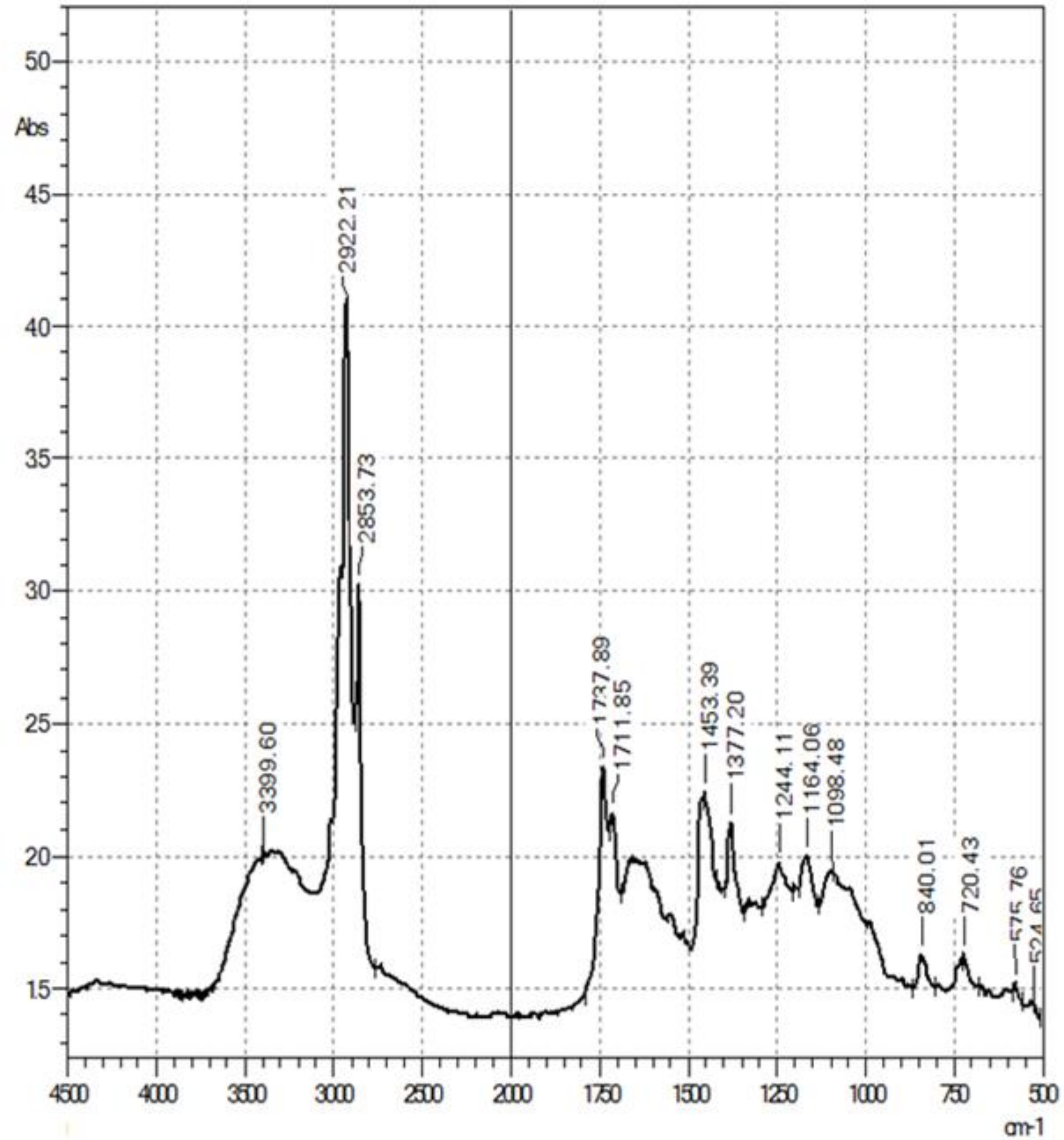


Figure 4.6: FT-IR spectrum for ML from hexane extract

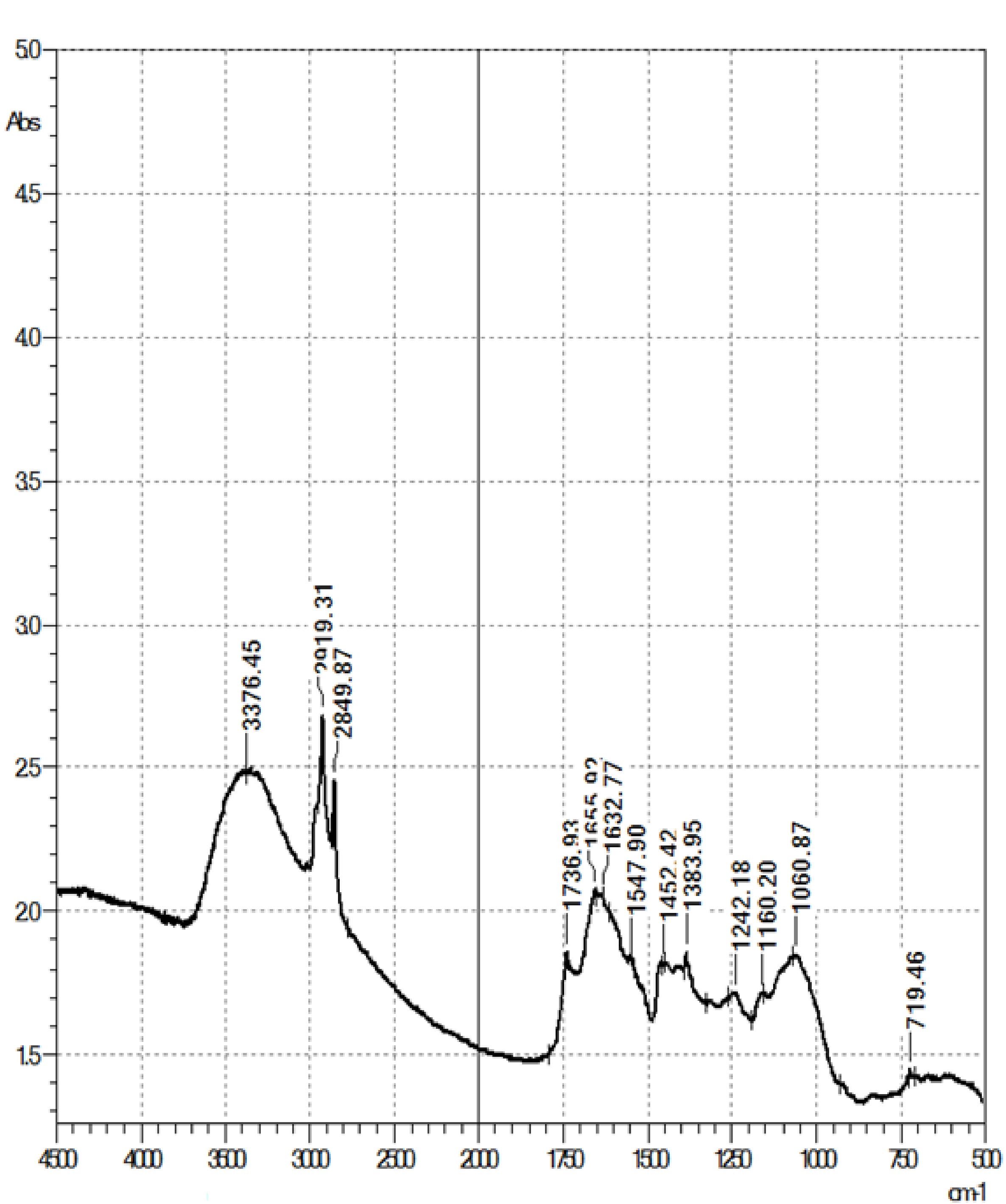


Figure 4.7: FT-IR spectrum for ML from the chloroform extract

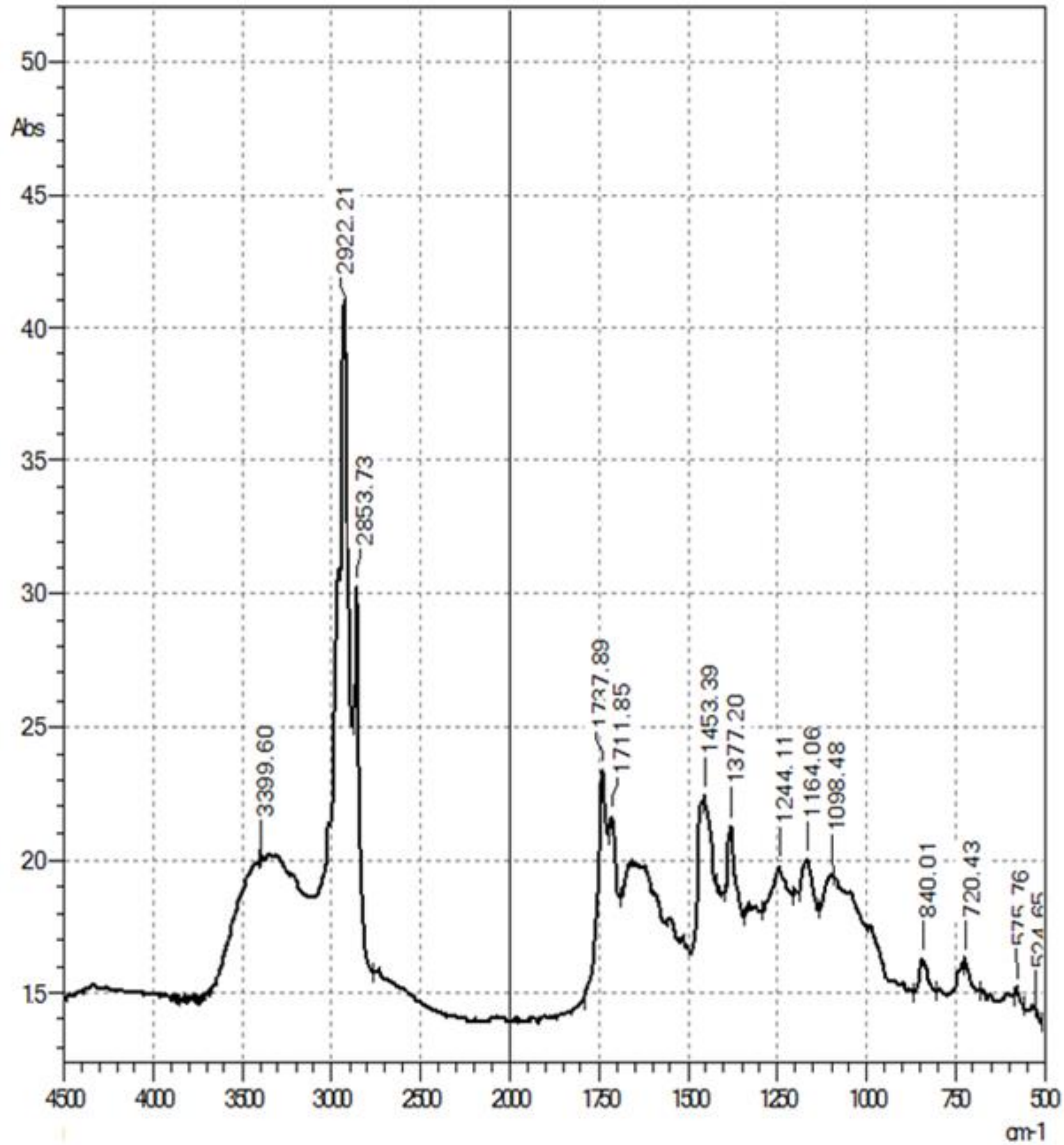


Figure 4.8: FT-IR spectrum for ML from methanol extract

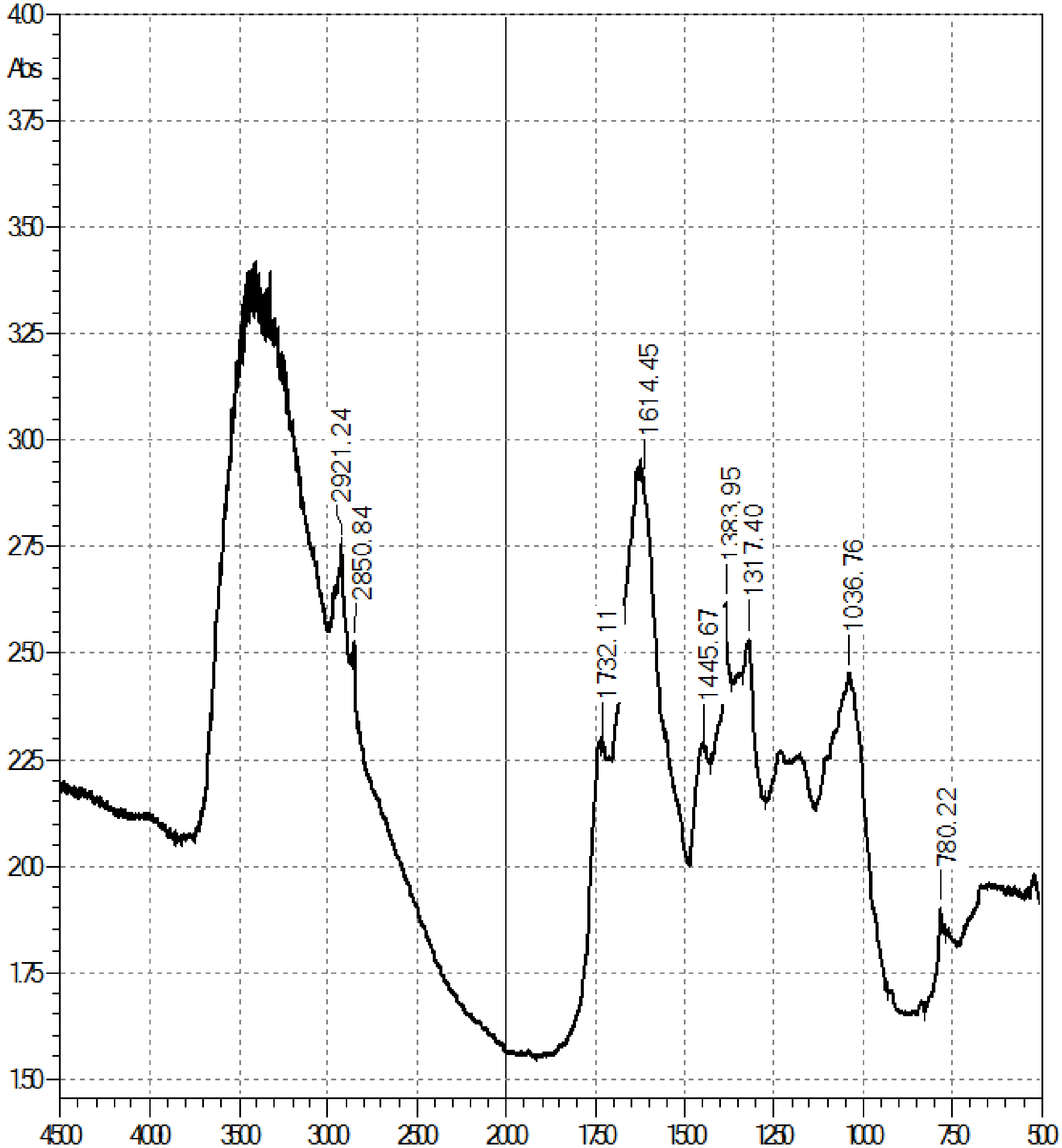


Figure 4.9: FT-IR spectrum for TL powder

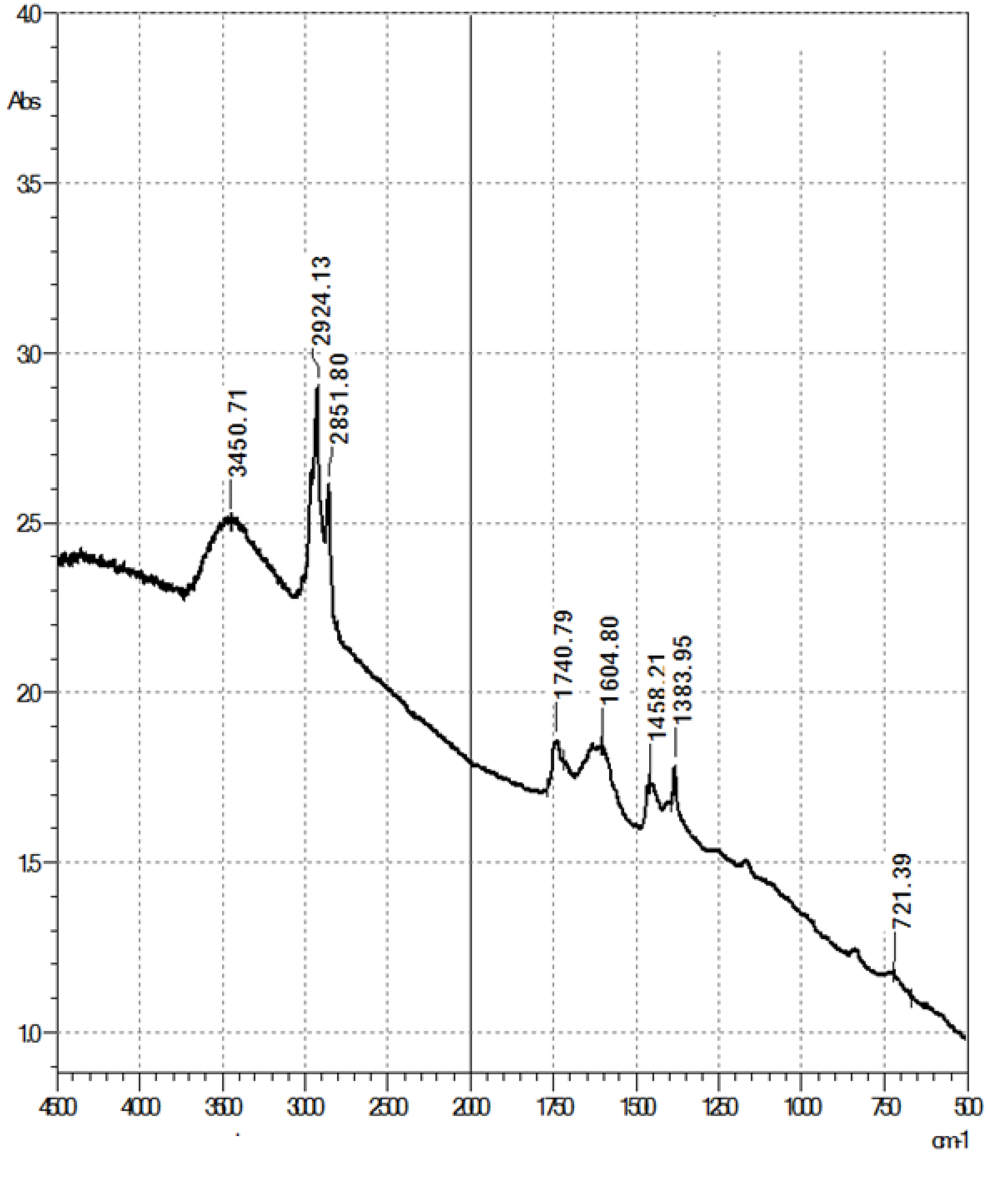


Figure 4.10: FT-IR spectrum for TL from hexane extract

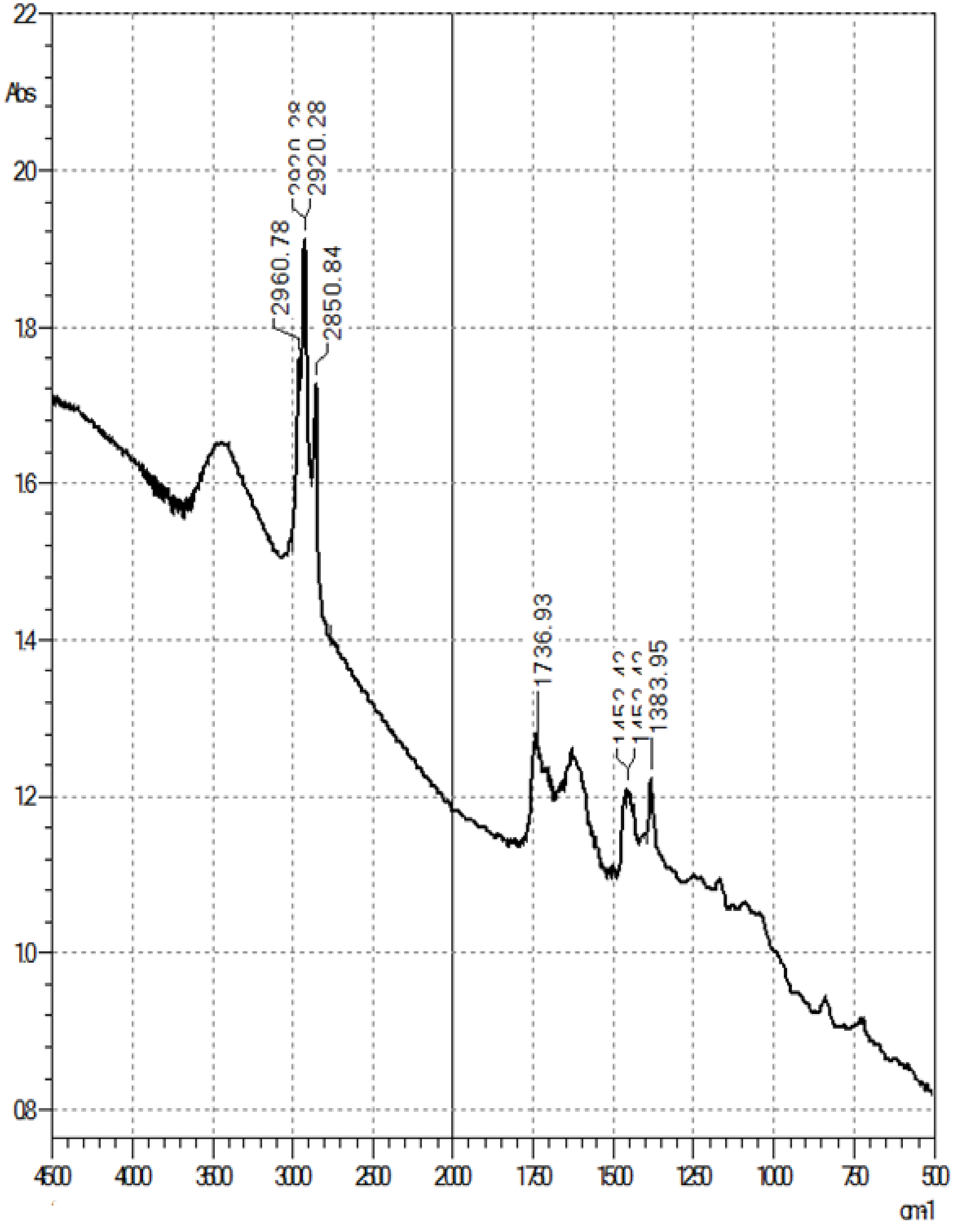


Figure 4.11: FT-IR spectrum for TL from the chloroform extract

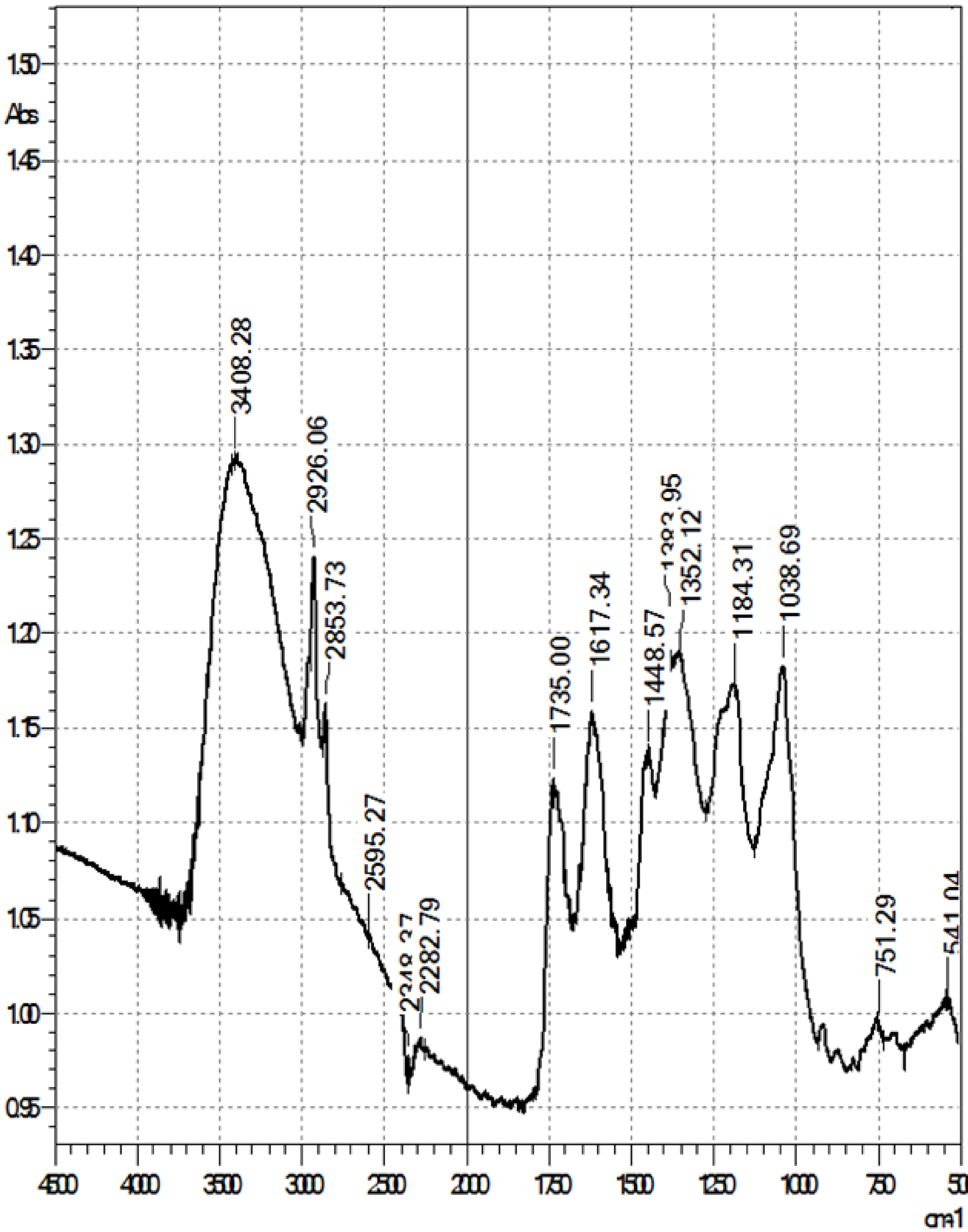


Figure 4.12: FT-IR spectrum for TL from methanol extract

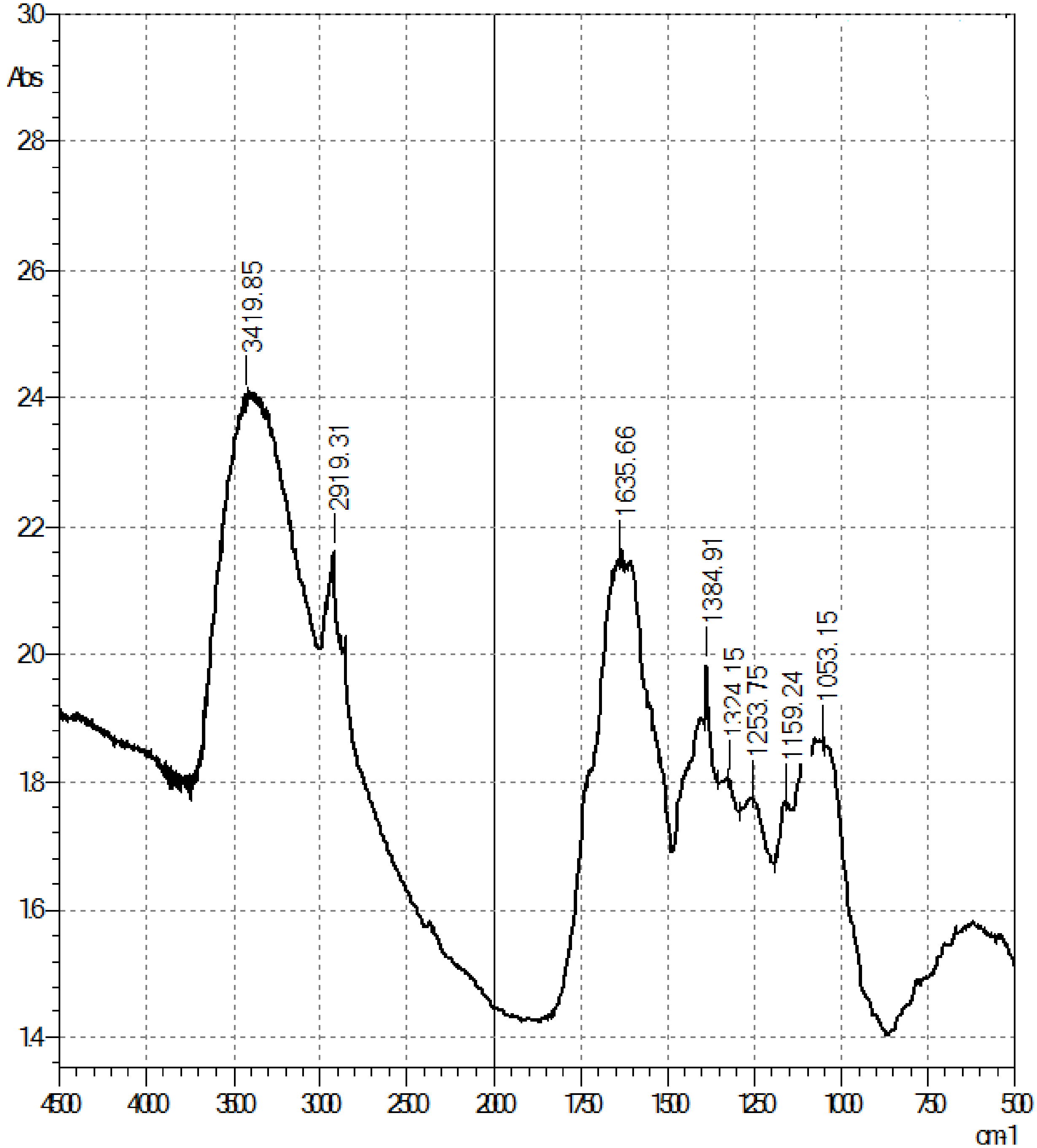


Figure 4.13 FT-IR spectrum for BL powder

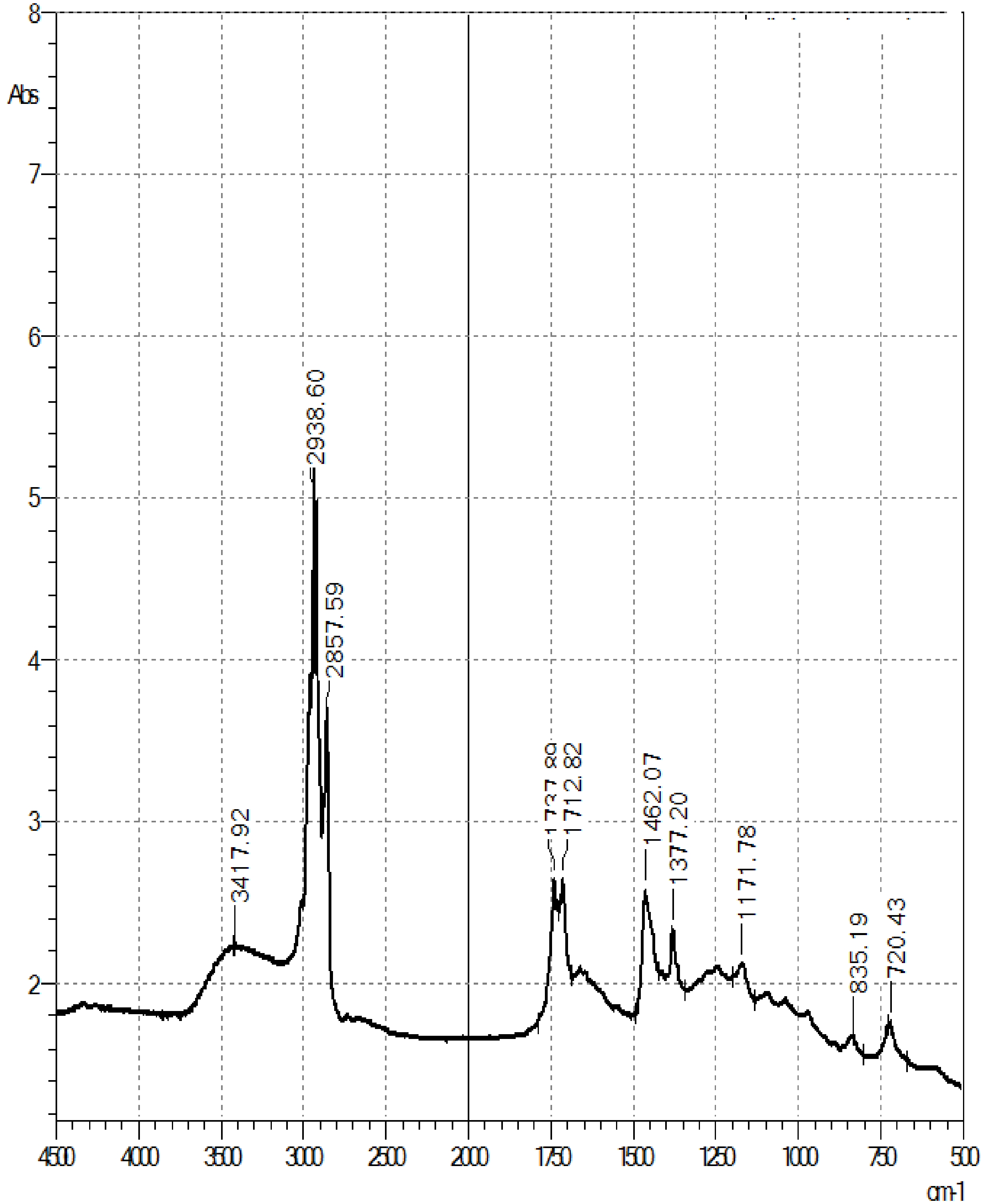


Figure 4.14 FT-IR spectrum for BL from hexane extract

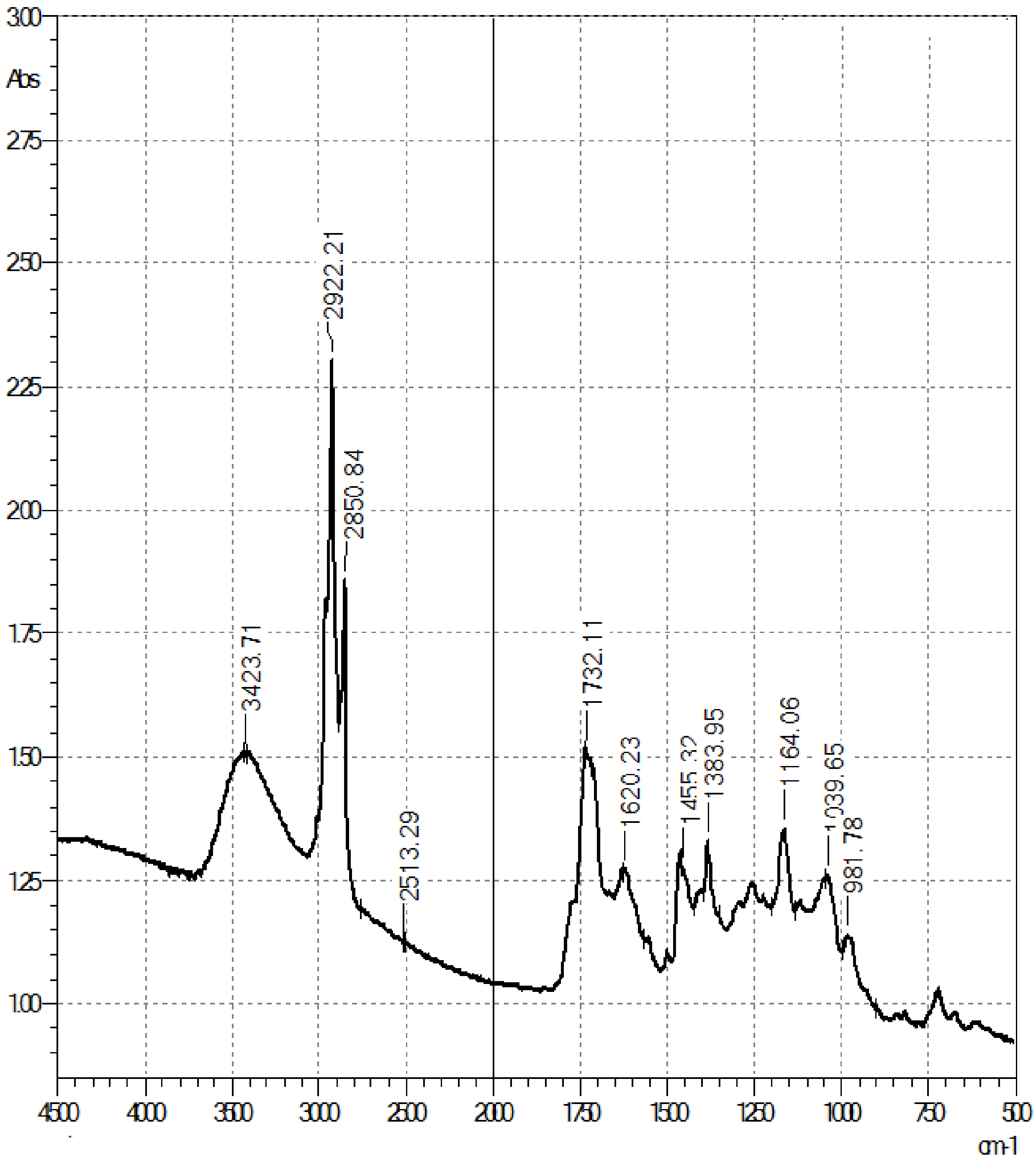


Figure 4.15 FT-IR spectrum for BL from chloroform extract

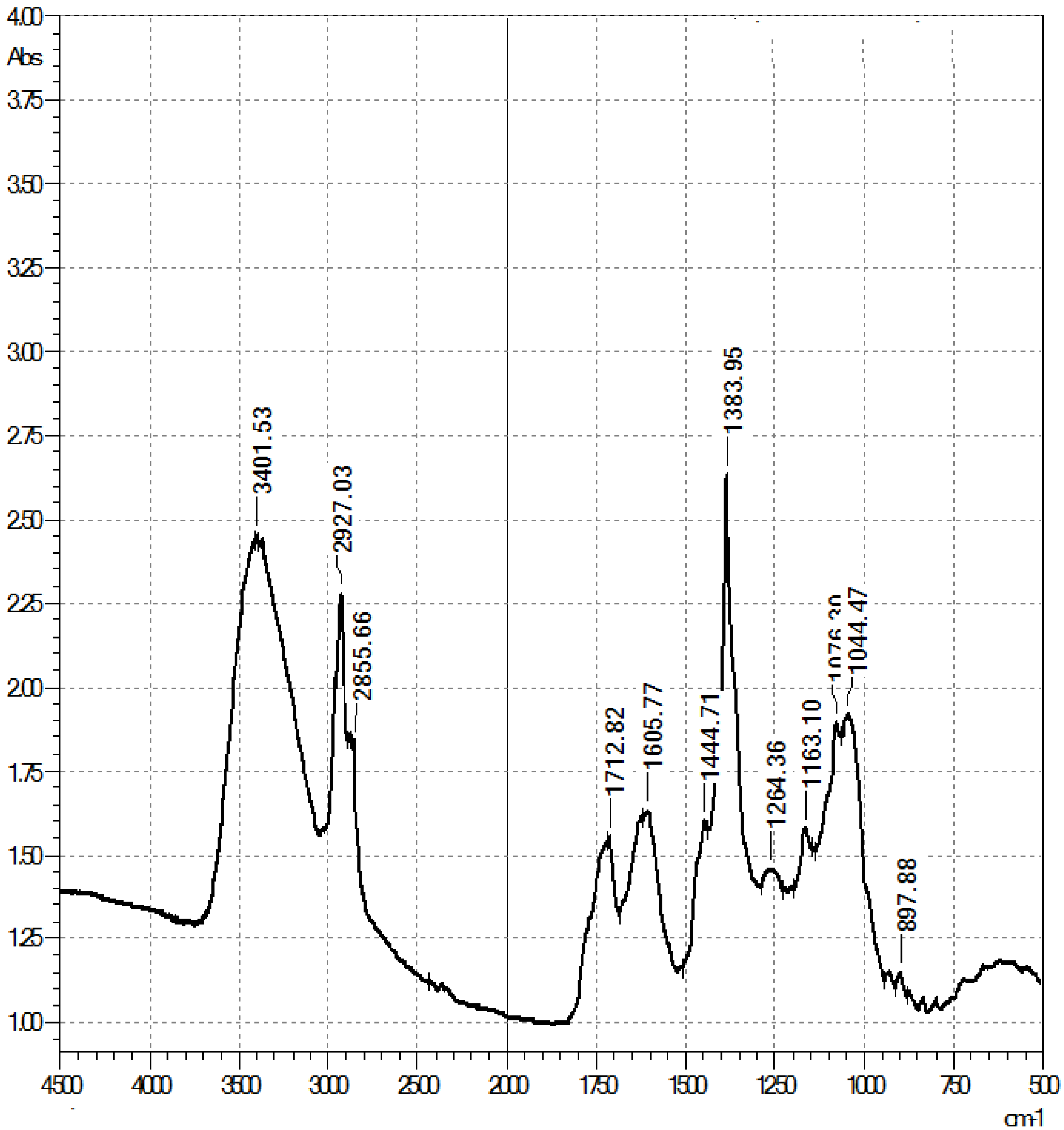


Figure 4.16 FT-IR spectrum for BL from methanol extract

4.1.4 Drilling mud properties

Properties of the drilling mud that were checked are; pH, mud weight, plastic viscosity (PV), yield point (YP), filtration loss and filter cake. The drilling mud's thermal stability was also checked after aging the samples for 16 hours using three aging temperatures (35, 65 and 93° C).

4.1.4.1 pH and mud weight

The pH results of the formulated muds are shown in Table 4.8 while mud weight is shown in Table 4.9.

Table 4.8: pH of the mud

Concentration	Mud Sample pH					
	MTL	**MML**	**PLM**	**MBL**	**C1**	**C2**
0%					10.44	
1%	**7.6**	8.6	9.12	9.28		10.05
2%	7.5	8	8.29	8.1		10.1
3%	7	7.45	7.53	7.5		10.1
4%	6.9	7.63	7.4	7.11		10.2

Table 4.9: Mud weight

Concentration	Mud sample mud weight (ppg/ft^2)					
	MTL	**MML**	**PLM**	**MBL**	**C1**	**C2**
0%					10.3	
1%	10.06	10.03	10.6	10.2		9.8
2%	10.3	10.63	10.63	10.7		10.1
3%	10.8	10.6	10.33	10.1		10.1
4%	10.3	10.6	10.6	10.2		10.2

4.1.4.2 Mud rheology

The formulated drilling mud's rheology was investigated at different temperatures to mimic the rise in temperatures as the hole depths increases. Drilling muds of different formulations: with commercially used surfactant (C2), without any of the surface-active agent (C1), mud with plant leaves were compared. Mud rheology results are presented in Tables 4.10 - 4.13.

Abbreviations used in the graphs are:

MML = Mud with *M. Oleifera* leaves, MPL= Mud with *C. papaya* leaves, MBL = Mud with *V. amygdalina* leaves and MTL = Mud with *T. mantaly* leaves.

The dial readings' values were increasing progressively from 3 rpm to 600rpm. The optimal concentration of the plant sample chosen was 2 % for a further test of mud properties i.e, thermal stability.

Table 4.10: Mud Rheology of MTL different concentrations and temperatures

	Concentration / Temperature															
RPM	1%				2%				3%				4%			
	30°C	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**
600	100	66	64	49	114	75	73	57	166	125	118	102	166	125	118	102
300	60	39	37	29	69	44	42	34	99	76	73	65	99	76	73	65
200	45	28	28	20.3	50	32	31	24	74	55	54	51	74	55	54	51
100	26	16	15	11	30	19	18	14	47	35	23	34	47	35	23	34
6	3	2	2	1	4	2	2	2	9	6	5	12	9	6	5	12
3	2	1	1	1.1	3	2	2	2	8	6	6	11	8	6	6	11
PV	40	27	26	20	45	31	31	23	67	49	45	37	67	49	45	37
YP	20	12	10	9	23	13	11	11	32	27	27	28	32	27	28	28
GEL STRENGHT																
10 sec	1.7	1.2	1.6	1	4.7	3.5	4.1	2.3	7.9	6.8	7.5	5.4	7.9	6.8	7.5	5.4
10 min	5	4.1	5.1	3.1	12	10.7	11.7	9.1	20.2	15.2	19.5	12.5	20.2	15.2	19.5	12.5

Table 4.11: Mud Rheology of MBL with different concentrations and temperatures

	Concentration / Temperature															
	1%				**2%**				**3%**				**4%**			
RPM	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**
600	126	79	73	70	138	95	94	74	235	136	204	136	337	338	367	337
300	77	48	43	43	80	57	55	42	148	88	131	88	328	236	225	294
200	58	23	33	31	61	42	41	32	115	72	101	72	260	188	179	247
100	37	22	19	23	37	26	25	20	76	54	70	54	176	131	169	182
6	6	4	3	3	6	4	4	3	17	19	21	19	49	42	74	93
3	4	3	3	4	57	3	3	3	17	20	20	20	43	40	74	89
PV	49	31	30	27	59	38	39	32	87	48	73	56	9	102	112	43
YP	28	17	13	17	21	19	16	10	61	40	57	46	319	133	113	251
GEL STRENGHT																
10 sec	3.6	3.5	3.6	1.2	7.2	6.5	8.5	2.5	12.1	8.1	11.9	8.1	20	19.3	21.2	15
10 min	6.2	5.1	6.8	4.2	14.2	13	16	11.7	25	19.6	24.3	19.6	85	72.5	84.1	60.1

Table 4.12: Mud Rheology of MML with different concentrations and temperatures

	Concentration / Temperature															
RPM	**1%**				**2%**				**3%**				**4%**			
	30°C	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**
600	102	71	74	55	169	113	119	108	233	176	207	184	275	225	207	183
300	61	41	41	33	104	72	77	69	144	110	123	112	166	133	123	112
200	45	30	30	24	79	54	57	55	107	82	95	87	125	99	96	87
100	26	18	18	14	50	35	37	37	66	50	61	56	77	62	61	56
6	3	2	2	2	9	7	8	12	12	13	12	18	14	12	13	18
3	2	1	1	1	8	6	8	11	11	9	11	17	13	12	11	17
PV	41	30	33	23	61	41	42	39	89	66	84	72	109	92	84	72
YP	21	9	10	10	37	32	35	30	55	44	44	40	57	41	39	41
GEL STRENGHT																
10 sec	1.7	0.9	2	1.1	7.5	5.8	7.9	5	14.3	9	14	6	17.2	14	15.3	12
10 min	4.5	3.7	4.4	2.1	15.8	12.9	15.3	10.5	30.2	25	31.1	20	47	40	48	30

Table 4.13: Mud Rheology of PML with different concentrations and temperatures

	Concentration / Temperature															
RPM	**1%**				**2%**				**3%**				**4%**			
	30°C	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**	**30°C**	**49°C**	**50°C**	**70°C**
600	103	78	76	55	76	48	47	36	128	84	110	63	301	185	181	137
300	61	46	45	31	43	26	26	20	74	45	65	34	181	111	110	79
200	44	33	33	23	30	18	18	14	53	33	47	25	135	80	81	58
100	26	19	19	13	15	10	10	7	29	19	28	14	80	47	47	35
6	3	2	2	1	1	1	1	1	3	2	3	2	10	6	6	5
3	2	1.2	1	1	1	1	1	1	2	1	2	1	7	5	4.3	4
PV	42	32	31	24	33	22	21	16	54	38	44	29	120	74	71	58
YP	19	14	13	7	10	6	5	4	20	13	21	5	61	37	39	21
GEL																
STRENGHT																
10 sec	2.6	2	2	0.6	6	6	4	3	5.6	4	8.4	1.6	8.4	5.7	6.5	4.5
10 min	4.5	4.6	4.2	4.7	9	8	8	6	7.3	6.3	12	2.4	13.8	11.8	12	9

4.1.4.3 Filtration loss and the filter cake

Figure 4.13 shows the temperature effect and concentration on filtration loss of the drilling mud.

Figure 4.14 shows the temperature and concentration effect on filter cake formed after the filtration loss test.

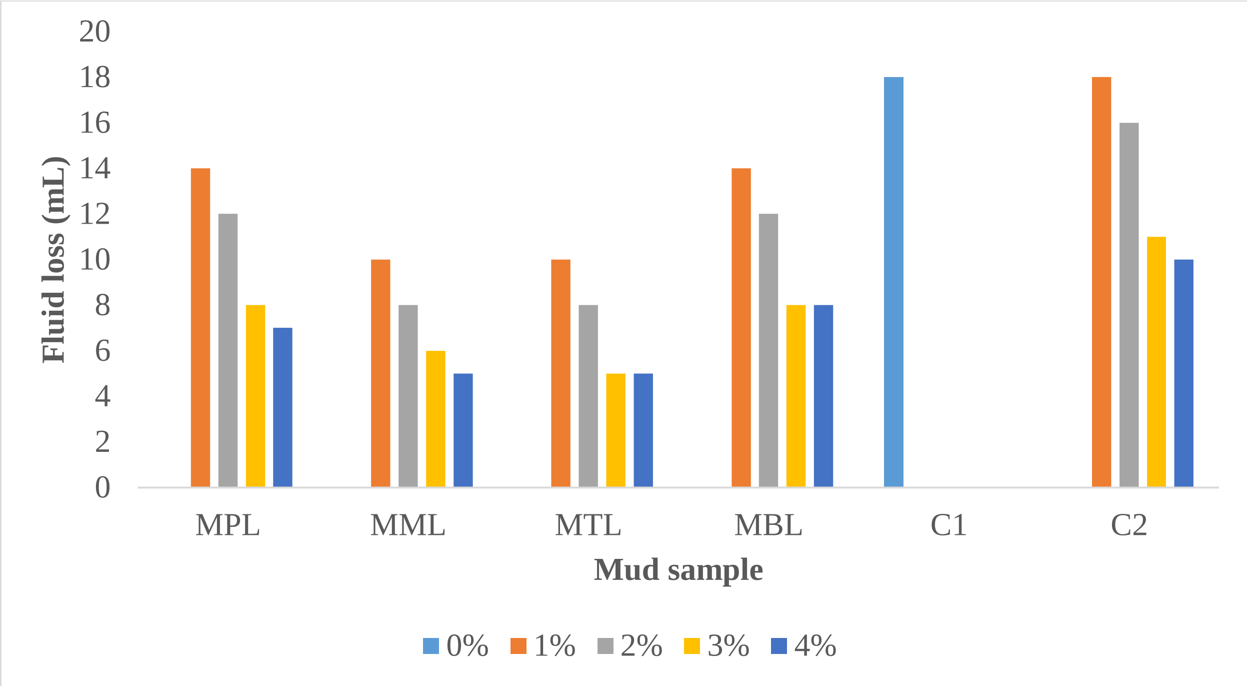


Figure 4.17: Effect of temperature and concentration on filtration loss

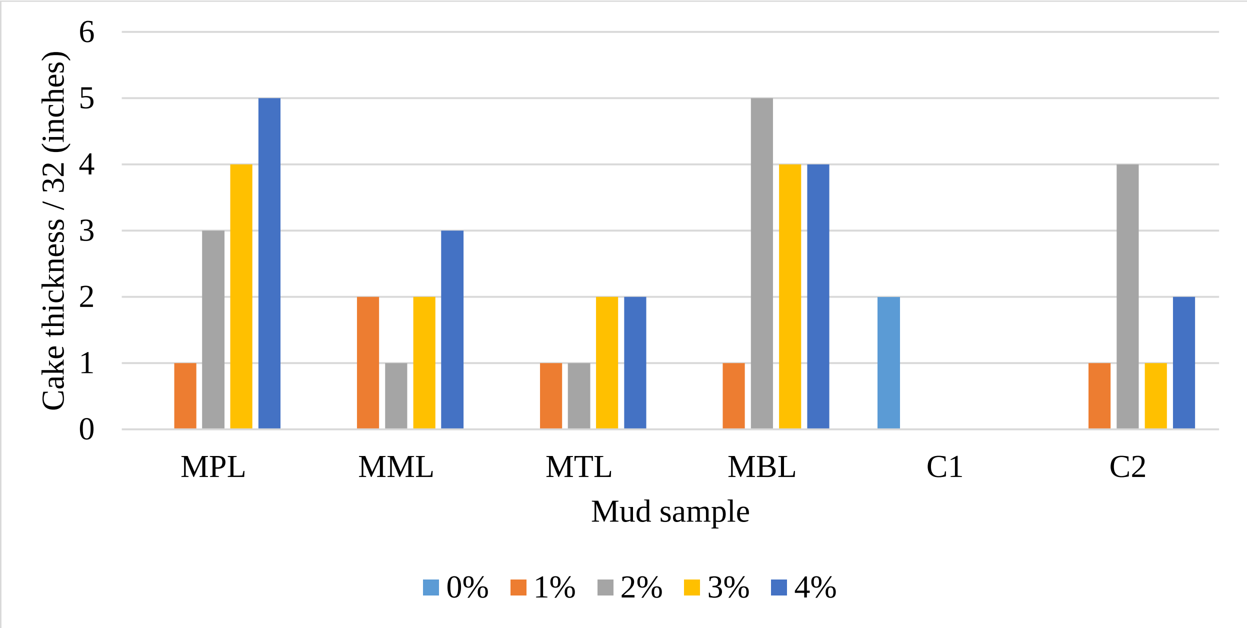


Figure 4.18: Effect of temperature and concentration on filter cake

4.1.5 Thermal stability test

4.1.5.1 pH and mud weight

Figure 4.15 and Figure 4.16 shows pH and mud weight respectively.

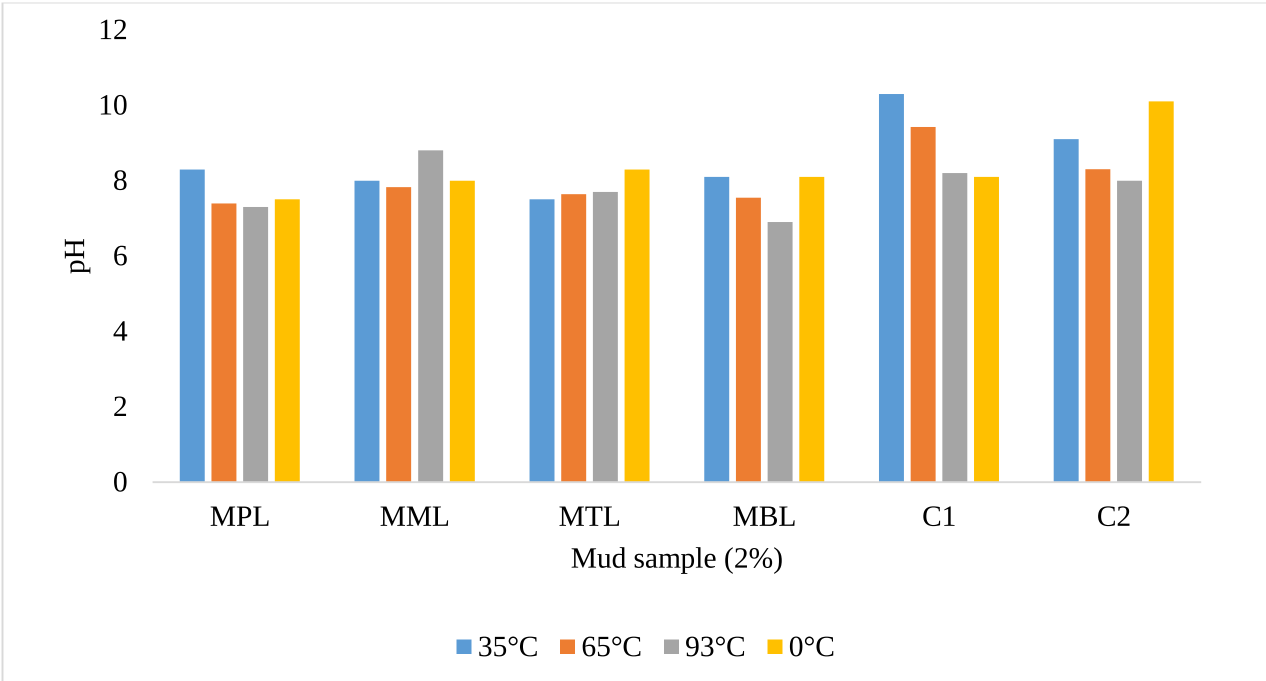
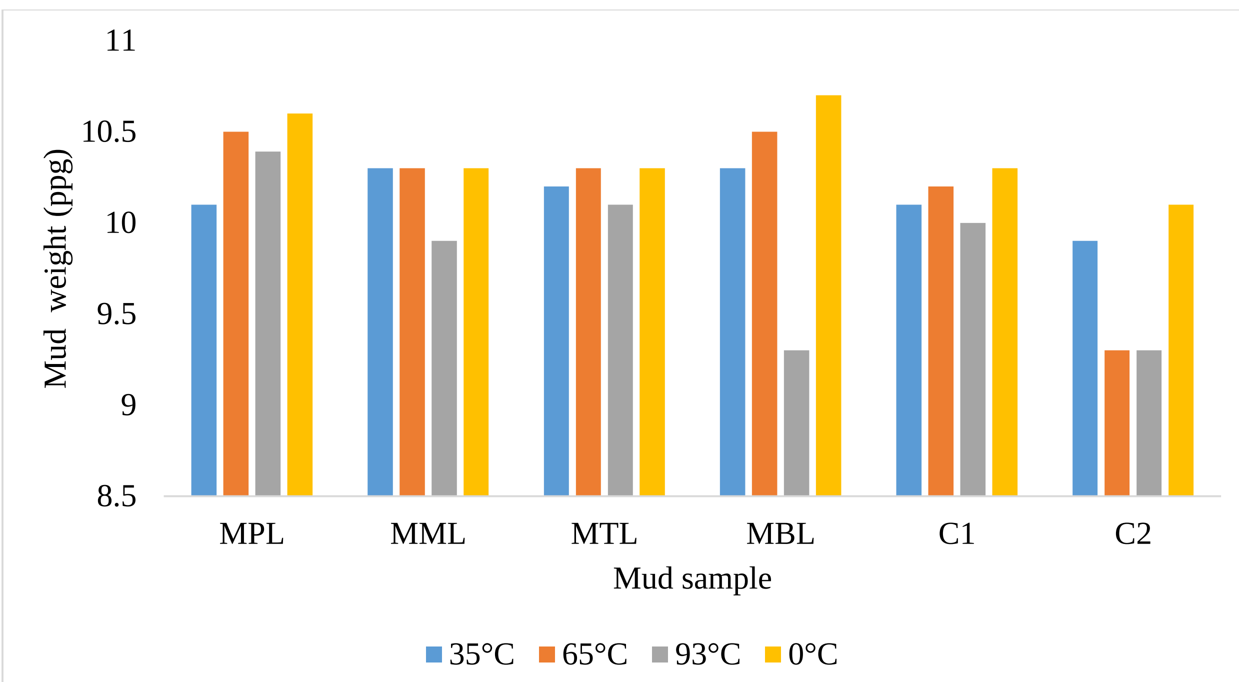


Figure 4. 19: pH of the mud after aging at different temperatures.



Key: ppg = pounds per gallon

Figure 4.20: Effect of aging on mud weight

4.1.5.2 Rheological properties after aging

Table 4.14 shows dial readings, gel strength yield point and plastic viscosity at different aging temperatures.

Table 4.14: Mud Rheology of mud samples of 2% concentration of the plant sample taken at 49°C after aging at different temperatures.

	AGING TEMPERATURES / MUD																	
	35°C						65°C						93C°					
RPM	**MTL**	**MML**	**MBL**	**MPL**	**C1**	**C2**	**MTL**	**MML**	**MBL**	**MPL**	**C1**	**C2**	**MTL**	**MML**	**MBL**	**MPL**	**C1**	**C2**
600	96	111	90	41	91	13.8	42	64	57	77	136	12.5	42	64	57	77	136	12.1
300	63	78	62	23	63	8.1	24	36	33	47	100	8	24	36	33	47	100	7.2
200	49	63	43	20	46	5.2	18	27	24	34	85	5.2	18	27	24	34	85	5
100	30	39	27	11	32	2.4	10	13	14	20	65	2.4	10	13	14	20	65	2.3
6	5	6	5	3	6	0.2	2	2	1	2	32	0.2	2	2	1	2	32	0.3
3	5	5	4	3	4	0.2	2	2	1	1	33	0.2	2	2	1	1	33	0.2
PV	33	33	28	14	28	5.8	17	28	24	30	36	4.5	18	28	24	30	36	4.9
YP	30	45	34	9	35	1.1	7	8	7	16	64	3.5	6	8	9	16	64	2.3
GEL STRENGHT																		
10 sec	4	6	4	4	4	0.1	0.8	1.6	0.6	1.3	26.8	0.1	0.8	1.6	0.6	1.3	26.8	0.1
10min	5	8	8	6	6	0.1	5	7.2	4.1	5.1	55.8	0.1	5	7.2	4.1	5.1	55.8	0.1

4.1.5.3 Filtration loss and mud cake after aging

Figures 4.16 and 4.17 shows the temperature effect on fluid loss and filter cake of the mud with 2% plant leaves.

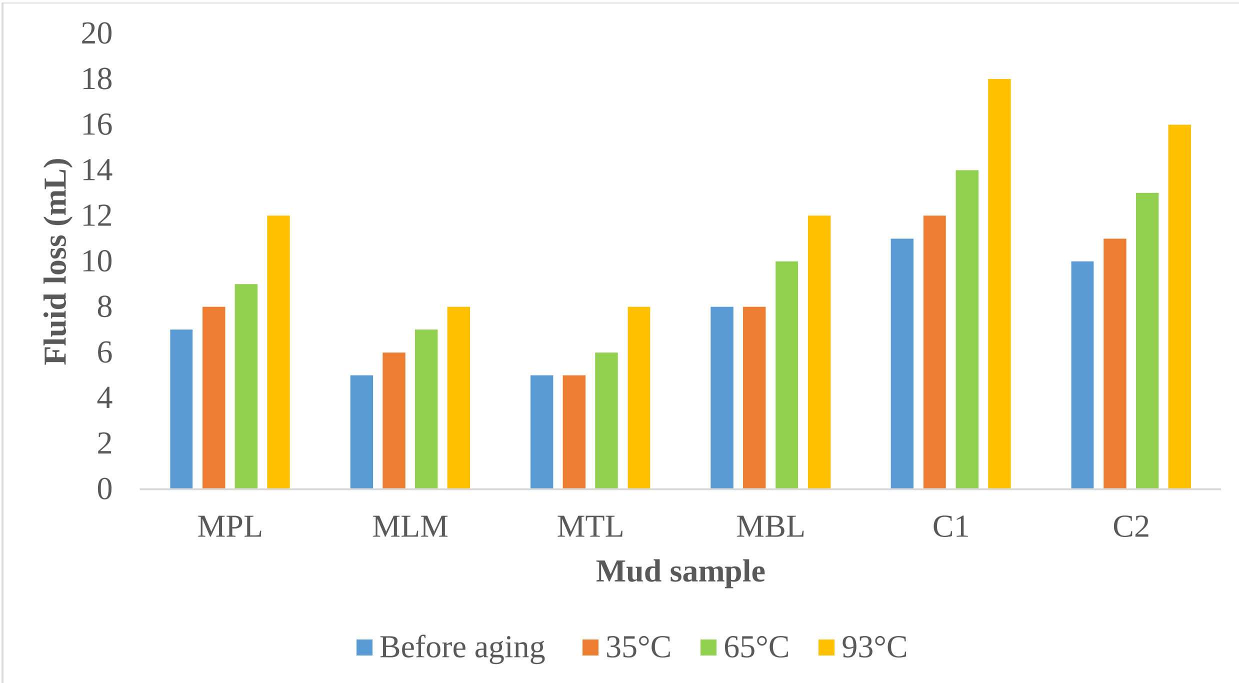


Figure 4.21: Effect of temperature on the fluid loss of the mud

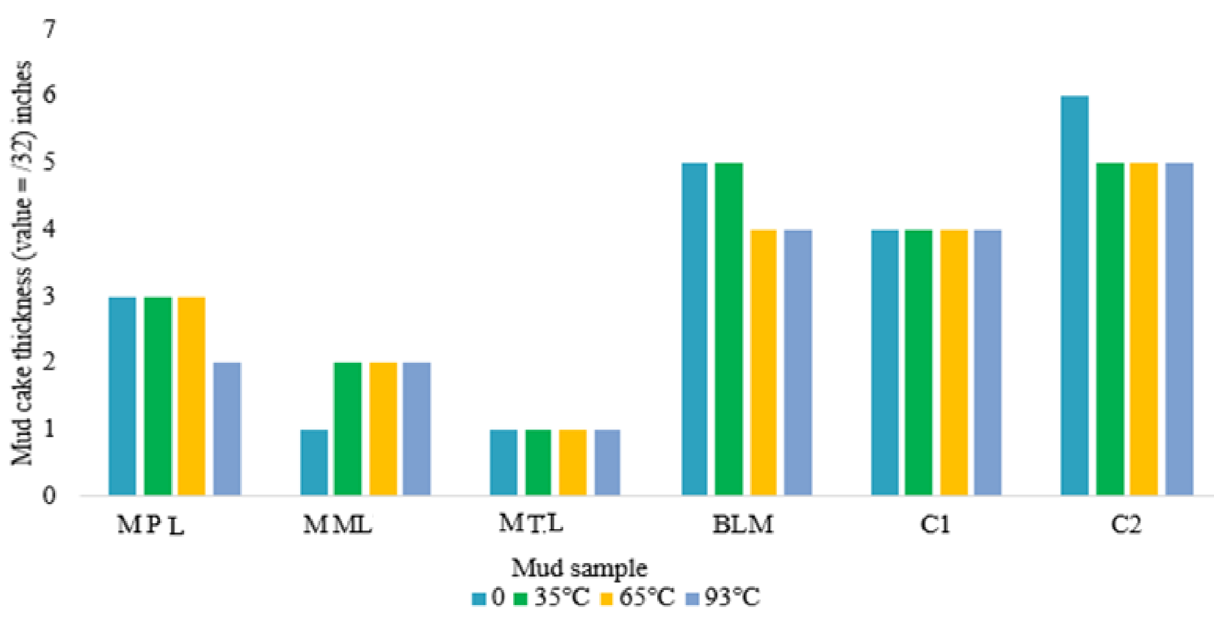


Figure 4. 22: Mud cake after aging at different temperatures

4.2. Discussion of results

4.2.1 Sample collection and preparation

Four plant leaves were collected and prepared for analysis. The plant samples were labeled PL (*Carica papaya* leaves), BL (*Veronia amygdalina* leaves), TL (*Terminalia mantaly* leaves) and ML (*Moringa oleifera* leaves).

4.2.2 Phytochemical screening

Drainage affects the foam's lifetime because when the bubbles are closer to each other, liquid's films break ultimately leading to the collapse of the foam (Rio *et al.*, 2014). Foaming of *Carica papaya* leaves (PL) and *Veronia amygdalina* leaves (BL) took the longest time by recording 89.66 and 89.33 minutes respectively. This long time taken by the foam reflects the strength of the foam which directly implies that PL and BL have a large number of foaming components which is in agreement with other studies (Huo *et al.*, 2016). BL recorded the least of 28.33 minutes and this could be because its foaming properties are not more or they get deactivated fast. The same results were observed when investigating saponins from the leaves of *Tephrosia vogelii* (A. Jain *et al.*, 2019).

The BL formed the highest foam height (56mL), followed by PL, *Terminalia mantaly* leaves (TL) and lastly *Moringa oleifera* leaves (ML). Foam height was proportional to foaming time and agreed with (Hill and Eastoe, 2017). Dispersions of gas bubbles in liquids or solids form foam. Foams occur commonly naturally and are used in commercial applications and industrial processes (Langevin, 2017). Liquid foams are important in liquid/gas contacting processes like floatation of minerals. Aqueous surfactant solutions are utilized in making foams that are used widely in cosmetic, fire-fighting and oil production companies in oil recovery (Le Merrer *et al.*, 2013) hence these plant's foams can be utilized. Qualitative tests on saponins on the crude extracts showed that all the selected plants had saponin.

4.2.3 Spectroscopic characterization

4.2.3.1 FT-IR for *Carica papaya* leaves

Figures 4.1 to 4.4 and Appendix 1 shows the FT-IR obtained from *C. papaya.* In Figure 4.1, the spectrum from PL powder gave peaks at 3422.7cm^{-1} for O-H stretch (monomeric alcohols). The small band at 2924.1cm^{-1} is for a C-H stretch and 1383.95cm^{-1} for O-H bending in alcohol. The medium and broadband at 1628.9cm^{-1} correspond to N-H bend for primary amines. The band at 1247.79cm^{-1} is for a C-N stretch for amines and at 1051cm^{-1} is for C-O stretch in alcohols. The peak at 668.35cm^{-1} corresponds to C - O in (COO) stretching for a carboxylic group.

The FTIR spectrum in Figure 4.2, shows hexane extract spectrum with band at 3399 cm^{-1} which is for O-H stretching in alcohol (Fadare *et al.*, 2015). The sharp peak at 2925.10cm^{-1} is for O-H stretching in alcohol. The small band at 2853.73 cm^{-1} is for C-H stretching in alkanes. The peak obtained at 1735 cm^{-1} indicates the presence of C=O for ketones or carbonyl group belonging to flavonoids and the broad peak at 1631.81cm^{-1} is for N-H bend in amines. The small peak at 1383.95cm^{-1} shows the presence of O-H bending for phenols. The last peak at 1076.30 cm^{-1} relates to C-O stretch in alcohol.

In Figure 4.3 the spectrum seen at 3430.5cm^{-1} is for O-H stretch in hydrogen-bonded alcohols. The peak at 2930.9cm^{-1} is for N-H stretch in amine and 2851.8cm^{-1} is for C-H alkanes. The peak obtained at 1377cm^{-1} is for O-H bending in alcohol while at 1165cm^{-1} corresponds to C-H waging for alkyl halides ($-CH_2X$). Other peaks were observed at

1629.9cm^{-1} for N-H bend in primary amines, 1086.9cm^{-1} for C-O stretch in alcohols and 836.16cm^{-1} for C-H for aromatic.

In Figure 4.4 FT-IR bands are seen between 2852 – 2929cm^{-1}, 1736.9cm- and 1384cm- for C- H stretch corresponding to alkanes. The small peak at 1736.9cm^{-1} is for C=O stretching for aldehydes. The peak at 1464.06 cm^{-1} indicates the presence of C-C stretch in-ring for aromatics. The same peaks were observed by (Prasetya *et al.*, 2018). The bands between 1066.7 and 1164.1 cm^{-1} is for C-O stretching belonging to carboxylic acid, alcohols, ethers and esters (Juárez-Rojop *et al.*, 2014).

The different functional groups detected in the extracts of *C. papaya* from different solvents indicate the presence of amino acids shown by the presence of amine groups, lipids, carotenoids, flavonoids, and saponins. The presence of steroids is indicated by wavenumbers at 1383.95 cm^{-1} and 1460.14 cm^{-1} in hexane extract, 1377.20 cm^{-1} and 1454.35 cm^{-1} in chloroform extract and lastly in peaks at 1383.95 cm^{-1} and 1460.14 cm^{-1} in methanol extract as reported in the literature (Fasya *et al.*, 2019).

The FT-IR analysis for *C. papaya* leave extract from different solvents revealed that all the extracts showed the presence of O-H, C-H, C = O, COO, N-H, and C- O (aromatics) in all the extracts and powder. C-N group was only shown in leaves' powder and not in the crude extracts which could be attributed to its insolubility in the solvents. Amines were seen in the *C. papaya* powder, hexane and chloroform extract. Methanol extracted most compounds that were in agreement with other reported literature (Khaw *et al.*, 2019).

4.2.3.2 FT-IR for *Moringa oleifera* leaves

Figures 4.5 to 4.8 and Appendix 2 shows the FT-IR obtained from *M. oleifera*. In Figure 4.5 the band seen at 3380.3cm^{-1} is for O-H stretch in alcohols, 2917.4 cm^{-1} and 2849.9 cm^{-1} are for C-H stretch in alkanes. A small peak at 1737.9cm^{-1} corresponds to C = O stretch for esters and 1384 cm^{-1} for O-H bending in phenols. The peaks at 1319.3cm^{-1}, 1243.1cm^{-1} and 1052cm^{-1} for C-O stretch in alcohols and 780.22cm^{-1} for =C-H bend for alkenes. In Figure 4.6, peaks were seen at 3399.6cm^{-1} being O-H stretch and 1377.2cm^{-1} for O-H bend in alcohol. Peaks at 1098.5cm^{-1}, 1164.1cm^{-1} and 1244cm^{-1} is for C=O stretch in alcohols. Other peaks were observed at 2922.2cm^{-1}, 2853.7cm$^{-1,}$ and 1453.4cm^{-1} for C-H stretch in alkanes. Another peak was seen at 1711.9cm^{-1} for C=O stretch due to the presence of carboxylic acid. For Figure 4.7, peaks were seen at 3376.45 cm^{-1} for O-H stretch in alcohols, 1242.4, 1160.2 and 1060.9 cm^{-1} for C= O stretch in alcohols. 2919.3 cm^{-1} and 2849.9 cm^{-1} for C-H stretch in alkanes. Other peaks were seen at1736.9 cm^{-1} for C=O stretch in aldehydes, 1655.9 cm^{-1} is for C=C stretching in alkenes and 1632.8 cm^{-1} for C=C stretching in cyclic alkenes and 719.46 cm^{-1} for C-H in aromatics.

4.2.3.3 FT-IR for *Terminalia mantaly* leaves

Figures 4.9 to 4.12 and Appendix 3 shows the FT-IR obtained from *T. mantaly*. In Figure 4.9, the pectrum peaks were observed at 2921.24 cm^{-1} are for C-H stretch in aromatics, 1732.11 cm^{-1} for

C = O stretch in aldehydes, 1614.45 cm^{-1} for C=C stretching in saturated ketone where 1445.67cm^{-1} for C-C stretch in in-ring aromatic. Other peaks were seen at 1383.95cm^{-1} and 1036.76cm^{-1} for C-O stretching found in alcohol. The peak at 780.22cm^{-1} is indicating the C-H bending for 1, 2, 3 tri substituted compound. In Figure 4.10, peaks seen at 3450.71cm^{-}

^{1} is for O-H which is associated with the alcohols, 2924.3cm^{-1}, 2851.8cm^{-1} and 1740.79 cm^{-1} for C-H stretch in alkanes. Peaks seen at 1458.21cm^{-1} is for C-C stretch in in-ring, 1383.95 cm^{-1} for O-H bending in phenol and 721.39 cm^{-1} for C-H rock in alkanes. In Figure 4.11, peaks were seen at 3408cm^{-1} for O-H stretch in alcohols or phenols, 2853.73cm^{-1} and 2926.06cm^{-1} for C-H stretch in alkanes, 1735 cm^{-1} for C=O stretch in esters or saturated aliphatics. Other peaks were seen at 1617.34 cm^{-1} for C=C stretching in alkenes, 1448.57 cm^{-1} for C-C stretching in in-ring and 1383.95 cm^{-1} for O-H bending in phenols. Peaks were also seen at 1352.12 cm^{-1} for C-H rocking in alkanes, 1184.31cm^{-1} for C-H waging and 1038.69 cm^{-1} for C-N stretching in aromatic amines.

Figure 4.12 shows spectrum peaks which are seen at 2960.78 cm^{-1}, 2920.28 cm^{-1}, 2920.84 cm^{-1} and 2850.84 cm^{-1} for C-H stretch in alkanes. The peaks at 1736.93 cm^{-1} correspond to C=O stretch in esters, 1452.42 cm^{-1} for C-H bend in alkanes and 1383.95 cm^{-1} for O-H bending in alcohols. *T. mantaly* peaks illustrate the presence of aromatics, alcohols, esters and ketones. The different vibrations observed might be because of the presence of proteins, terpenoids, and other phytochemicals. Such compounds with the observed functional groups have been detected in *Terminalia* species plants and have been documented (El-Rafie and Hamed, 2014).

4.2.3.4 FT-IR for *Veronia amygdalina* leaves

Figures 4.13 to 4.16 and Appendix 4 shows the FT-IR obtained from *V. amygdalina* leave extracts. In Figure 4.13, peaks were observed at 3419.85 cm^{-1} for O-H stretch in alcohol, 2919.31cm^{-1} C-H stretch in alkanes, 1635.66 cm^{-1} for N-H bend in primary amines, 1384.91 cm^{-1} for C-H bending in aldehydes, 1324.15cm^{-1} and 1253.75cm^{-1} for C-N stretch in aromatic amines. Other peaks were seen at 1159.24 cm^{-1} and 1053.15 cm^{-1} for C-O stretch for alcohol.

As for Figure 4.14, peaks were observed at 3417.92cm^{-1} for O-H stretch in alcohols, 2938.60 cm^{-1} and 2857.59 cm^{-1} and 1462.07 cm^{-1} for C-H stretch in alkanes, 1137.89 cm^{-1} for C=O stretch in esters. Other peaks seen at 1171.78 cm^{-1} correspond to C-O stretch associated with alcohols, 835.19 cm^{-1} for C-H aromatic and 720.4 cm^{-1} for C-H rocking in alkanes.

In Figure 4.15, peaks are seen at 3423.71 cm^{-1} for O-H stretch in alcohols and phenols, 2922.21 cm^{-1} and 2850.84 cm^{-1} for C-H stretch in alkanes. Other peaks were seen at 1732.11cm^{-1} for C=O stretching aldehydes, 1620.23cm^{-1} for C=C stretching in unsaturated ketones, 1455.32 cm^{-1} and 1383.95 cm^{-1} for C-H bend in alkanes. The peaks at 1164.06 cm^{-1} correspond to C-H waging in alkyl halides, 1039.65 cm^{-1} for C-O stretch in alcohols and 981.78 cm^{-1} for a =C-H bend in alkenes.

In Figure 4.16 shows peaks at 3401.53 cm^{-1} corresponding to O-H stretch in alcohols, 1076.30 cm^{-1} and 1044.47 cm^{-1} for C-O stretch in alcohols and peaks at 2927.06 cm^{-1} and 2855.66 cm^{-1} for C-H stretch in alkanes. Other peaks were seen at 1712.82 cm^{-1} for C=O stretching in carboxylic acids, 1605.77 cm^{-1} for N-H bend in primary amines, and 1444.71cm^{-1} for C-C stretching in-ring aromatics. Peaks observed at 1383.95 cm^{-1} correspond to aldehydes, 1264.36 cm^{-1} for C-O stretch in aromatic esters and at 1163.10 cm^{-1} for C-O stretch in aromatic amines.

Spectrum from *V. amygdalina* leaf powder, hexane, chloroform and methanol extracts shows major functional groups found in *V. amygdalina* leaves. Many peaks were seen between 1750 and 1000cm^{-1} which indicates the chemical composition associated with carbohydrates, lipids, secondary structures of proteins and phenolic compounds. The same observation was also reported in other literatures (Alara *et al.*, 2019). It is also observed that there are proteins, lipids, esters, and aromatic groups. Methanol extract had a higher extracting ability of the bioactive compounds from *V. amygdalina* leaves. Previous studies using the High-Performance Liquid Chromatography revealed that squalene, phytol, ethyl-2 –O- benzyl-d-arabinofuranoside and 9,12,15 octadecatrienoic acid and hexadecanoic acid ethyl ester were present in *V. amygdalina* leaves (Oladunmoye *et al.*, 2019).

4.2.4 pH

The pH of the leave extracts from the selected plants was determined and the results showed that all plant leave extracts were acidic. *T. mantaly* extract (TL) recorded the lowest pH of 4.61 while BL recorded the highest pH of 6.57. It is therefore expected that the plant leaves as mud additive might affect the overall drilling mud's pH by lowering its pH. At a pH of 7, the substance is neutral and above 7 means it is alkaline and below 7 it is acidic (Zhang *et al.*, 2016).

the pH of mud additives is very important because any slight change in pH can lead to several reactions or can stop reactions hence leading to the low activity of the drilling mud (Gholami *et al.*, 2018). Effectiveness of drilling mud additives is pH-dependent because it affects the interactions of clay, the solubility of contaminants and various components in the mud (Stuckman *et al.*, 2019) which makes pH parameter very important.

4.2.5 Drilling mud properties

4.2.5.1 pH of the drilling mud

In drilling mud, the main chemicals that are used in monitoring or controlling the alkalinity are carbonate ions, bicarbonate ions, and hydroxyl ions. In Appendix 5, control mud with the presence and absence of commercially available surfactant (C2 and C1 respectively) had higher values of pH. For the test muds, mud with *V. amygdalina* (MBL) recorded the highest pH of 9.28 at a concentration of 1%. The lowest pH of 6.9 was given by MTL at 4% concentration. It was also seen that as the concentration of the plant's leave increases, the pH decreased. This could be because the pH of the powdered plants' sample was lower and so contributes to the low pH of the formulated mud. Also, mud additives contribute to the overall pH of the drilling mud. In turn, acidic mud affects rheological properties by increasing YP, PV and gel strengths (Abduo *et al.*, 2016).

4.2.5.2 Rheological properties of the drilling mud

The obtained results from the mud rheology are grouped into three main categories:

(i) temperature effect

(ii) concentration effect and

(iii) plant extract effect, on the drilling mud properties.

Table 4.14 shows the recommended values of the rheological properties of the mud by API.

Table 4.14: API recommended values for plastic viscosity (PV), yield point (YP) and gel strength

Property of the mud	Recommended value
Plastic Viscosity (Cp)	8- 35
Yield point (1b/100ft^2)	Minimum= 5
	Maximum= $YP \leq 3 \times PV$
10sec gel strength (1b/100ft^2)	2 - 5
10 Minute gel strength (1b/100ft^2)	2 - 35

(Amosa *et al.*, 2010)

4.2.5.2.1 Plastic viscosity (PV)

As shown Appendix 6 - 9, the temperature effect on PV of the drilling mud formulated from different plant materials and drilling mud with the commercially available surface-active agent and without any surface-active agent. It was seen that PV values decreased as the temperature was increasing from 30 -70°C. This observation might be due to the degradation of mud components (Avci and Mert, 2019). All the PV at 30°C of the mud increased with the rise in the concentration of plant material. The observed increase reflects the difference in the number of dissolved solids in the mud sample which alters the viscosity of the water. At 2% concentration, the MPL mud sample recorded the lowest PV of 33cP among the drilling mud with plant samples at 50°C for 1% plant concentration. PV for the drilling mud with 1% of the plant's leaves at 30°C was all above the permitted values excluding that of the control sample with the commercially available surface-active agent (C1). The other PV values at temperatures of 49-70°C were within permissible values (8-35Cp) for mud with a 1% plant sample.

Mud samples with a 2% plant sample had average plastic viscosities because most of the values were between 8-35cP except for the mud tested at 30°C which was high. The control mud sample was capable of maintaining the recommended PV values at all testing temperatures at 2%. At a concentration of 3 %, the PV values were above the recommended values except that of MPL at 70°C and C1 mud at 50°C. At 1-3% plant samples, MPL mud had lower PV values matched to other mud samples with corresponding temperatures. At 4% plant sample, MPL had the highest PV of 120cP at 30°C while MTL mud recorded the lowest PV of 38cP. It was noted that at 4% plant sample, PV was higher than recommended. This suggests that if 3 and 4% plant sample is added to the mud, it can lead to well problems due to high PV that it will be generated (Ma *et al.*, 2019). High plastic viscosities can result to increase in the surge (when bottom hole pressure increases because of high speed of drill string in the hole and swab (when bottom hole pressures reduce below formation pressure) pressures and can decrease the rate of penetration leading to differential of pipe sticking (Akpan *et al.*, 2019).

It was observed in Appendix 9 that plastic viscosities of the drilling mud increased as the concentration of plant material increased. The same tendency was observed when grass was used to formulate water-based drilling mud (Hossain and Wajheeuddin, 2016) where it increased from 8-9cP. Compared with other mud samples, PV of mud with a commercially surface-active agent (C2) did not degrade more but their PV values were low (21-30cP).

4.2.5.2.2 Yield point (YP)

The yield point of formulated mud samples varied with a change in concentration and temperatures. Appendices 10 to 13 show the temperature and plant sample effect in YP of the drilling mud. It has been reported from the literature that properties of the drilling mud like YP affect the rate of penetration (ROP) considerably. The existence of unremoved solids can result to chip hold down effect which increases differential pressure which reduces ROP (Hossain and Wajheeuddin, 2016).

From Appendix 10 for (1% concentration), C2 had a higher YP of 71 1b/100ft^2 at 30°C while MPL mud had the lowest YP of 7 1b/100ft^2 at 93°C. However, all the other YP were within the recommended value at 1% concentration except that of C1 at 30°C. In Appendix 11, it shows that at a concentration of 2%, C2 had relatively high YP of 75, 75 and 73 1b/100ft^2 at temperatures of 30, 49 and 50°C respectively. At this concentration (1%), MPL gave the lowest values of YP (10, 6, 5 and 4 1b/100ft^2) at temperatures of 30, 49, 50 and 70°C respectively.

Adequate YP is required in the drilling mud to guarantee the well cleaning of the whole process (Yunita *et al.*, 2016). At 70°C, the 4 1b/100ft^2 is considered low YP according to (Ojewumi *et al.*, 2019) which can result in problems down hole like settlement of the drill cuttings and accumulation hence leading to stuck pipe (A. Amanullah, 2016). At 3% plant concentration, it can be realized from the results that MBL recorded the highest YP of 133 1b/100ft^2 at 70°C. C1 mud sample had relatively high YP at temperatures of 30, 49 and 50 °C where at 50°C is considered undesirable because it may cause high pressures during the drilling process as reported (Mao *et al.*, 2015).

At 4 % concentration (Appendix 13), MBL mud showed the highest YP of 251 1b/100ft^2 which is high. As reported in the literature, an increase in YP can be due to the effect of particles surface at a certain temperature that leads to high particle interactions and hence a higher YP (Ghosn *et al.*, 2017). Also, PV of the drilling mud can be affected by contaminants like salt in the drilling fluid (Raheem, 2018).

4.2.5.2.3 Gel strength

Appendices 16 to 23 shows comparisons of gel strengths between the formulated muds (test mud and control muds). Gel strength at 10 seconds should be between 2 and 5 1b/100ft^2 and that at 10 minutes should be between 2 and 351b/100ft^2. In Appendices 16 to 19, MPL mud gave values of 2.6, 2 and 2 1b/100ft^2 at test temperatures of 30°C, 49°C and 63°C respectively at 1% concentration. MML mud had 2 1b/100ft^2 at 50°C at 1 % concentration while the rest were lower than 2 1b/100ft^2. At 1% concentration, MBL mud gave good values of 3.6, 3.5,3.6 1b/100f^2 at 30°C, 49°C and 50°C respectively. C1 mud recorded 4, 1, 2 and 2.3 1b/100f^2 at testing temperatures. C2 mud recorded higher and undesirable values of gel strength at 10 seconds (12- 51.2 1b/100f^2) at all testing temperatures hence high pressures needed to resume drilling process leading to risks in operation. At higher concentrations (2 to 4%), all the 10 seconds gel strengths were higher except MTL mud which had accepted values of 4.7, 3.5, 4.1 and 2.3 1b/100f^2 at all testing temperatures.

From Appendices 18 to 21, at 10 minute gel strength, most values were within recommended values (2-35 1b/100f^2) in most testing temperatures. However, the MBL mud with 4% concentration had higher gel strengths of 72, 58, 84 and 60.1 1b/100ft^2 at 30, 49, 50 and 70°C respectively. C2 mud sample had gel strength of 50, 48 and 43 1b/100ft^2 at 2 % concentration which was high at 30, 49 and 63°C respectively. At 4 % concentration at a temperature of 70°C, C2 mud gave a value of 201b/100f^2 which was within the acceptable values. It is quite important to note that high gel strength leads to high pump initiation pressure which may cause formation fracture and loss in circulation. Also, low gel strengths will lead to the powerlessness of the drilling mud to suspend the drilling cuttings which is one of the main roles of drilling mud (Ranjan and Dutta, 2017).

It was observed that gel strengths increased after the addition of the plant material. The influence of plant material on the gel strengths could be accredited to hydrogen bonding of the hydroxyl group in the plants with other mud additives like bentonite clay and hydro PAC (Barry *et al.*, 2015). Gel strength depends mainly on the attractive forces and it is associated with the yield point (Salih and Bilgesu, 2017). Hydro PAC is anionic, therefore, adsorption and flocculation results due to hydrogen bonding between hydroxyl groups on the polymer and the solids' surfaces (Shaikh *et al.*, 2017).

4.2.5.2.4 Filtration loss

Filtration loss was measured at 500 psi at 93°C after 30 minutes. From Appendix 24, that there was a decrease in the fluid loss after adding the plant material (18 to 5mL). In another research, they discovered that after adding a grass sample in the drilling mud, water loss was decreased by 19% (Hossain and Wajheeuddin, 2016). Also, there was a decrease in concentrations of 3% and 4% mud sample. The same trend was also observed by (Agin *et al.*, 2019) after adding 1,6- hexamethylenediamine in the drilling mud formulations. Normally, drilling fluid contains the suspended particles and these particles flow laterally into the formation (Fink, 2015) which is filter paper in this research work. Filter paper plays a role as a sieve and captures the particles hence forming filter cake that reduces the fluid loss. Thus, the more suspended particles are present in the drilling mud, the smaller amount of the fluid loss and this could be the due to the drilling mud with plant material showed decrease in the fluid loss by 72%.

Control mud without any plant or surfactant lost a higher volume (18mL) of water through the filter paper. In water-based mud, loss of fluid or water into the formation may lead to a change that is irreversible in drilling mud properties like rheology and density (Assem *et al.*, 2019). The plant materials or most of the natural components have some properties that give them the ability to be utilized as fluid control agents when formulating drilling mud like tannins, flavonoids, and cellulose as surface-active agents (Brockman, 2016).

4.2.6 Thermal stability test

Successful drilling operation is achieved when used drilling mud is monitored and controlled continuously. Drilling mud properties at the surface environments do not symbolize bottom-hole environment because the properties change due to rising temperatures and aging time at the surface environment. This change in properties of the drilling mud might lead to sequences of downhole environment problems. The thermal stability test for drilling mud rheological properties was carried out at 49°C as this is the standard temperature according to American Petroleum Institute (Indulkar *et al.*, 2018). The mud sample tested for aging had 2% of the plant sample and commercially available surfactant. This concentration was chosen based on the results of rheological properties that were within / close to the recommended values.

4.2.6 .1 Effects of aging on yield point and plastic viscosity of the muds.

Drilling mud must be designed in a way that it should be capable of transporting the cuttings, lubricate and cool the drill bit, to control the formation pressures and maintain hole integrity (Sadeghalvaad and Sabbaghi, 2015). To achieve its goal, drilling mud must have a balance between having enough plastic viscosity and gel strength which requires a lot of effort to accomplish it (Igwe, 2016). As shown in Appendix 10, the drilling mud's PV decreased after aging at all the temperatures as it was also observed by (Yunita *et al.*, 2016). C2 mud recorded the highest decrease in PV of 84.6% after hot rolling 35°C and 93°C respectively. C1 mud kept its PV at 36cP at aging temperatures of 65°C and 93°C. The PV of C1 increased with aging temperatures which could have been caused by dehydration (Xiong *et al.*, 2019) of the mud but the value was still recommendable. It was also found out that MBL mud's PV was consistently higher than the other formulations with plant material. Drilling mud viscosity is influenced by flocculation on heating which causes the aggregation of the platelets to increase leading to a general reduction in solid volume. This behavior of solid reduction causes the clay aggregates to move freely in the aqueous phase. This lowers the internal friction and when internal friction is reduced, mud viscosity decreases eventually (El-Sukkary *et al.*, 2014) and that could be the cause for the observed changes in the mud properties. After aging at 35°C, PLM mud and MPL mud showed no significance by recording P values of 0.074 and 0.321respectively while TML and MBL showed the significance of P values of 0.023 and 0.01 respectively. After aging at 93°C, MML, MTL, and MBL showed significance where the PV dropped, but PLM was stable by having $P = 0.43$.

In Appendix 11, the yield point of the reference mud (C2) experienced a drastic decrease by 97.3% (75 – 2 1b/100ft^2). There was a 67.1 % increase in YP of the reference drilling mud (C1) after aging at 93 °C. Mud samples with plant material (MTL, MML, MBL, and MPL), the YP decreased significantly after aging at 65 °C when compared with the YP after aging at 35 °C. It is known that high temperatures increase the YP in WBM

(Elochukwu *et al.*, 2017). However, the plants' mud samples were capable of maintaining the YP after aging at 93 °C i.e, there was no significant difference at aging temperatures of 65 °C and 93 °C. The addition of plant samples in the drilling mud was stable even at high test temperatures meaning the plant leaves were efficient. It is reported in the literature that most plants have surface-active agents (S. Jain, 2015) and these agents molecules help the flowing behavior of water-based mud by successfully improving its thermal resistance (Yunita *et al.*, 2016).

4.2.6.2 Effect of aging on gel strength

Appendix 26 shows that all the gel strengths at 10 seconds increased when aged at 35°C excluding that of MPL and MBL that reduced from 6 to 4 1bl/100ft^2 and 8 to 41bl/100ft^2 respectively. After aging at 65°C and 93°C, at 10 seconds gel strength, none mud samples with plant leaves recorded gel strength within values that are recommended (2 to 51bl/100ft^2). The plant mud samples recorded gel strength values of 0.8, 1.6, 0.6 and 1.31bl/100ft^2 for MTL, MML, MBL, MPL respectively. At temperatures of 65 and 93°C, the C1 mud sample recorded the highest gel strength (26.8 bl/100ft^2) which is undesirable. It was observed that 10 minutes of gel strength of the drilling mud samples reduced with increasing aging temperatures except for control (C1) sample which increased from 5.3 to 55.8 1bl/100ft^2 after aging at 93°C. For the C1 mud, there was the same trend for 10seconds and 10minutes gel strengths i.e. increase in gel strength with the rise in aging temperatures. It is important to note that high gel strengths are not desirable because it will require high pressures to be able to manage the gel strength and to resume the circulation which might fracture the formation during drilling processes (Li *et al.*, 2016). Gel's strengths rely on colloidal clays concentrations in the mud and this dependence also applies to YP of the mud (Igwe, 2016).

4.2.6.3 Effect of aging on fluid loss and mud cake

Fluid loss test was done to check if the drilling mud could be able to inhibit the loss of fluid into the formation and making a light filter cake which is thin and less permeable. In Figure 21, the fluid loss was increasing with aging temperatures. MML and MTL mud recorded the lower amount of filtrate loss at corresponding aging temperatures. Control mud (C1) had the highest fluid loss of 18 mL when aged at 93°C while C2 mud lost 16 mL at the same temperature. MBL and PLM muds recorded the highest amount of filtrate loss (12mL) at 93°C when compared to other mud with plant leaves while TML had the lowest volume (5mL) of fluid lost after aging at 35°C. The thinner filter cake is produced by the low permeability of mud cake which results in a lower amount of filtrate loss (Muhsan *et al.*, 2017).

There was a significant difference in the filtration after aging and between control muds (C1 and C2) and mud with plant leaves (test muds) where test mud showed improved filtration loss prevention more than the control mud. The influence of plant leaves in the drilling mud could have affected the thickness and arrangement of the mud cake. Bentonite clay and polymers are unable to keep the honeycomb pattern of the filter cake due to electrolyte contamination. Sodium and potassium ion can be affected by considerable chemical stress which results in to increase in the size of the pores, therefore, allowing fluids to flow into the formation (Mahmoud *et al.*, 2018).

In Figure 22 results of the filter cake formed during the filtration loss test is shown. C2 mud sample recorded the thickest mud cake (5/32 inch) at all aging temperatures. MBL had the same mud cake thickness as C2 after aging at 35°C and reduced after aging at 65 and 93°C (4/32 inches"). Thinnest mud of 1/32" inches cake was recorded by the MTL mud at all aging temperatures. MML had the same thickness of 1/32 inches" before aging. Mud cake is made on the formation through the fluid loss process. The penetrability of the cake is influenced by the particle size and distribution. An increase in particle size results in a decrease in permeability due to the tightly-packed particles (Xu *et al.*, 2018).

Comparing the fluid loss volume and mud cake formed thickness, it was seen that C2 mud lost a high amount of the fluid and had the thickest mud cake because its cake was too soft and could permit more fluid to pass through. A good mud formulation should maintain the fluid loss to the formation as low as possible and equally, it should not form a thick filter cake. MML had the least fluid loss and smaller filter cake (1/32 inch) which can be caused by less permeability to prevent loss of fluid and should resist high differential overbalance pressure (Bageri *et al.*, 2019). The change in fluid loss volume could be accredited to the clay properties of the filter cake which has control over the physical properties of the filter cakes (Farahbod and Farahmand, 2018). The test mud had different plant materials that tested positive towards saponins. The saponins are surface-active agents with different structures of long molecule chains which could have made the fluid to be more viscous and formed tighter filter cake. Addition of surfactant in the water based drilling mud reduces the fluid loss significantly (Ozkan *et al.*, 2018; Yunita *et al.*, 2016).

CHAPTER FIVE

SUMMARY, CONCLUSION, AND RECOMMENDATIONS

5.1 Summary

In this research work, six different water-based mud were formulated were two of them acted as control mud (C1 and C2) and four of which were formulated using plant leaves (*Carica papaya*-MPL, *Terminalia mantaly* - MTL, *Vernonia amygdalina* - MBL and *Moringa oleifera*-MML). Physicochemical properties of the mud revealed that the plant leaves (*Carica papaya*- PL, *Vernonia amygdalina*- BL, *Terminalia mantaly*- TL and *Moringa oleifera* -ML) had foaming properties where BL had the highest length of the foam and both BL and PL took the longest time for the foam to vanish. The presence of saponins was examined in the plant extracts from different solvents which revealed that all the leaves had saponins. The plant leaves had varied pH with TL having the lowest pH while BL had the highest.

FTIR spectral analysis allowed the identification of the functional groups present in the plant extracts from different solvents. The FTIR results indicated that all the plant extracts had several compounds displayed by various functional groups. Among the functional groups identified in the extracts were; esters, aldehydes, alcohols, phenols, aromatics, amines, carboxylic acids, nitro compounds, and alkanes.

Plastic viscosity of all the mud formulated (with plant leaves and commercially available surfactants) was increasing with concentration but decreased with an increase in temperature were mud with plant leaves had more stability towards temperature. At lower concentrations of plant leaves and commercial surfactants, the YP values were lower compared with the mud without plant leaves or surfactants. MBL mud had higher yield points compared to other mud formulations. The control mud with commercially available surfactant gave lower values of yield points at all testing temperatures and concentrations. The 10 seconds gel strength of the formulated drilling mud remained within the acceptable values at the lower concentration for muds with plant leaves. The control mud (C2) had too high values of gel strength (26 1b/100ft^2) at 10seconds which is undesirable after aging at 93°C. The gel strengths after 10 minutes showed that C2 mud could pose problems in drilling processes hence not good formulation because it registered too high values of gel strengths. MML had relatively high gel strengths when compared to the other muds with plant leaves.

Fluid loss test revealed that the plant leaves played a part in preventing the loss of the fluid where MML and MTL had more control over fluid loss hence the plant leaves are good additives as fluid loss control. C1 and C2 recorded a high amount of fluid loss which might lead to sticking of the pipe in the drilling process. Thermal stability of the formulated muds indicated that the mud with plant leaves was thermally stable up to the temperature of 93°C unlike the drilling mud with commercially available surfactant on the PV values. The yield point of the formulated mud with plant leaves decreased with aging temperatures but showed good values after aging at 35°C. MPL mud was affected but could still show higher values of YP than the other formulated muds with plant leaves. The gel strength of C1 mud was highly dehydrated after aging because it gave high gel strength both at 10 seconds and at 10 minutes. Mud with plant leaves showed flat gel strength at all aging temperatures and is a good property during the drilling process since it will not lead to differential pressures down the hole. Fluid loss test showed that after aging, the mud components were degraded causing a surge in the fluid loss. Nevertheless, the mud with plant leaves showed that it could withstand the testing temperatures because they showed a lower value of fluid loss when compared to the control muds.

5.2 Conclusion

This research work has successfully formulated and characterized mud formulations with four

different additives from the selected plants (*Carica papaya, Moringa oleifera, Terminalia mantaly and Veronia amygdalina*). From this work, the results obtained showed that:

1. All the plant leaves improved rheological properties of the formulated muds in comparison with the mud with commercially available surfactants. The MML (mud with *Moringa oleifera* leaves) showed better mud properties than the other muds formulated with the plant leaves.
2. Formulated mud with plant leaves reduced the filtration loss by 44.4 %.
3. Mud with plant leaves was thermally stable where they had higher values by PV - 79%, YP- 96%, Gels strength 10seconds and 10 minutes was 97% and 51 % respective after aging at high temperatures (93°C).
4. The FT-IR characterization showed that the plant leaves had various components and the phytochemical screening indicated the presence of saponins which could have led to observed effect in the drilling mud properties.

Following the performance of mud with plant leaves (MML, MTL, MPL, and MBL) that met most of the parameters and even exceeded the performance of the control mud (with commercially available surfactant) suggests that the plant leaves should be considered as an additive of water-based mud.

5.3 Recommendations

It is hereby recommended that further research work be carried out on the;

1. toxicity test of the formulated mud with plant leaves additives to determine if they meet the API standards.
2. characterization of the active components in the selected plants.

5.4 Contributions to knowledge

1. Data on drilling mud properties with selected plant leaves ***(****Carica papaya Moringa oleifera*, *Terminalia mantaly*, *Veronia amygdalina*) as mud additives that have shown better properties over the commercially available surfactant has been documented.
2. The viability of these extracts in the drilling process can be used in place of commercially available surfactant since it is eco-friendly.

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APPENDICES

Appendix 1: Infrared spectral results of the crude extracts of the PL sample from different solvents.

	FT-IR (Wavenumber cm-1)										
No extraction	3422.7	2924.1	1628.9	1384	1321.3	1248	1051.2	781.2	668.35	520.79	
Hexane extract	3399.6	2925.1	2853.7	1736	1631.8	1384	1163.1	1076			
Chloroform extract	3430.5	2930.9	2851.8	1737.9	1629.9	1551.8	1454.4	1377	1166	1086.9	836.16
Methanol extract	2929	2852.8	1736.9	1460.1	1384	1164.1	1066.7				

Appendix 2: Infrared spectral results of the crude extracts of the ML sample from different solvents.

Extract	FT-IR (Wave numbers cm^{-1})											
No extraction	3380.3	2917.4	2849.9	1737.9	1384.9	1319.3	1243.1	1052	780.22			
Hexane extract	3399.6	2922.2	2853.7	1737.9	1711.9	1453.4	1377.2	1244	1164.1	1098.5		
Chloroform extract	337.45	2919.3	2849.9	1736.9	1655.9	1632.8	1452.4	1384	1242.4	1160.2	1060.9	719.

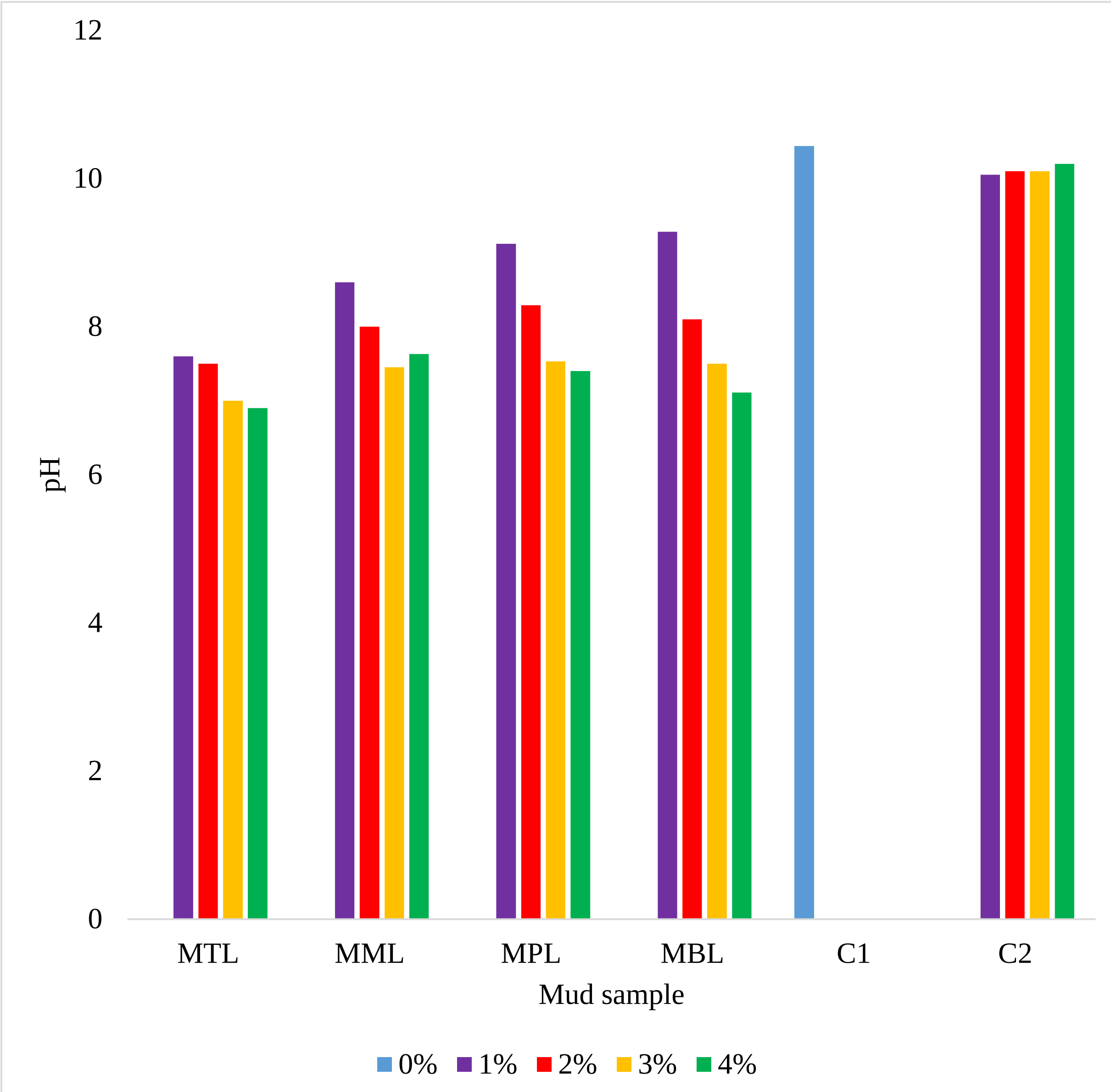
Appendix 3: Infrared spectral results of the crude extracts of the TL sample from different solvents.

Extract	FT-IR (Wave numbers cm^{-1})											
No extraction	2921.2	2850.8	1732.1	1614.5	1445.7	1384	1445.7	1384	1317.4	1036.8	780.22	
Hexane extract	3450.7	2924.1	2851.8	1740.8	1458.2	1384						
Chloroform extract	2960.8	2920.3	2850.8	1736.9	1452.4							
Methanol extract	3408.3	2926.1	2853.7	1735	1617.3	1448.6	1384	1352	1184.3	1038.7	751.29	541.04

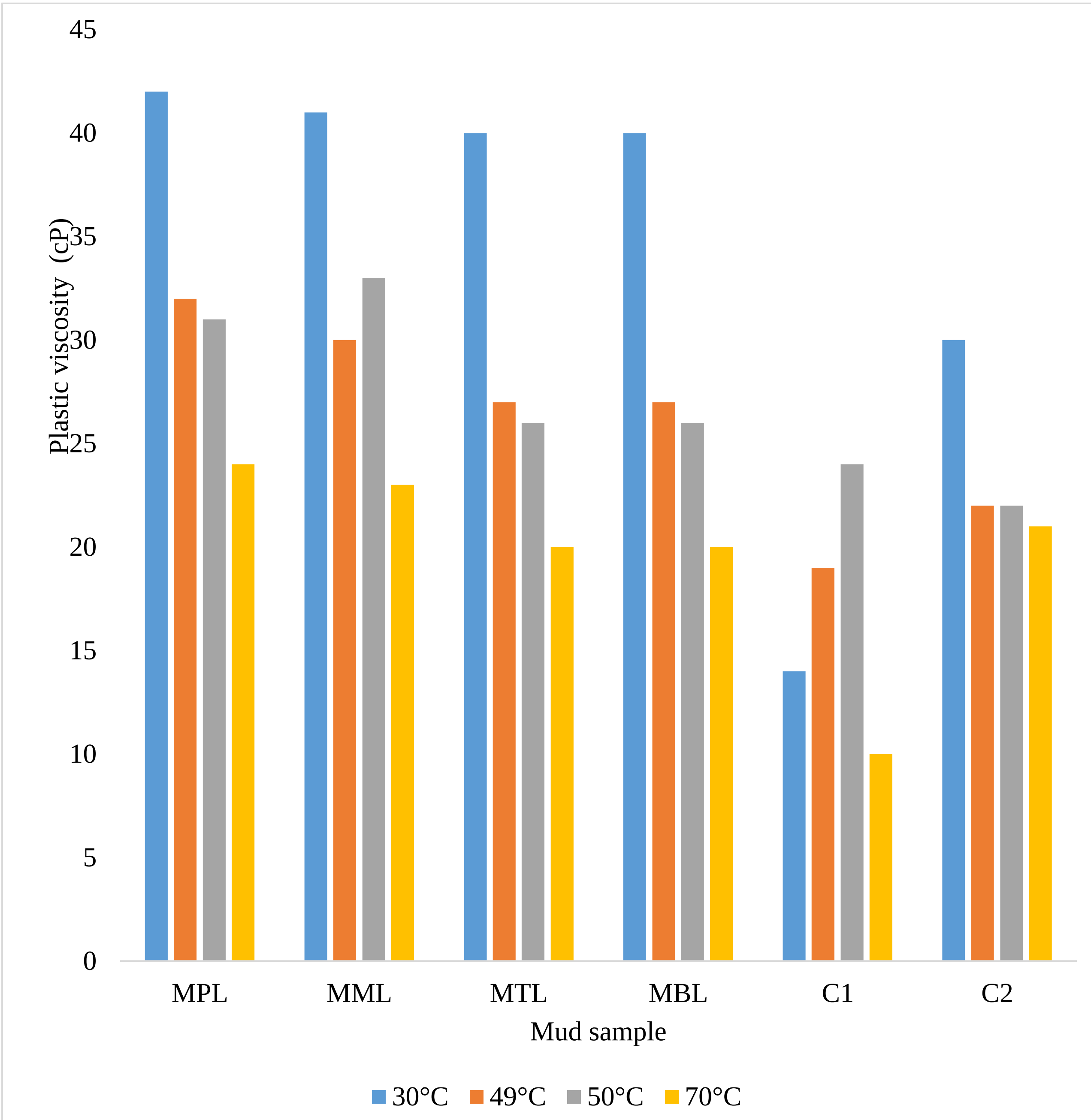
Appendix 4: Infrared spectral results of the crude extracts of the BL sample from different solvents.

Extract	FT-IR (Wave numbers cm^{-1})											
No extraction	3419.9	2919.3	1635.7	1384.9	1324.2	1253.8	1159.2	1053				
Hexane extract	3419.9	2919.3	1635.7	1384.9	1324.2	1253.8	1159.2	1053				
Chloroform extract	3423.7	2922.2	2850.8	2513.3	1732.1	1732.1	1455.3	1164	1039.7	981.78		
Methanol extract	3401.5	2927	2855.7	1712.8	1605.8	1444.7	1350	1264	1163.1	1076.3	1044.5	89[illegible]

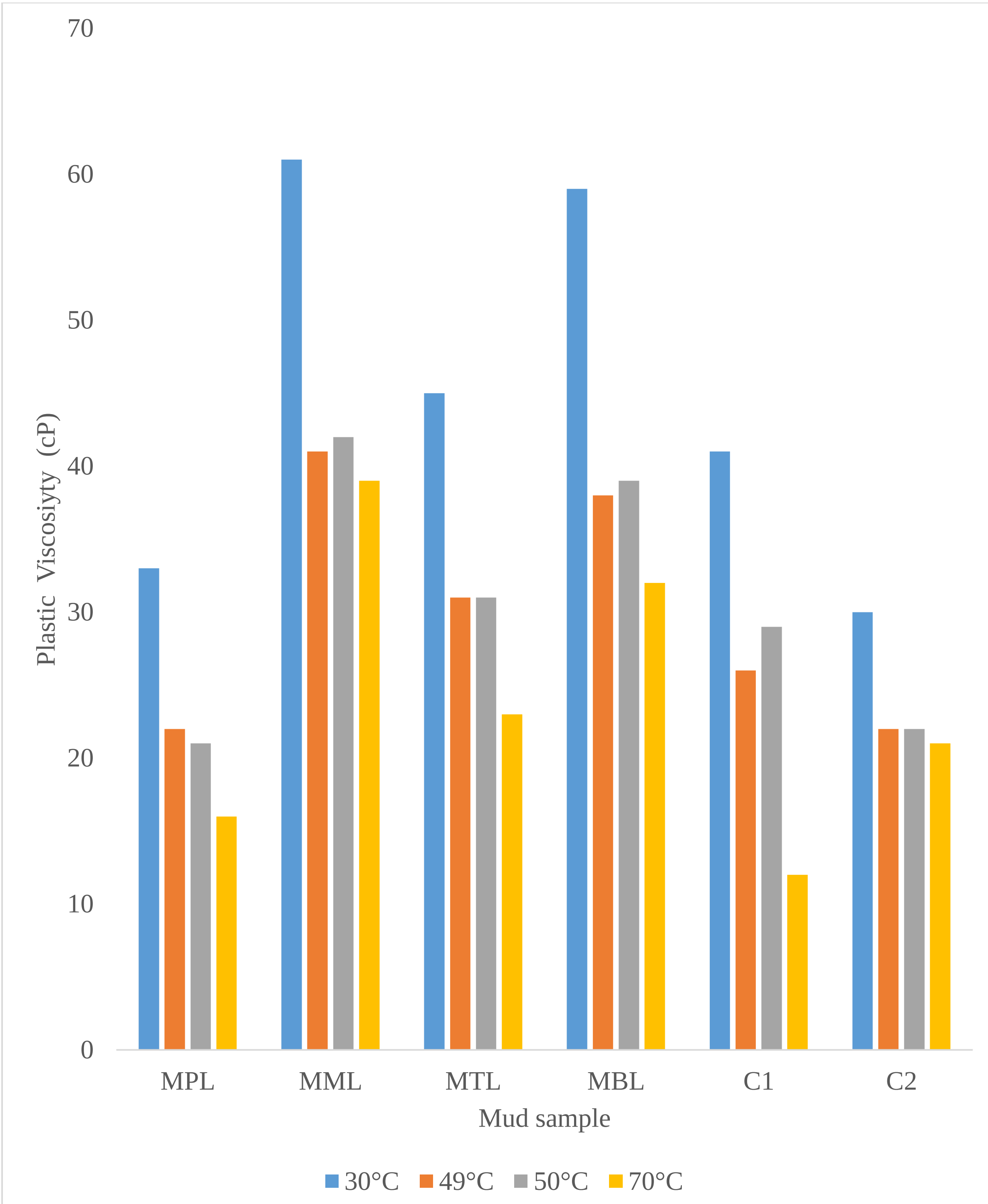
Appendix 5: pH of the formulated mud



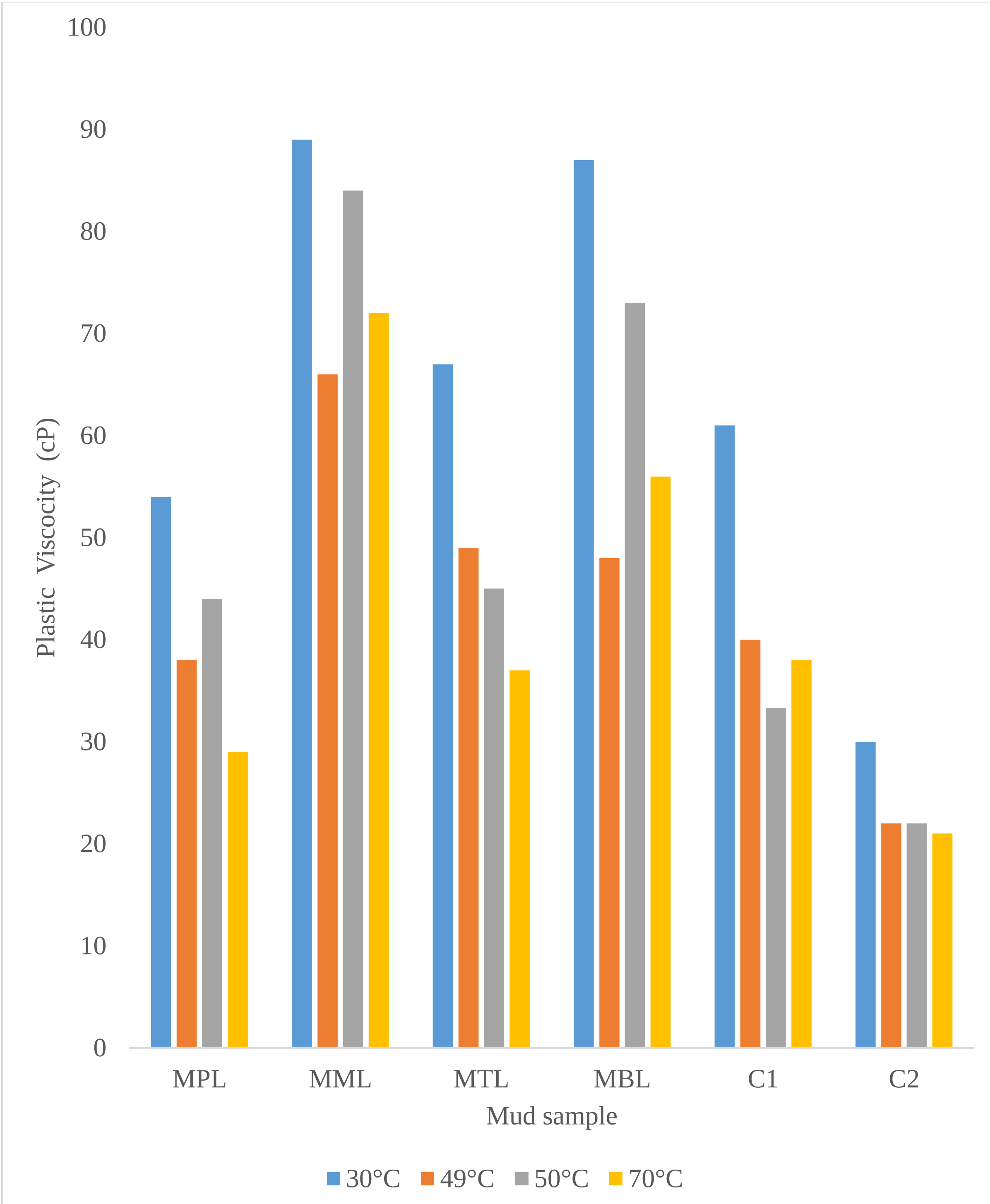
Appendix 6: Effect of temperature on 1% plant sample on PV of the drilling mud from different plant material



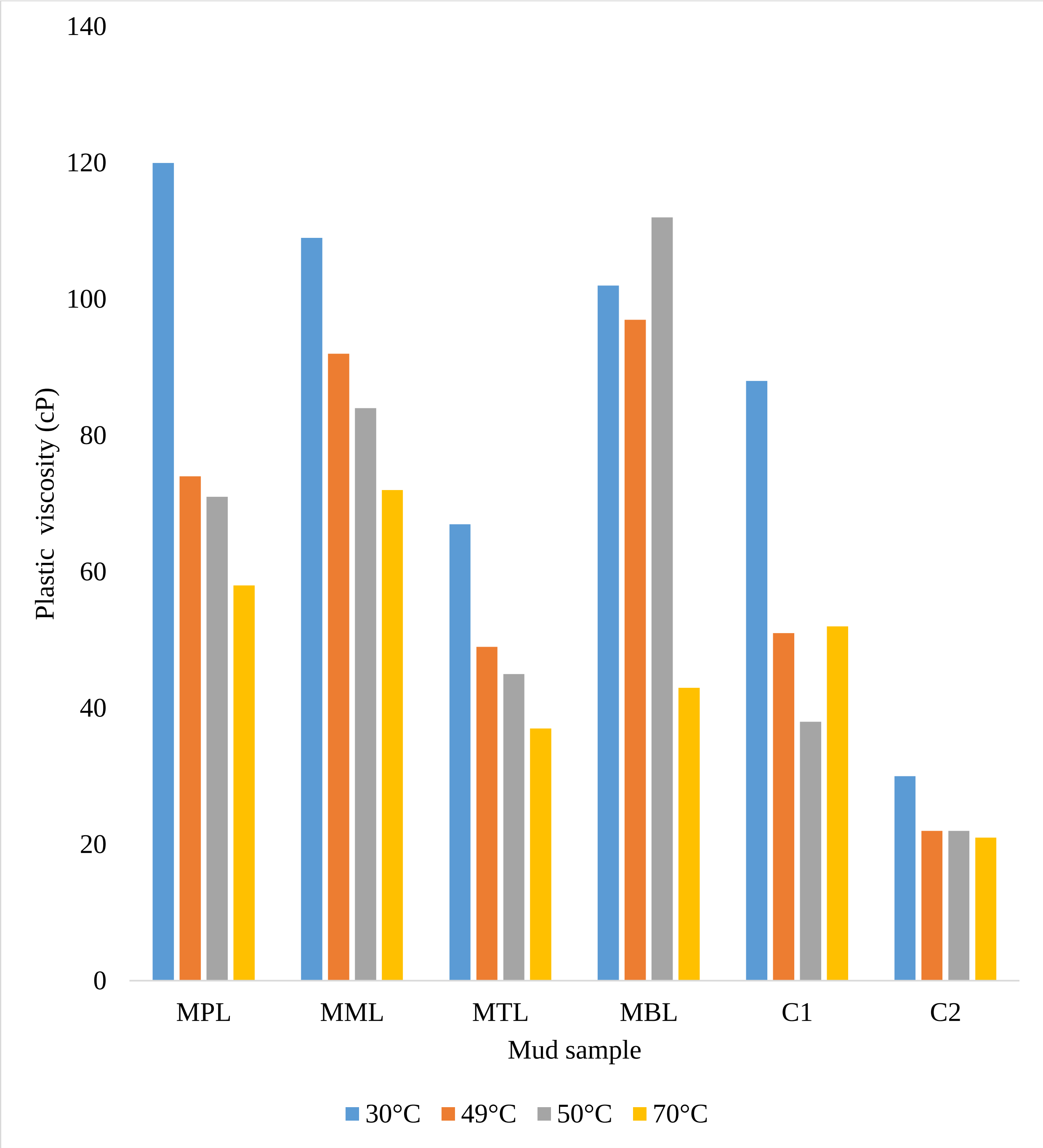
Appendix 7: Effect of temperature and 2% plant sample on PV of the drilling mud from different plant material



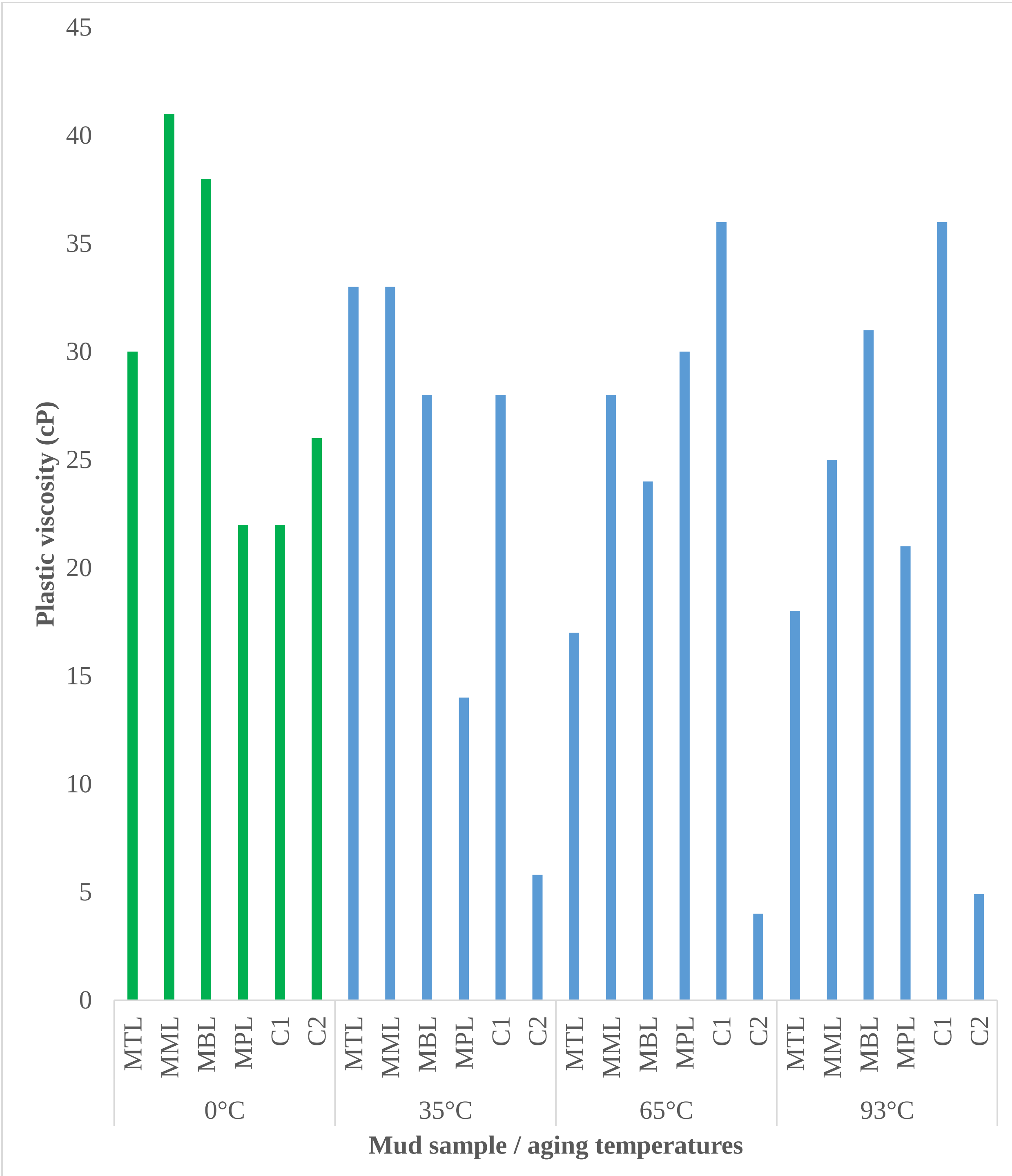
Appendix 8: Effect of temperature and 3% plant sample on PV of the drilling mud from different plant material



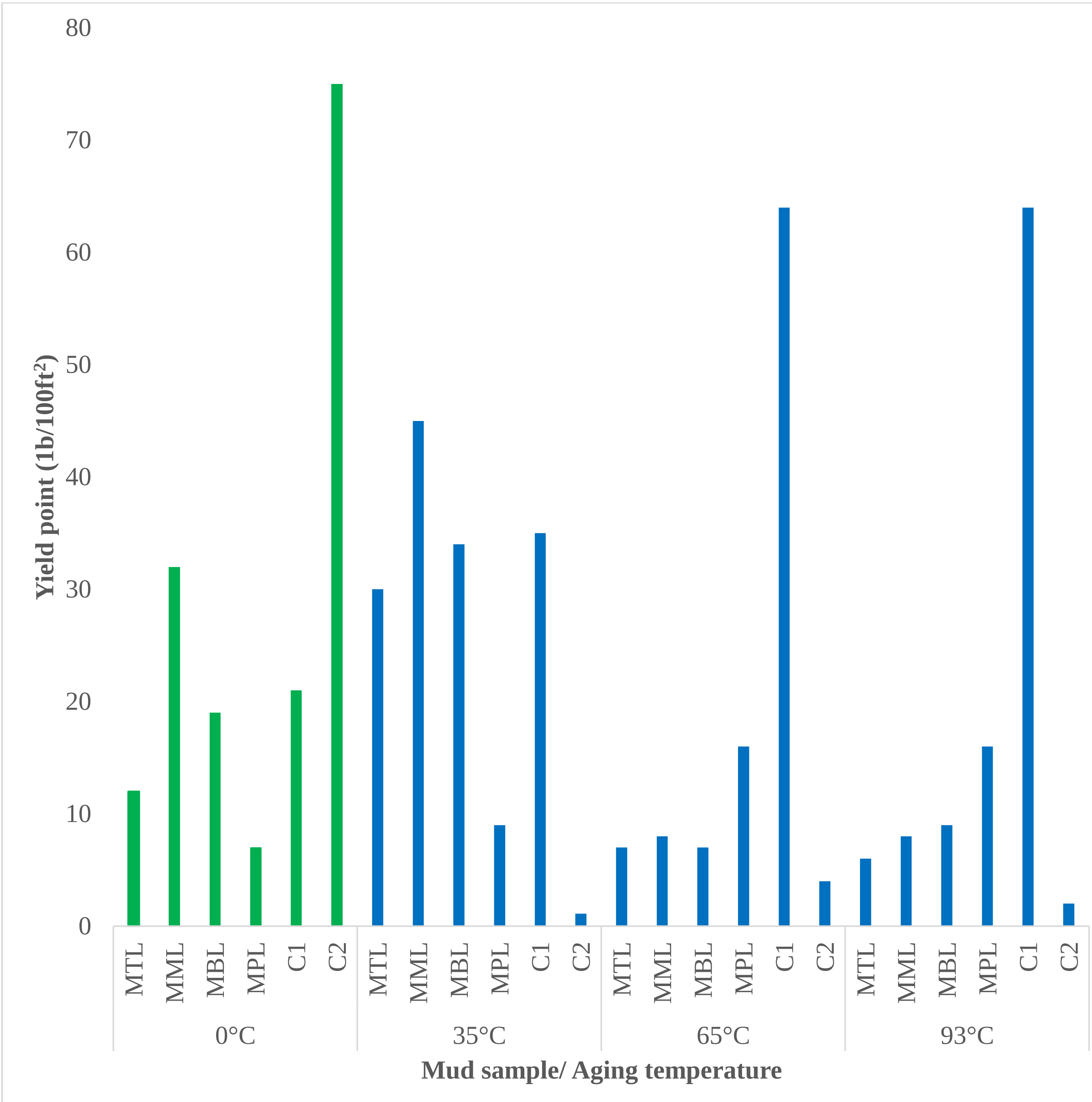
Appendix 9: Effect of temperature and 4% plant sample on PV of the drilling mud from different plant material



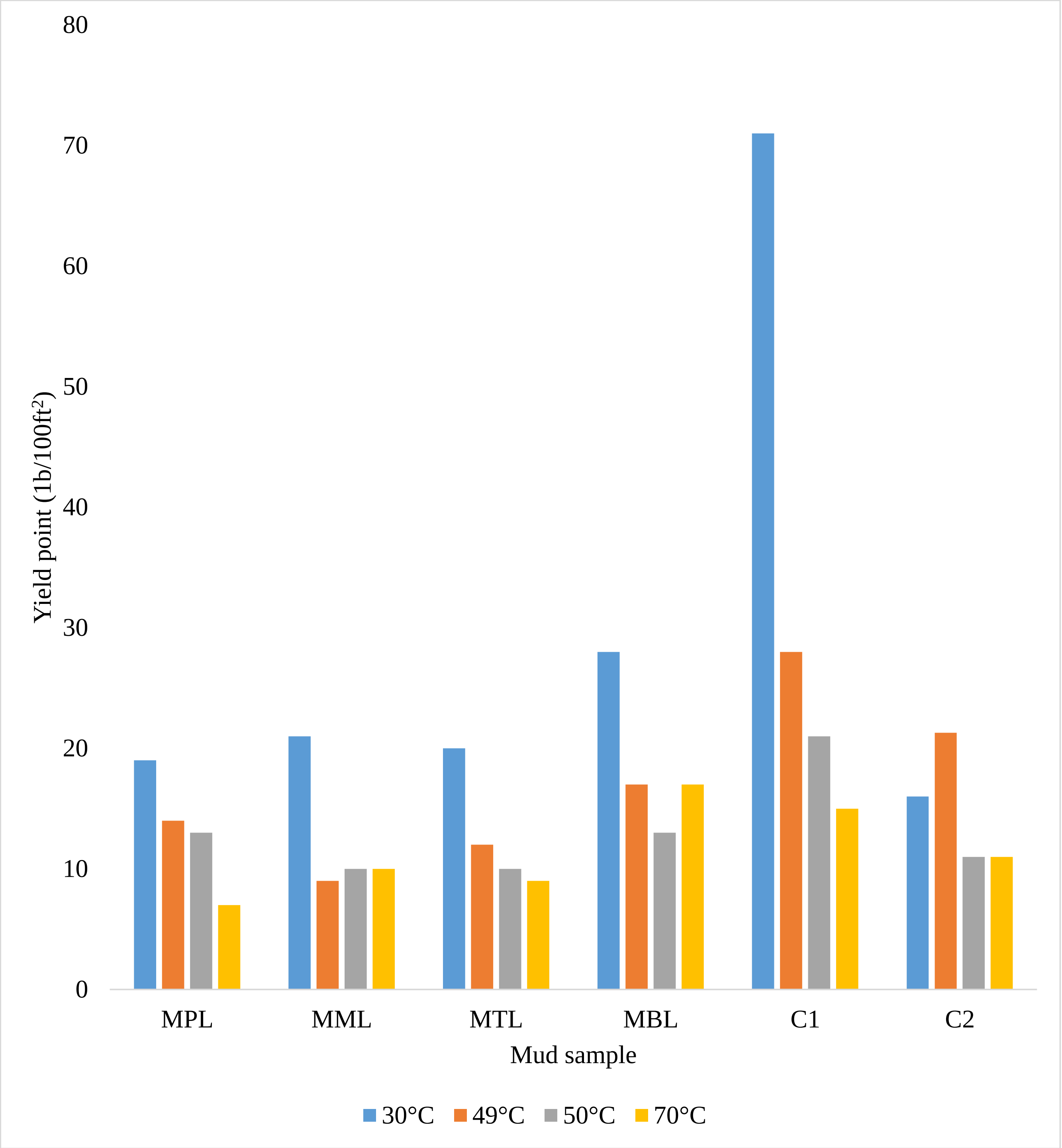
Appendix 10: Effect of aging on plastic viscosity



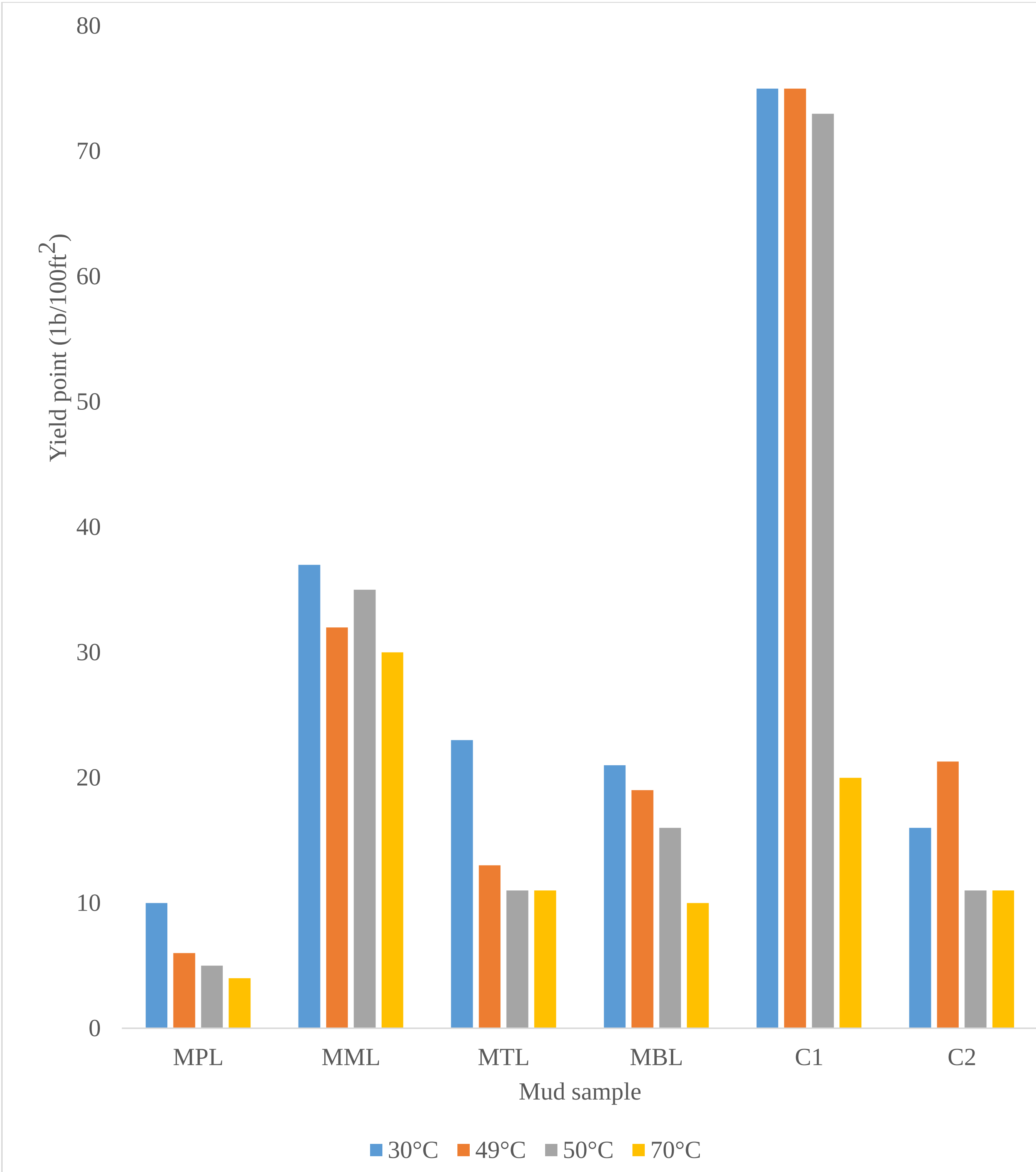
Appendix 11: Effect of aging on yield point



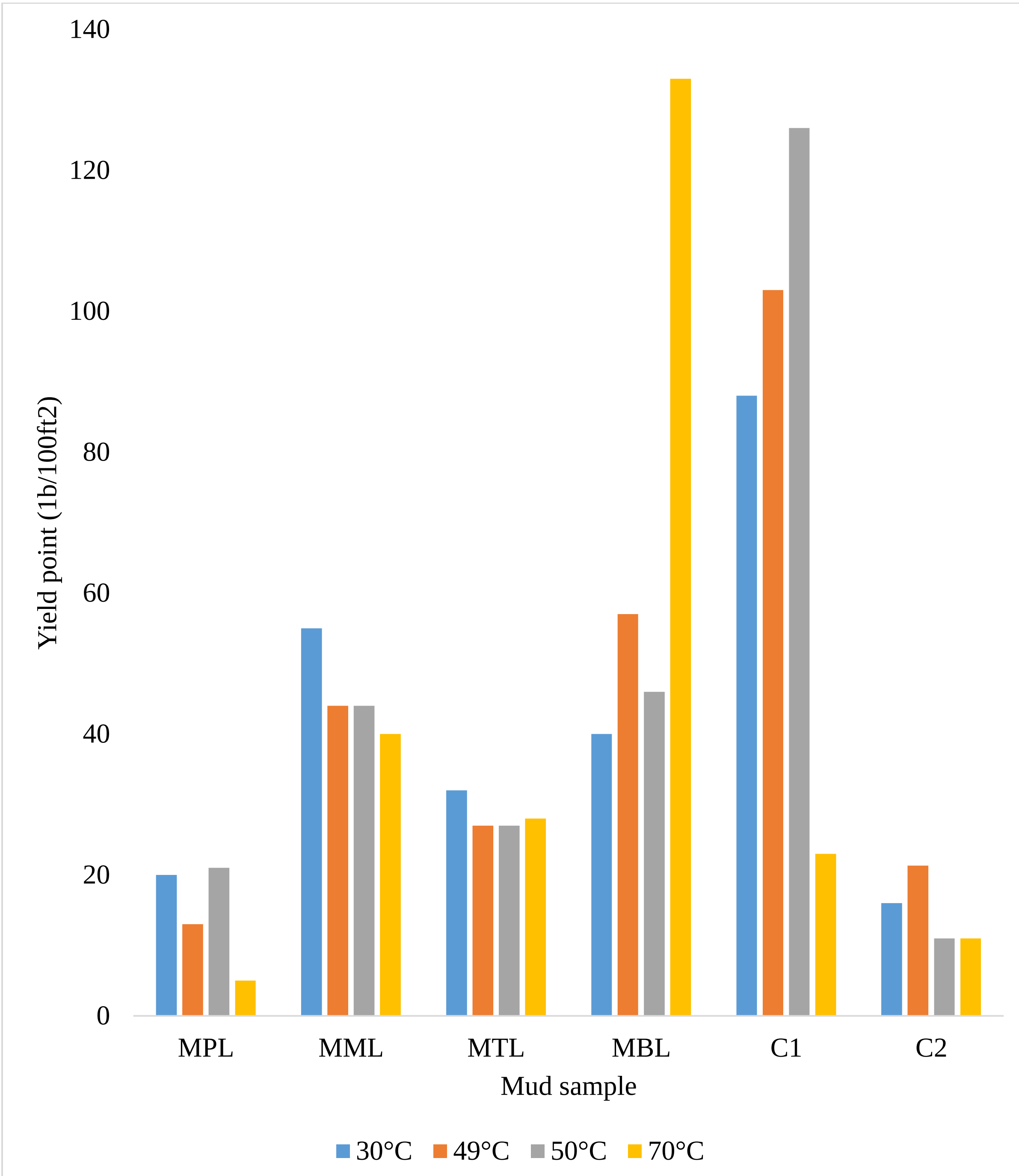
Appendix 12: Effect of temperature in 1% plant mud sample on YP



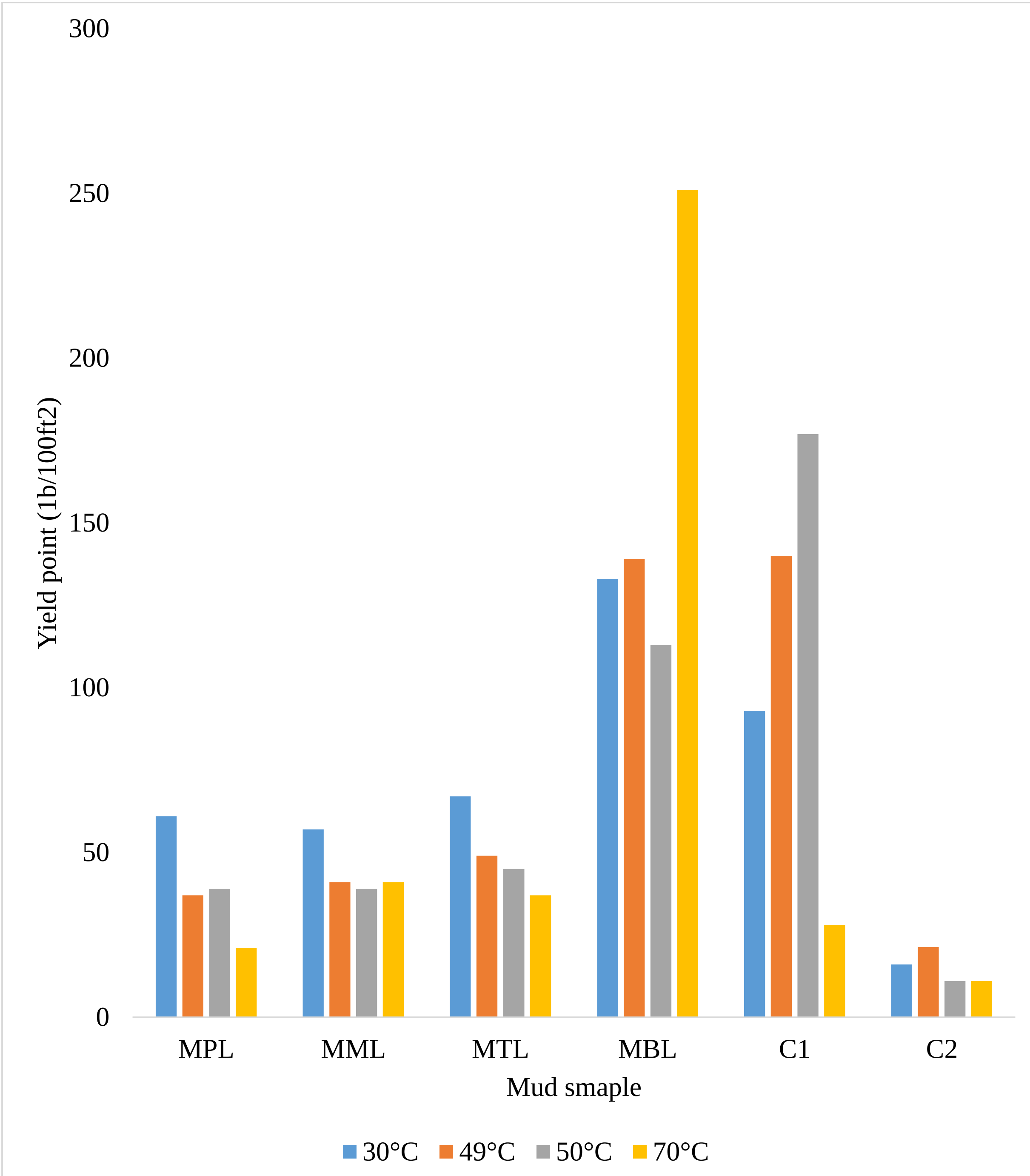
Appendix 13: Effect of temperature in 2 % plant mud sample on YP



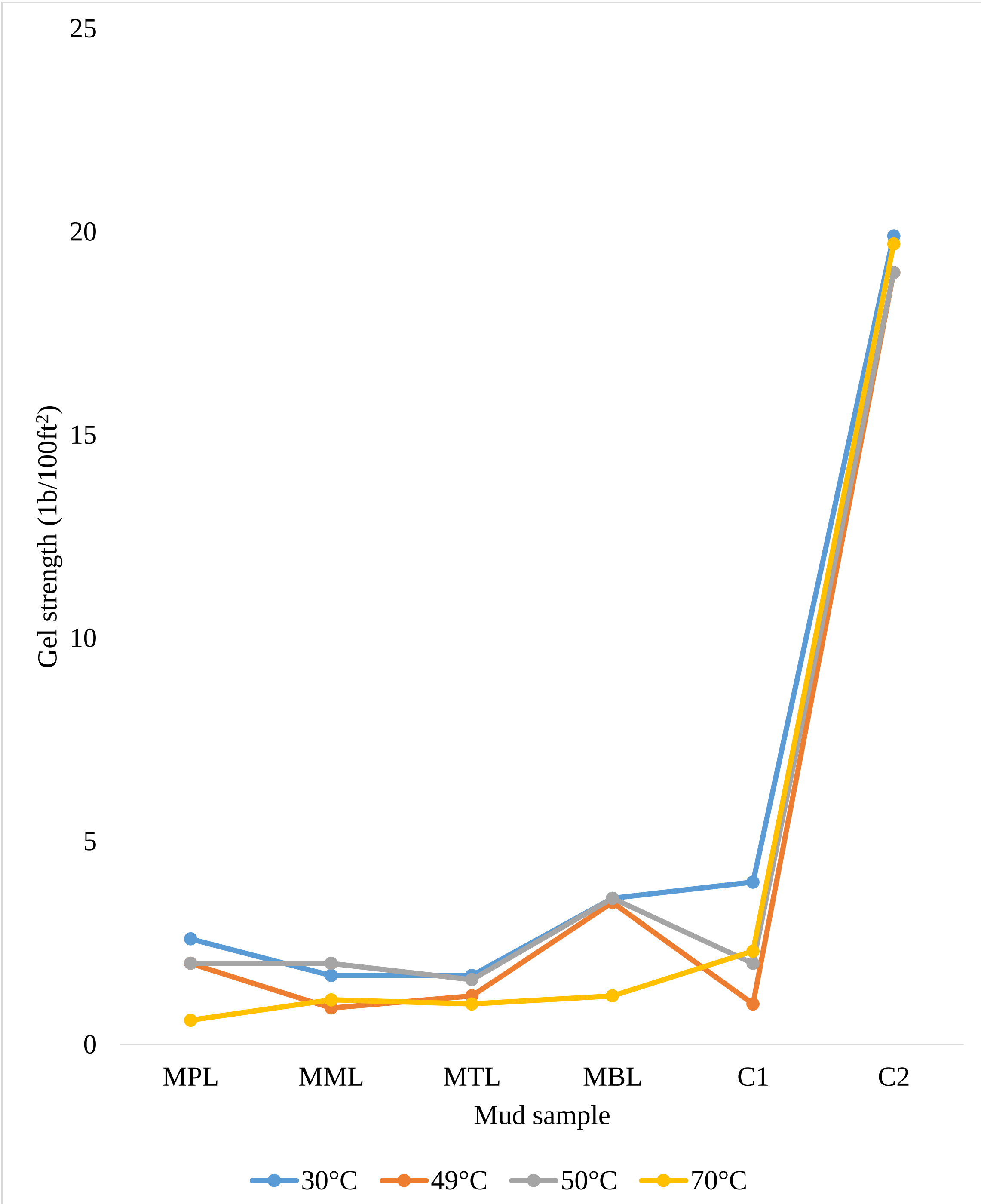
Appendix 14: Effect of temperature in 3 % plant sample mud on YP



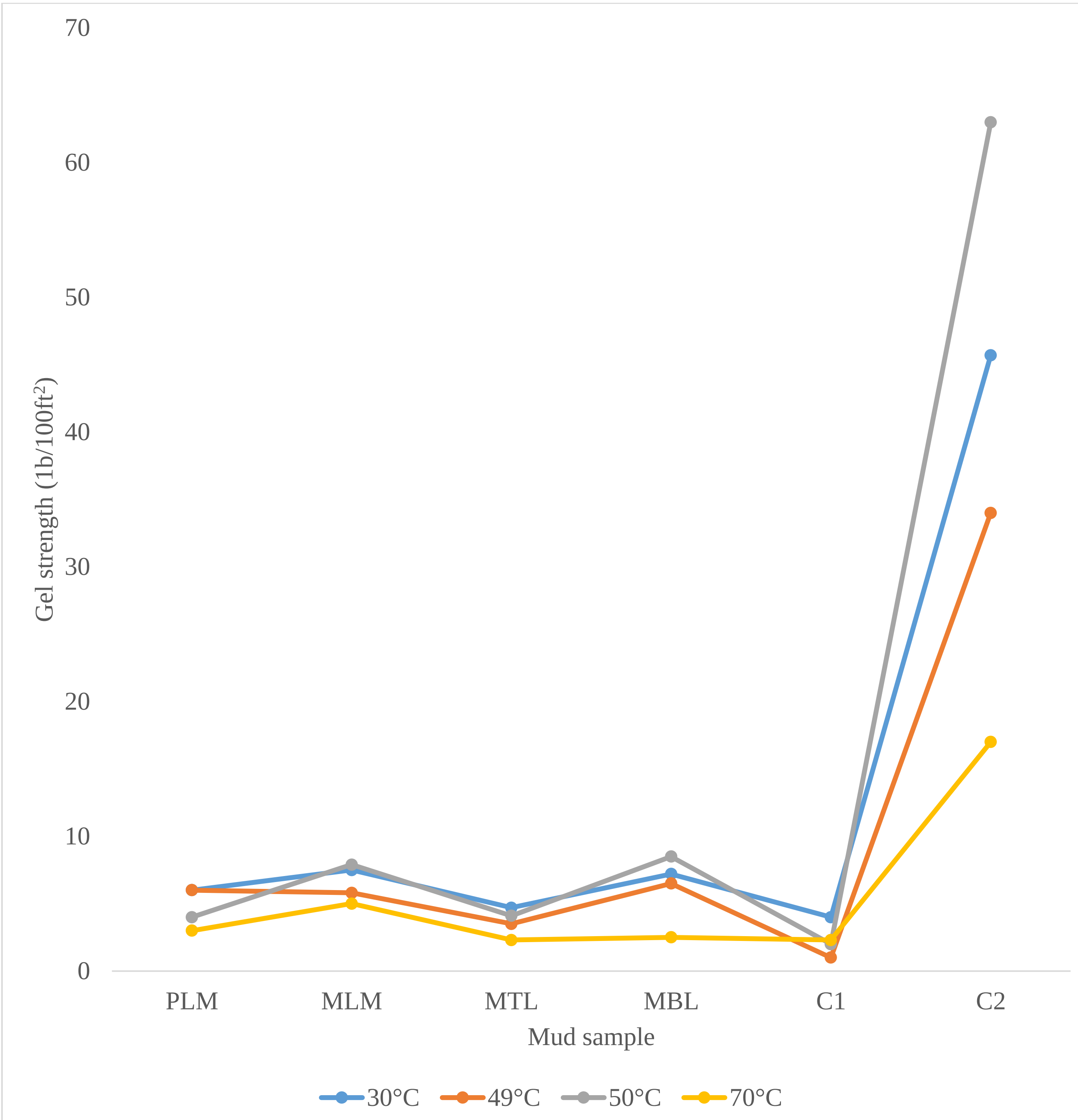
Appendix 15: Effect of temperature in 4 % plant sample mud on YP



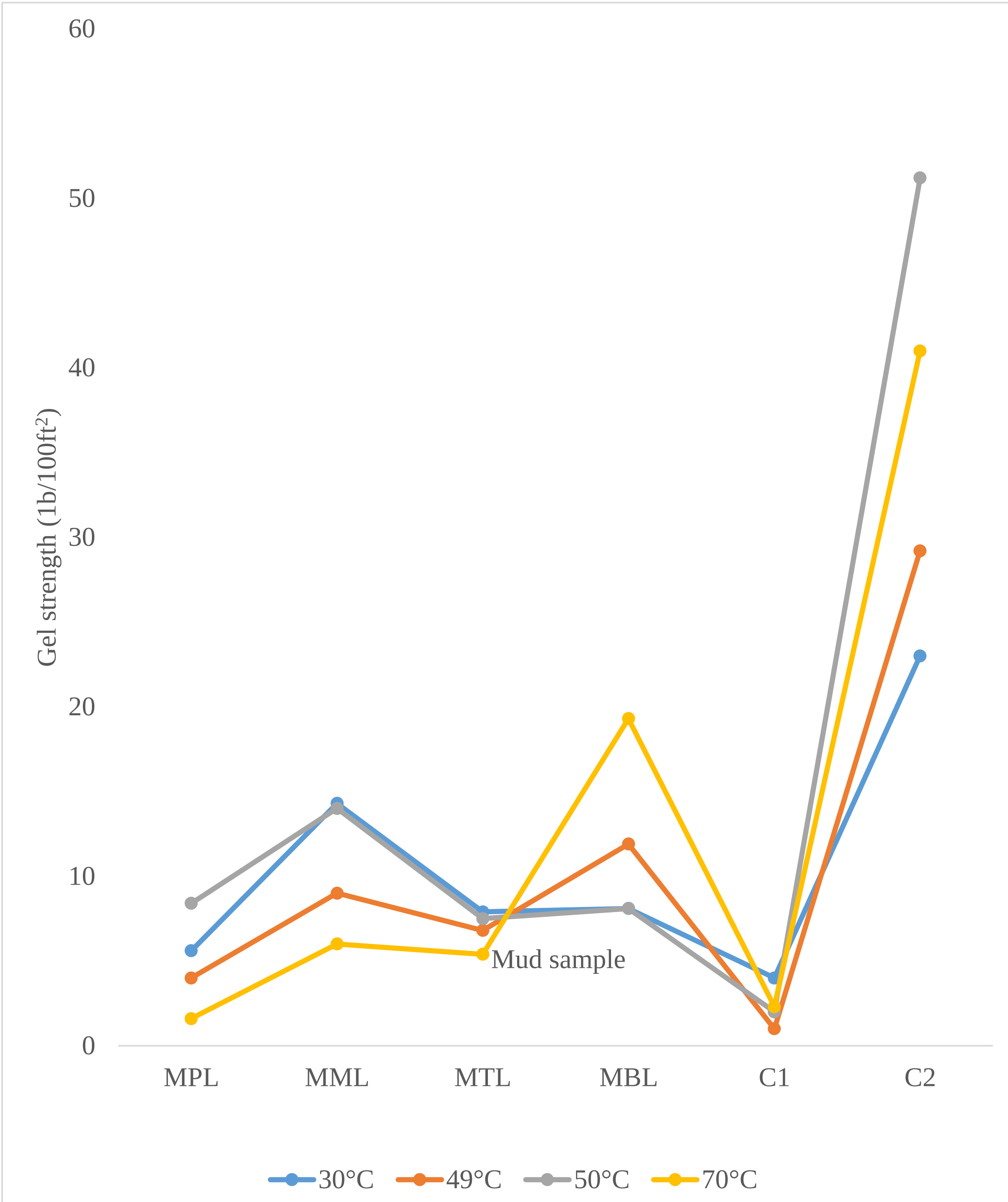
Appendix 16: Effect of temperature in 1 % plant sample mud on 10 second gel strength



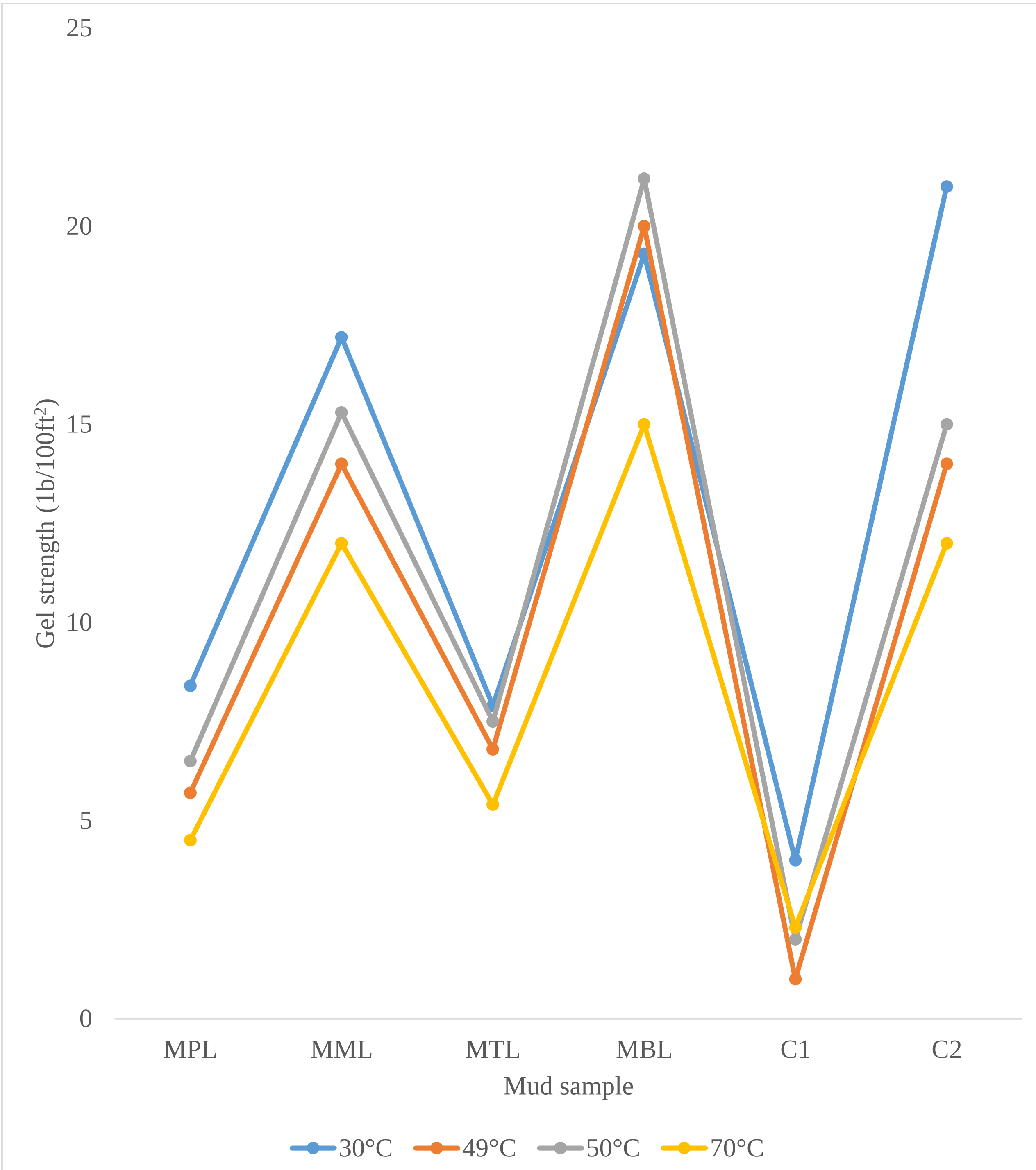
Appendix 17: Effect of temperature in 2 % plant sample mud at 10 second gel



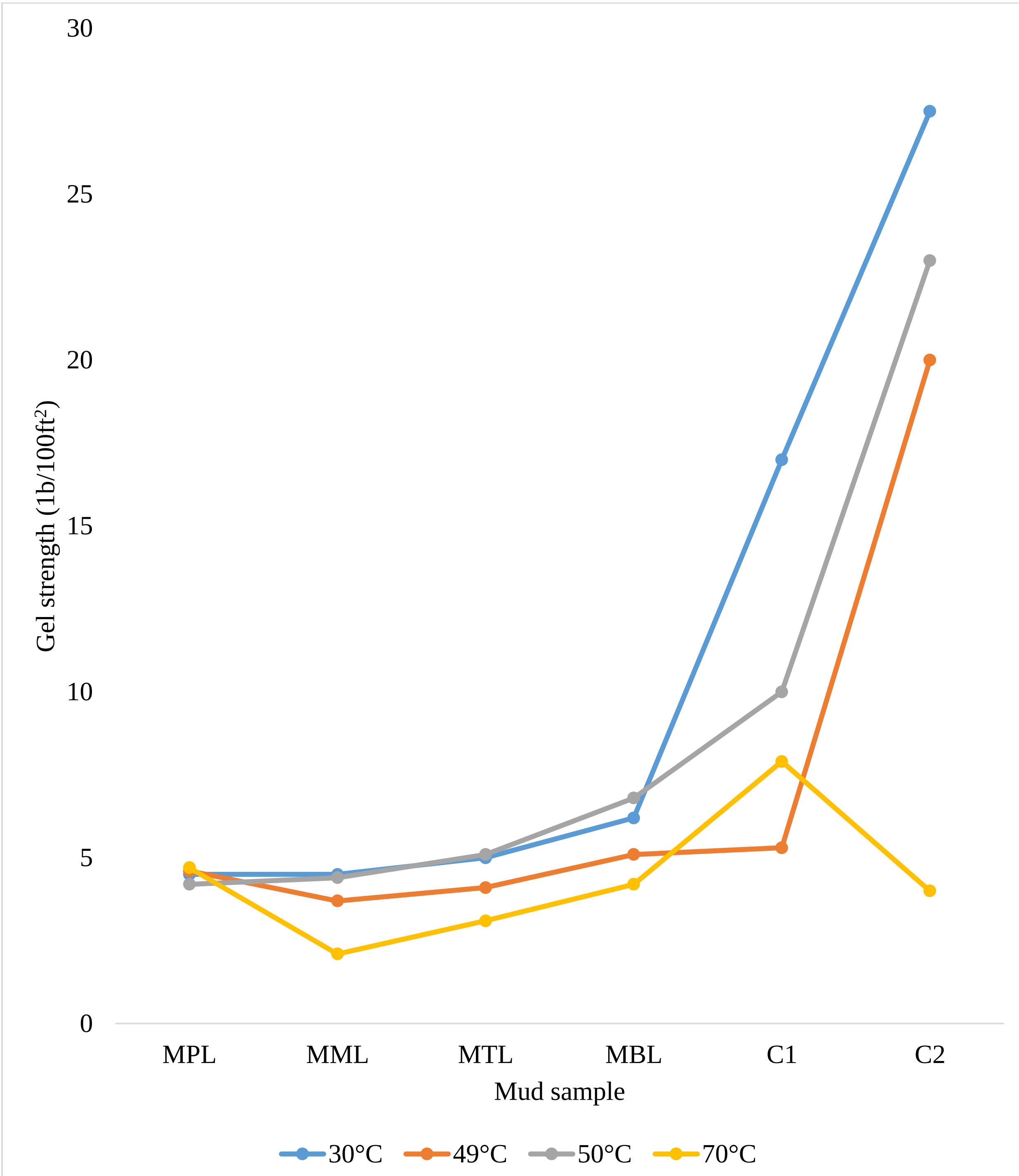
Appendix 18: Effect of temperature in 3 % plant sample mud at 10 second gel strength



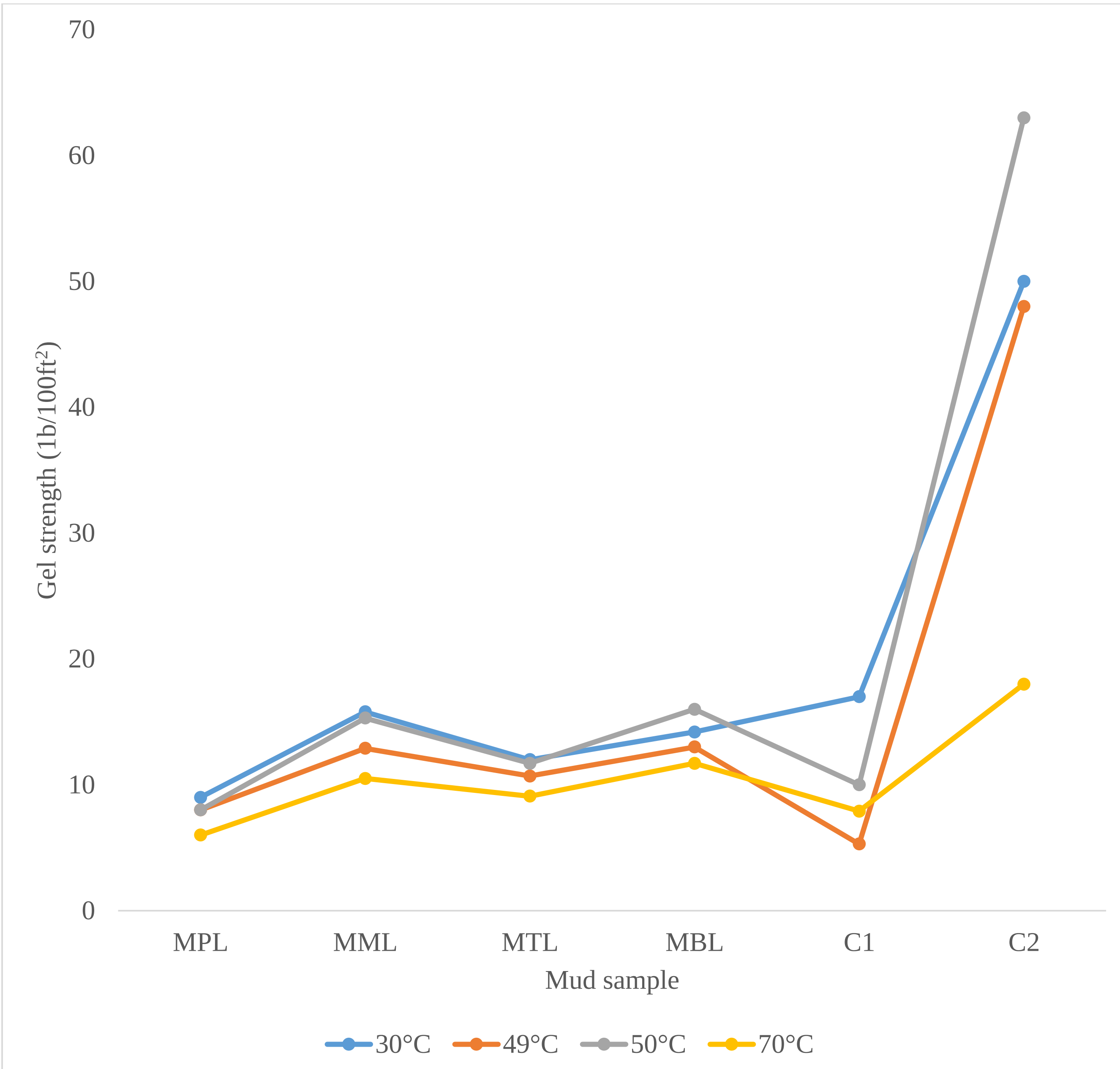
Appendix 19: Effect of temperature in 4 % plant mud sample



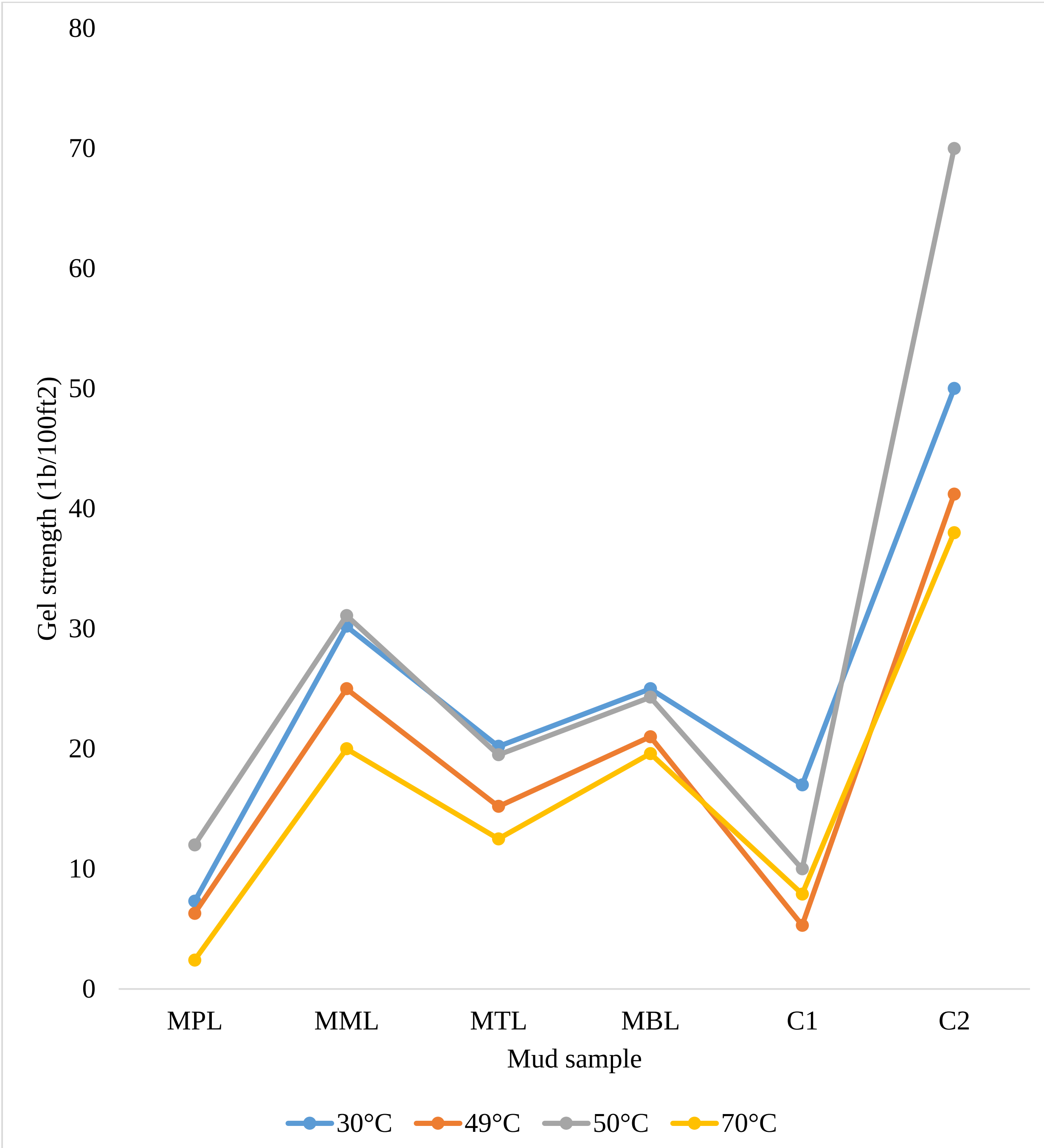
Appendix 20: Effect of temperature in 1 % plant sample mud at 10 minutes gel strength



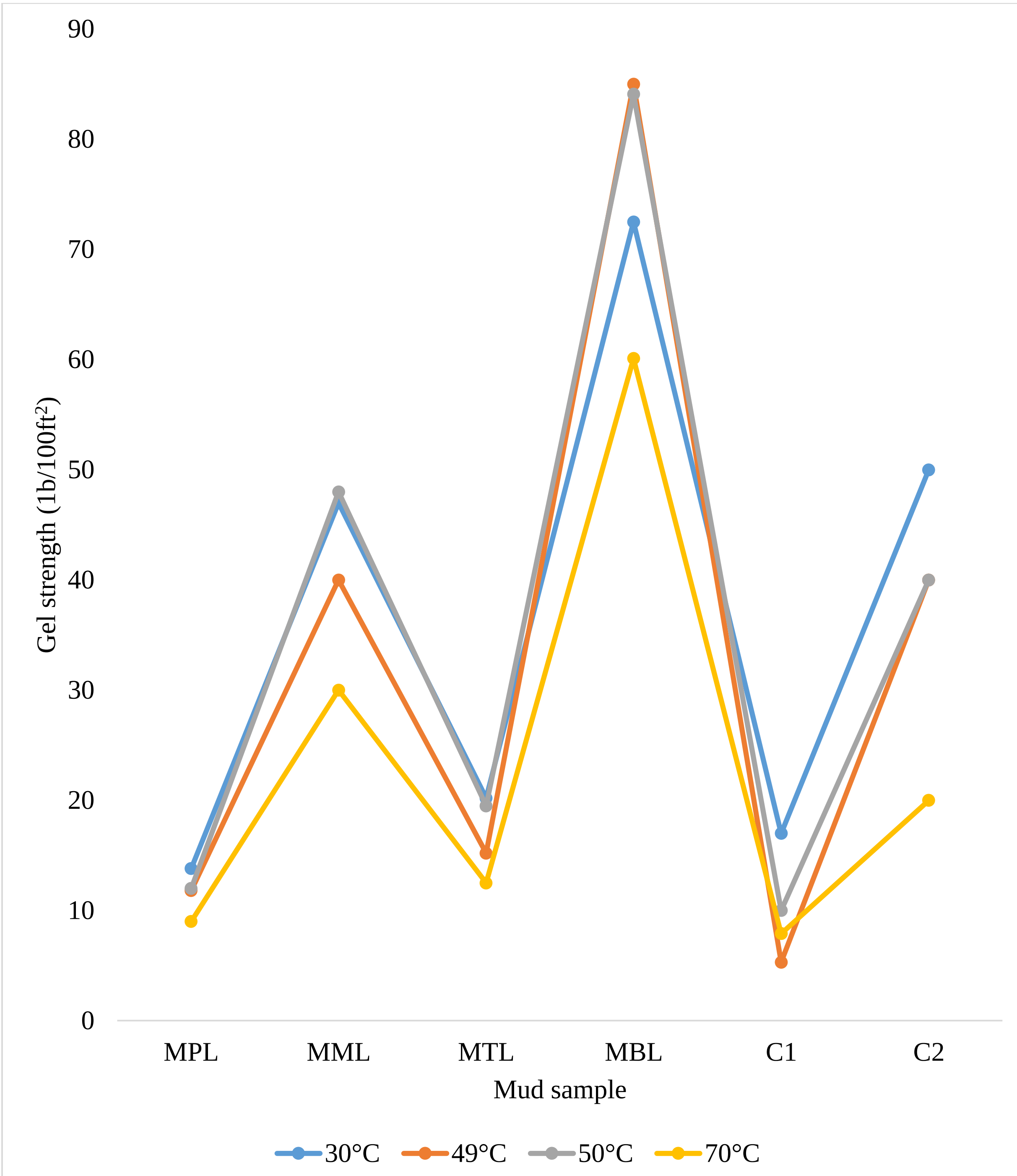
Appendix 21: Effect of temperature in 2 % plant sample mud at 10 minutes gel strength



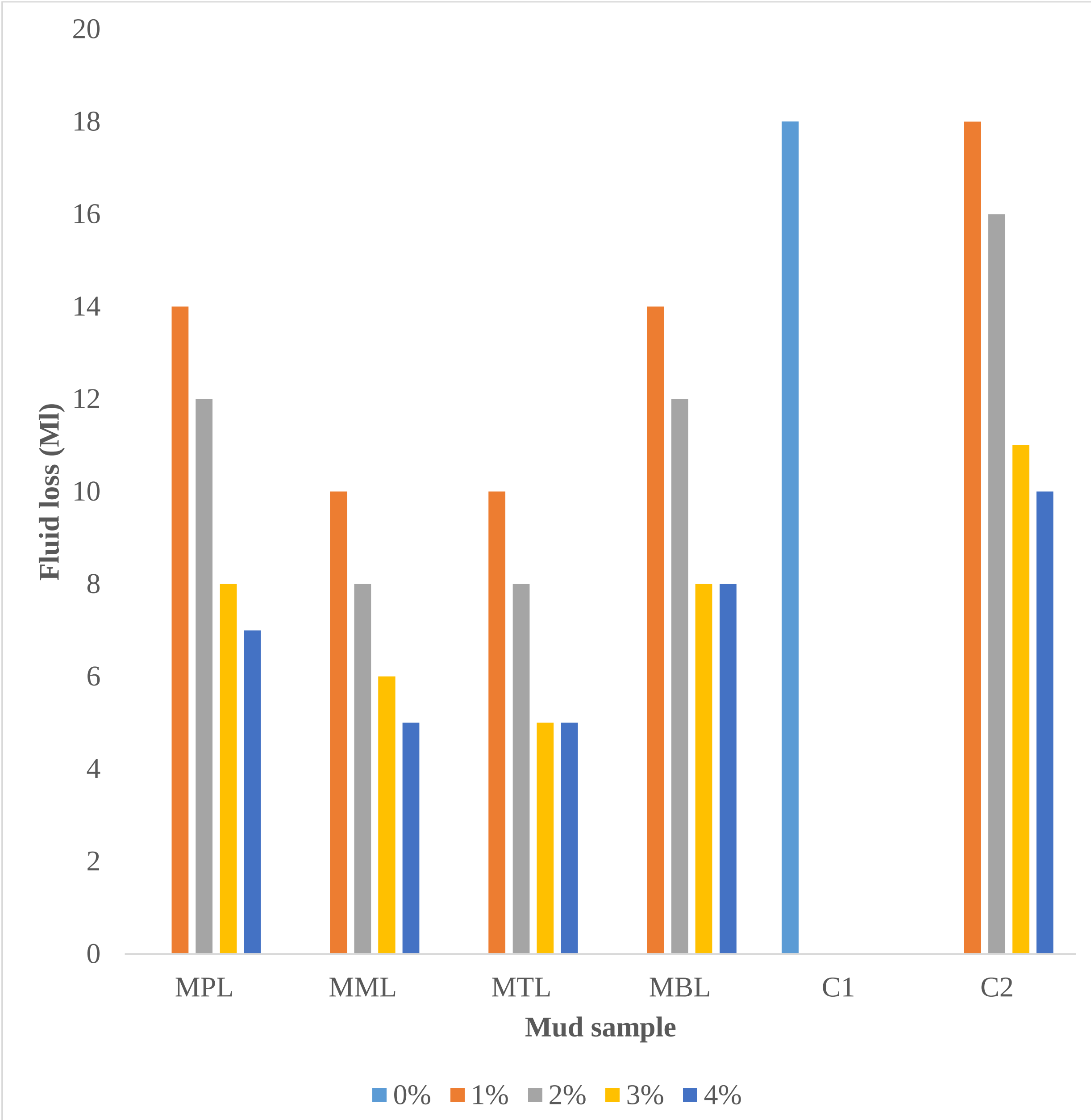
Appendix 22: Effect of temperature in 3% plant sample mud at 10 minutes gel strength



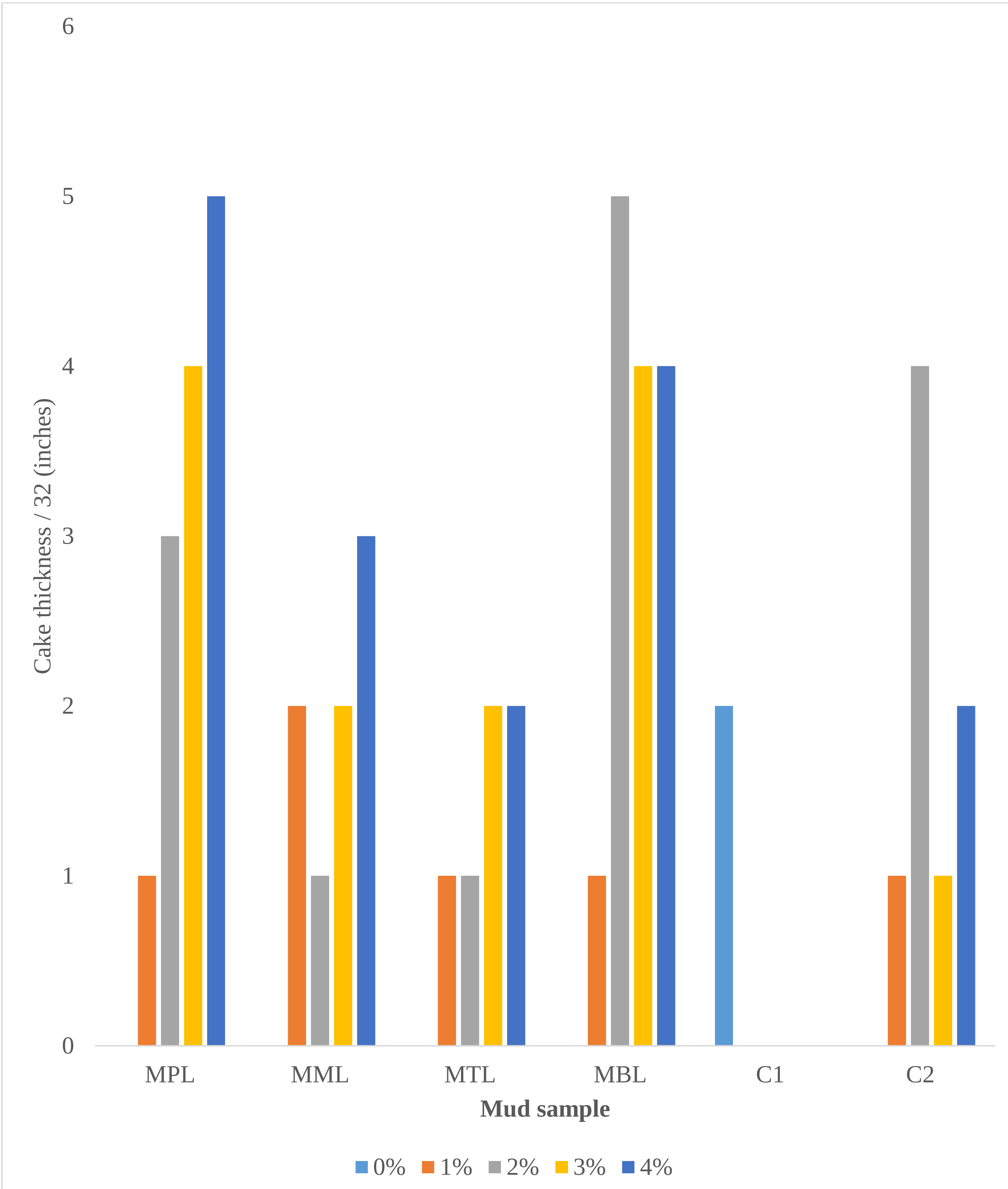
Appendix 23: Effect of temperature in 4% plant mud sample at10 minutes gel strength



Appendix 24: Effect of concentration on fluid loss control



Appendix 25: Effect of concentration on fluid loss control



Appendix 26: Effect of aging on 10seconds and 10 minutes gel strength at 120°F of the drilling mud