

**SYSTEMATIC INVESTIGATION OF DRILLING FLUID
IMPACT ON NMR INTERPRETATION FOR
FORMATION EVALUATION**

BY

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I dedicate this work to those holding on amidst the storm.

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In the Name of Allah, Most Gracious, Most Merciful. May the Peace and Blessings of Allah be upon our Noble Prophet Muhammad SAW, His Household, His Companions, and all people that follow Him upon righteousness. I thank Almighty Allah for granting me knowledge, energy, and endurance to undertake this task.

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LIST OF ABBREVIATIONS

NMR	:	Nuclear Magnetic Resonance
T ₂	:	Transverse Relaxation Time
T _{2B}	:	Bulk Relaxation
MF	:	Mud Filtrate
XC	:	Xanthan Gum Polymer
LPSA	:	Laser Particle Size Analyzer
MCT	:	Micro Computed Tomography scan
SEM	:	Scanning Electron Microscopy
XRD	:	X-ray Diffraction

ABSTRACT

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The invasion of mud filtrate into the formation during drilling alters the near-wellbore properties, potentially affecting well logging measurements. This invasion can lead to inaccuracies in the evaluation of key reservoir parameters such as porosity, permeability, and fluid saturation, resulting in unreliable assessments of the formation's storage and flow characteristics. Given the shallow depth of investigation associated with nuclear magnetic resonance (NMR) logging, the transverse relaxation time (T_2) spectrum is particularly vulnerable to the effects of this invasion. This study aims to explore how the bulk relaxation properties of drilling fluid and also experimental procedure during filtration experiments influence NMR T_2 relaxometry responses within the near-wellbore region. To begin, we systematically varied three factors that control the drilling fluid's bulk relaxation: 1) concentrations of XC polymer, 2) concentrations of perlite, and 3) concentrations of hematite in the drilling fluid. NMR measurements were performed on both highly permeable and tight limestone samples before and after mud filtrate invasion experiments to assess how the host rock properties influence the T_2 response. The results demonstrate that drilling fluid bulk relaxation significantly affects the NMR T_2 values and their distribution, particularly in invaded zones. Additives such as XC polymer, bentonite, hematite, and barite decrease the T_2 bulk values of the drilling fluid, which subsequently reduces the overall T_2 values in the invaded rock, especially in macropores. The pronounced impact in macropores is due to their higher sensitivity to bulk relaxation effects compared to micropores. Moreover, the study

reveals that rocks with larger pore sizes exhibit more substantial changes in T_2 values, while tighter rocks, where surface relaxation mechanisms dominate, show minimal changes. Lastly, we validated our experimental procedure of using rock as a filter during the filtration experiment against the common practice in the literature, where a standard filter paper is used instead. The results show that our experimental procedure provided a more realistic simulation of the reservoir's response to fluid invasion, leading to outcomes that more accurately reflect field scenarios. Overall, this study highlights the importance of accounting for drilling fluid bulk relaxation when interpreting NMR T_2 responses. It suggests that measuring the T_2 bulk relaxation properties of drilling fluids is essential for reliable NMR-based pore size and permeability evaluations. Additionally, employing filtration systems that closely mimic field conditions can significantly improve the accuracy of laboratory experiments, providing better insights into formation evaluation.

الملخص

الاسم الكامل: أكرمولا في فوزان أوريومي

عنوان الرسالة: التحقيق المنهجي في تأثير سائل الحفر على تفسير الرنين المغناطيسي النووي لتقييم التكوين

التخصص: الجيوفيزياء

تاريخ الحصول على الدرجة: 2024/12

غزو المرشح الطيني إلى المتكون أثناء الحفر يغير خواص المنطقة القريبة من البئر، مما قد يؤثر على قياسات تسجيل الآبار. يمكن أن يؤدي هذا الغزو إلى عدم دقة في تقييم المعلمات الرئيسية للخران مثل المسامية والنفاذية والتشبع بالسوائل، مما يؤدي إلى تقييمات غير موثوق بها لخصائص الخزان وتدفق المتكون. بالنظر إلى عمق التحقيق الضحل المرتبط بتسجيل الرنين يكون عرضة بشكل خاص لتأثيرات هذا الغزو. (T_2)، فإن طيف وقت الاسترخاء المستعرض (NMR) المغناطيسي النووي تهدف هذه الدراسة إلى استكشاف كيفية تأثير خواص الاسترخاء الكلي لسائل الحفر وإجراءات التجربة أثناء تجارب الترشيح داخل المنطقة القريبة من البئر. قمنا بتغيير ثلاثة عوامل تتحكم في الاسترخاء الكلي لسائل T_2 NMR على استجابات استرخاء و (3) تركيزات الهيماتيت في سائل الحفر. تم إجراء تركيزات البيرلايت، (XC2 الحفر بشكل منهجي: 1) تركيزات بوليمر على عينات من الحجر الجيري شديدة النفاذية والضيقة قبل وبعد تجارب غزو المرشح الطيني لتقييم كيفية تأثير NMR قياسات على عكس العديد من الدراسات التي تستخدم ورق ترشيح قياسي لتجارب الترشيح، T_2 خواص الصخر المضيف على استجابة استخدمنا نهجًا مختبريًا أكثر تمثيلًا باستخدام الصخر نفسه كمرشح. تم ذلك لمحاكاة الظروف الفعلية للمخزن بشكل أفضل. توضح وتوزيعها، خاصة في المناطق الغزو. تقلل T_2 NMR النتائج أن الاسترخاء الكلي لسائل الحفر يؤثر بشكل كبير على قيم في T_2 الكلية لسائل الحفر، مما يقلل بدوره القيم الإجمالية T_2 والبنطونيت والهيماتيت والباريت قيم XC الإضافات مثل بوليمر الصخر المغزو، خاصة في المسام الكبيرة. يرجع التأثير الواضح في المسام الكبيرة إلى حساسيتهم العالية لتأثيرات الاسترخاء الكلي مقارنة بالمسام الصغيرة. علاوة على ذلك، تكشف الدراسة أن الصخور ذات الأحجام المسامية الأكبر تظهر تغيرات أكثر ، بينما تظهر الصخور الأكثر إحكامًا، حيث تهيمن آليات الاسترخاء السطحي، تغيرات طفيفة. أدى استخدام T_2 جوهرية في قيم الصخر كمرشح في الإعداد التجريبي إلى محاكاة أكثر واقعية لاستجابة الخزان للغزو السيلي، مما أدى إلى نتائج تعكس بشكل أكثر دقة سيناريوهات الحقل مقارنة باستخدام ورق الترشيح. بشكل عام، تسلط هذه الدراسة الضوء على أهمية مراعاة الاسترخاء لسائل الحفر أمر T_2 إنه يشير إلى أن قياس خواص الاسترخاء الكلي. T_2 NMR الكلي لسائل الحفر عند تفسير استجابات

بالإضافة إلى ذلك، يمكن أن يحسن استخدام أنظمة NMR. ضروري لتقييمات الحجم المسامي والنفوذية الموثوقة المستندة إلى الترشيح التي تحاكي الظروف الميدانية بشكل وثيق دقة التجارب المختبرية، مما يوفر رؤى أفضل لتقييم التكوين.

CHAPTER 1

INTRODUCTION

Drilling fluids are essential for various functions such as removing cuttings, lubricating and cooling bits, and regulating formation pressure for a stable wellbore (Mohamed et al., 2021). In safety protocols, overbalance drilling is common when the bottom hole pressure exceeds the reservoir pressure (Adebayo et al., 2020). However, this can lead to drilling fluid infiltrating the formation, impacting petrophysical characteristics in the invasion zone (Gamal et al., 2021). The infiltration process depends on reservoir formation quality and external factors controlled by drilling parameters (Sun et al., 2021; Wu et al., 2019). Solid particles within the mud slowly aggregate to form impermeable cakes on wellbore walls, obstructing further penetration of mud filtrates. Figure 1.1 shows the process of mud invasion.

In oil and gas drilling, three main types of drilling fluids are commonly used: water-based mud, oil-based mud, and gas-based mud. Among these, water-based mud is the most widely employed, consisting of a mixture of solids, water, and chemicals. The solids in water-based mud can be either active, like clays that react with water or chemicals to perform specific functions, or inactive, like sand, which does not significantly react. The water used in water-based mud can vary based on availability, with seawater, brine, or freshwater being common options. Oil-based mud, where oil (often diesel) serves as the continuous phase, is used in conditions where shale swelling is a concern. Gas-based mud, composed of air or natural gas, may include foaming agents to enhance its properties. Water-based drilling fluids are favored for their environmental advantages and versatility, with advances in chemical additives and solid control technology enhancing their effectiveness (Growcock, 2004; Scott et al., 2015; Jaiswal and Verma, 2018). Figure 1.2 shows the general types of drilling fluid used in the oil and gas industry.

Drilling fluids have typically very different properties compared to the original formation fluid within the reservoir (Salaheldin et al., 2018). Understanding how the invasion of such fluids affects well logs is crucial for accurate petrophysical evaluation of the reservoir formation. Due to the shallow detection depth of NMR logging, the transverse relaxation (T_2) spectrum is highly sensitive to mud invasion (Walsh et al., 2001). The drilling mud filtrate invasion can significantly affect the depths that the NMR tool investigates, which range from 6.35 to 11.43 cm (Ge et al., 2021). As a result, invasion has an impact on the petrophysical properties estimated from NMR logs (Coates et al., 2000; Dunn et al., 2002; Minh et al., 2016; Ge et al., 2015; Zhao et al., 2017). The importance of NMR in formation evaluation cannot be overemphasized including porosity, pore geometry, permeability, and fluid saturation quantifications (Cheng et al., 2017, Elsayed et al., 2024, Elsayed et al., 2022., Yang et al., 2019; Abubakar et al., 2021, El-Husseiny and Knight, 2017; Kenyon et al., 1986). To better understand the impact of invasion on NMR formation evaluation, various researchers have conducted laboratory experiments to either examine the use of NMR for formation damage and mud cake evaluation or investigate the impact of drilling fluid invasion on NMR interpretation (e.g., Sun et al., 2021; Gamal et al., 2021; Wu et al., 2019; Chen et al., 2006; Marschall and Coates, 1997; Endurance et al., 2019; Liang et al., 2018; Hany et al., 2021; Bruno et al., 2021; Bageri et al., 2021; Adebayo et al., 2020). Figure 1.3 shows the drilling fluids: basic constituents, main functions, and classification.

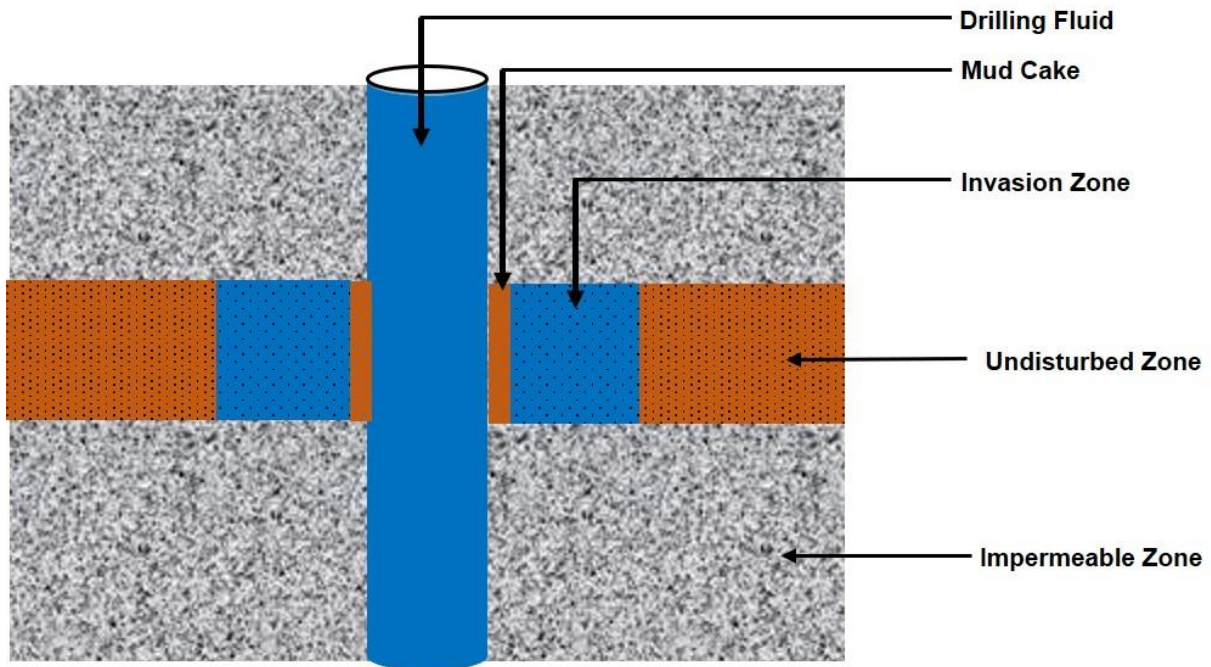


Figure 1.1: Diagram illustrating the process of mud invasion.

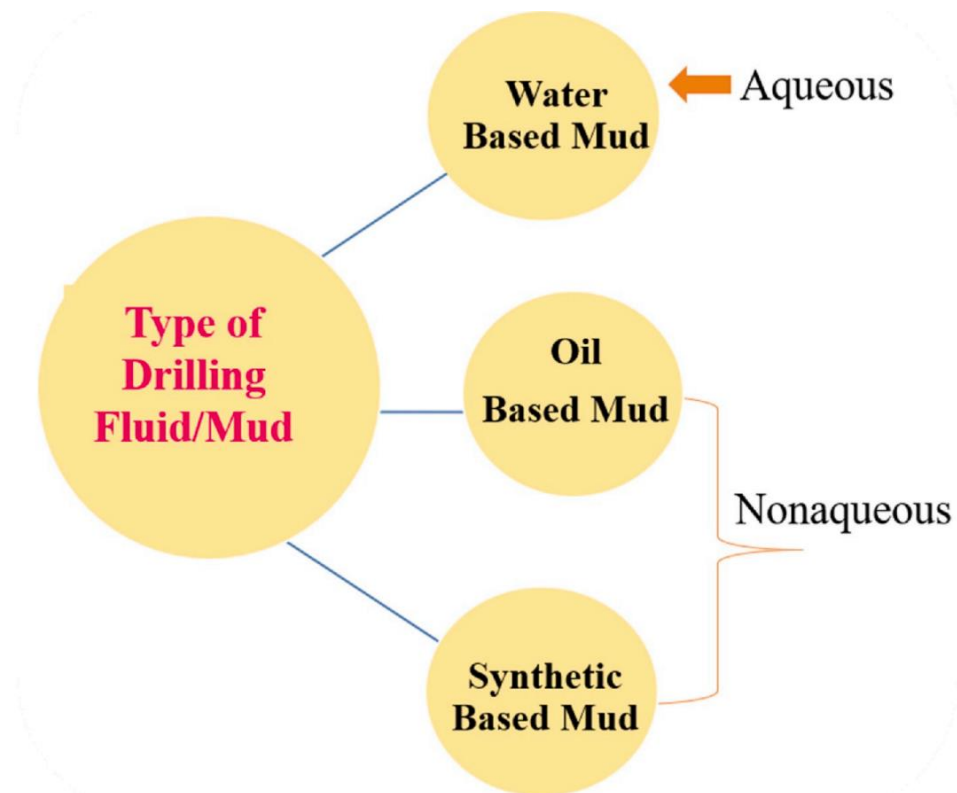


Figure 1.2: Type of drilling fluid used in the well drilling process

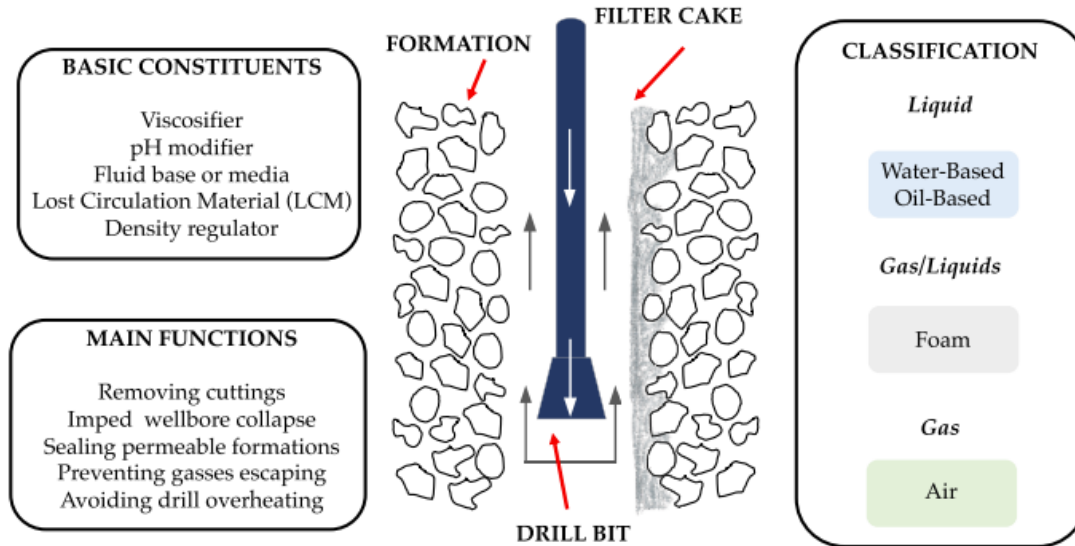


Figure 1.3: Drilling fluids: basic constituents, main functions, and classification.

1.1 Statement of the Problem

Although various researchers have explored NMR for filter cake characterization and studied the influence of additives on filtration control in different rock types, certain limitations remain. Notably, the bulk fluid relaxation of drilling fluids is generally lower than that of formation fluids, whether hydrocarbon or brine (Salaheldin et al., 2018). This difference can complicate NMR interpretation during invasion, as the altered relaxation times may affect the accuracy of petrophysical evaluations. However, the effect of bulk relaxation of drilling fluid was not considered in previous literatures. Additionally, it remains unclear how the host rock properties like pore size and permeability impact the extent by which NMR signal is altered by the drilling fluid invasion.

1.2 Aim and Objectives of the Study

The objective of this study is to examine the impact of drilling fluid invasion on NMR T_2 responses, with a focus on bulk relaxation of drilling fluid and particles invasion. To achieve this, the following specific research goals were established:

- i. Examine how the viscosifier and bulk T_2 relaxation time of drilling fluids affect NMR relaxation behavior and the response of the original reservoir fluids during mud filtrate invasion.
- ii. Investigate the influence of host rock properties, especially permeability and pore size distribution, on NMR measurements, with a focus on the potential impact of invading particles on rock damage and measurement accuracy.
- iii. Assess the impact of iron-rich particles used as drilling fluid additives on NMR signals, particularly the changes they induce in relaxation properties and their influence on measurement outcomes.
- iv. Validate our experimental procedure of using rock as a filter during the filtration experiment against the common practice in the literature, where a standard filter paper is used instead.

This study aims to provide insights into the importance of accounting for drilling fluid bulk relaxation when interpreting NMR T_2 responses. Additionally, it seeks to demonstrate that employing filtration systems that closely mimic field conditions can significantly improve the accuracy of laboratory experiments, ultimately enhancing formation evaluation.

1.3 Thesis Outline

In Chapter 2, I review the available literature on the influence of drilling fluid invasion on NMR T_2 response, offering a comprehensive analysis of the various approaches that past researchers have employed to understand and mitigate the distortion in NMR T_2 distribution. Several techniques have been tried to address the challenges posed by fluid invasion, each providing insights into specific aspects of the problem. However, these studies also exhibit significant limitations, leaving gaps in understanding. This chapter highlights these limitations, providing a critical perspective on where previous work has fallen short and setting the stage for further investigation.

In Chapter 3, I outline the research methodology designed to systematically investigate the effects of drilling fluid invasion on NMR T_2 measurements, organized into four key phases. The first phase involves varying the concentration of xanthan gum (XC) polymer in the drilling fluid to study its impact on viscosity and subsequent NMR bulk relaxation. In the second phase, I explore the effects of particle invasion by adjusting the concentration of perlite in the drilling fluid, aiming to observe the resulting changes in NMR responses. The third phase focuses on varying the concentration of iron-rich particles (hematite) to assess their effect on surface relaxivity and corresponding NMR T_2 measurements. Finally, the fourth phase consists of validating our experimental procedure of using rock as a filter during the filtration experiment against the common practice in the literature, where a standard filter paper is used instead. Through these phases, this study aims to elucidate the mechanisms by which drilling fluid invasion alters NMR T_2 measurements, thereby enhancing the understanding of factors affecting NMR interpretation in formation evaluation.

In Chapter 4, I present and discuss the results of the study. Each phase of the methodology yields specific insights into the relationship between drilling fluid invasion and NMR T_2 response. These findings are critically analyzed in relation to existing literature, confirming certain established trends while also offering new perspectives. The discussion highlights how the results contribute to a deeper understanding of the complex dynamics involved and points out any discrepancies with prior studies.

In Chapter 5, I conclude the study by summarizing its key contributions and stressing the importance of this research in advancing the understanding of drilling fluid invasion's impact on NMR T_2 responses. The study not only fills some of the gaps identified in the literature but also opens up new avenues for exploration. I suggest areas for further research, particularly in addressing the limitations of both this study and earlier works, paving the way for more refined approaches in future investigations.

CHAPTER 2

LITERATURE REVIEW

Drilling fluids are a crucial element in the drilling process, especially given the rapid advancements in the drilling industry. Enhancing the properties of drilling fluids is essential to meet the growing demands of modern operations. These fluids are primarily engineered to ensure that rotary drilling of subsurface formations is both feasible and cost-effective. Additionally, they serve several critical functions, including transporting cuttings to the surface, cooling and cleaning the drill bit, reducing friction, maintaining wellbore stability, and preventing premature entry of pore fluids into the wellbore. Moreover, drilling fluids are designed to form a filter cake, which aims to minimize filtrate loss to the formation, create a thin layer, and retain the drilling fluid within the wellbore (Watson et al., 2012).

The shallow detection depth of NMR logging makes its T_2 spectrum highly susceptible to the effects of mud invasion. While some degree of shallow particle invasion is necessary for filter cake formation on the formation face, excessive and uncontrolled invasion can negatively impact the near-wellbore region and the accuracy of petrophysical properties derived from NMR logs. The extent of particle invasion and deposition is influenced by factors such as particle concentration, size distribution, and the rheology of the drilling fluid, prompting laboratory experiments to replicate drilling fluid behavior and assess its impact on formation damage across various lithologies.

2.1 Types and Application of Drilling Fluids

Several types of drilling fluids are currently used in the drilling and petroleum industry, each selected based on the specific drilling conditions encountered. The main categories of drilling fluids used in the industry today can be classified into three groups.

2.1.1 Water-Based Mud (WBM)

This category of drilling fluid is characterized by the use of fresh or seawater as its continuous phase. Its formulation typically includes a blend of aqueous polymers, clay, and brine, which work together to enhance its performance. Water-based drilling fluids are favored for their environmental compatibility and cost-effectiveness. They are the most widely used type of drilling fluid in the industry due to their ability to effectively cool and lubricate the drill bit, maintain hydrostatic pressure, and carry cuttings to the surface. Additionally, these fluids can be easily modified with additives to suit specific drilling conditions and improve wellbore stability.

2.1.2 Oil-Based Mud (OBM)

This class of drilling fluids consists of oil, typically gas oil, as the continuous phase, with an average molecular weight ranging from about 120 to 1000. These fluids are synthesized from one or more olefin monomers featuring carbon chains that range from C₂ to C₁₄. Oil-based drilling fluids are primarily employed in situations where water-based muds are deemed inadequate. They are primarily formulated as reverse emulsions, where saltwater (brine) is dispersed in a continuous oil phase, stabilized by surfactants (Khodja et al., 2010). Although OBMs are less commonly used due to their higher cost, they offer improved performance in specific drilling applications, providing benefits such as enhanced lubricity, better hole stability, and reduced fluid loss to the formation.

2.1.3 Synthetic Based Mud (SBM)

This category of drilling mud, known as synthetic-based mud, is similar to oil-based mud but is less harmful in terms of toxicity and environmental impact. SBMs are primarily composed of olefins, esters, and sometimes paraffins, which are designed to provide optimal performance while minimizing environmental risks. One of the key advantages of SBMs is their low kinetic viscosity, allowing them to be effectively used under low-pressure drilling conditions. This property enhances flow characteristics, facilitating the efficient removal of cuttings from the wellbore and improving overall drilling performance. Additionally, SBMs offer excellent thermal stability and lubricity, making them suitable for challenging drilling environments, such as deepwater and high-temperature wells. Their reduced toxicity aligns with the industry's increasing focus on environmentally friendly practices, providing a balanced approach to drilling fluid technology by delivering necessary performance while being more compatible with environmental regulations.

2.1.4 Emulsion Drilling Mud (EDM)

These drilling fluids belong to a group characterized by emulsion-based phases and consist of two components: the outer phase, which is made up of saltwater (brine), and the inner phase, which consists of oil. To ensure the two phases are miscible, surfactants are added to counteract the opposing emulsion effects. In terms of application, these fluids are relatively inexpensive and environmentally friendly compared to oil-based muds (OBM), although they are costlier than water-based muds (WBM) (Shah et al., 2010). Their formulation allows for effective drilling in various conditions, making them a versatile choice for operators seeking to balance performance with environmental considerations. Overall, the combination of low cost and reduced environmental impact positions these emulsion-based drilling fluids as a practical option in the industry.

2.1.5 Invert Emulsion Mud (IEM)

This category of drilling fluids is quite similar to emulsion-based drilling muds (EDMs) regarding chemical composition, cost, environmental impacts, and applications, but it differs in phase distribution. As the name suggests, it is the inverse of EDMs, comprising two phases where the outer phase consists of oil and the inner phase consists of saltwater (brine). These inverse emulsion drilling fluids are characterized by their light viscosity, making them particularly suitable for high-temperature conditions. Additionally, they are easily biodegradable by microorganisms, which further enhances their environmental compatibility. This combination of properties allows them to effectively meet the challenges of various drilling environments while minimizing ecological risks, making them a favorable option in the industry.

2.1.6 Air Drilling Fluid

This category of drilling fluids primarily consists of aerosols and is mainly utilized for underbalanced pressure drilling, where there is minimal or no connection to the reservoir oil or water (Shah et al., 2010). The advantages of aerosol-based drilling fluids over other types lie in their high penetration rates, minimal damage to the formation, zero circulation loss, and little to no contamination of the surrounding rock. According to Shah et al. (2010), a thorough understanding of the formation hydraulics is crucial for the successful application of these fluids. This knowledge ensures that the unique properties of aerosol drilling fluids can be effectively leveraged to optimize drilling performance while maintaining well integrity.

2.1.7 Foam Drilling Fluids

Foam drilling fluids consist of approximately 5 - 25% liquid and 75 - 95% gas phases (Shah et al., 2010). They are primarily used in situations where the pressure of the drilling fluid is lower than the pressure of the hydrocarbons contained within the pores of the rock formation. As a result, they are commonly applied in underbalanced or deep-water wells, which is why they are often referred to as "underbalanced fluids" (Khodja et al., 2010). Foam drilling fluids are considered among the most effective drilling fluids available, as tests conducted under controlled drilling rates have demonstrated that air or gas is a faster drilling medium compared to water or oil. In contrast, water is regarded as the fastest drilling liquid when compared to oil. This combination of properties makes foam drilling fluids a preferred choice in challenging drilling environments.

2.2 Factors to consider while choosing a Drilling Fluid

The selection of a drilling fluid type or system typically begins with choosing a drilling fluid family based on the characteristics of the rock formation, while also considering financial implications and environmental impacts. Although drilling fluids account for only 5% to 15% of the total drilling cost, they can contribute to 100% of drilling problems (Bloys et al., 1994). When choosing a drilling fluid, emphasis is placed on several key factors, including well design, formation pressure, rock mechanics, and the chemistry of the formation. Additionally, it is essential to limit negative impacts on the producing formation while adhering to environmental issues and regulations. Logistics and cost considerations also play significant roles in the decision-making process, ensuring that the selected drilling fluid is not only effective but also economically viable and environmentally responsible. Figure 2.1 shows the schematic of a drilling mud circulation system.

2.3 Properties of Drilling Fluids and well control effects

To effectively address these critical factors, a drilling fluid should exhibit five fundamental properties: rheology, fluid loss, chemical properties, solid content, and density. Each of these properties can be influenced by various chemicals and additives (Bloys et al., 1994). Rheology pertains to the viscosity of the fluid; a highly viscous fluid is capable of efficiently transporting drill cuttings and weighting agents to the surface. Density is crucial for maintaining sufficient hydrostatic pressure to prevent borehole collapse and inhibit formation fluids from entering the wellbore, with drilling muds having a higher density than the cuttings facilitating quicker and easier wellbore cleaning. A good drilling fluid also minimizes fluid circulation loss by maintaining low permeability, effectively sealing the pores between the formation and wellbore. The solid content can include high-gravity solids (HGS), such as barite and other weighting materials, or low-gravity solids (LGS), such as clays and polymers; higher solid contents typically lead to increased plastic viscosity and gel strength, resulting in thicker filter cakes and slower drilling rates compared to lower solid content fluids. Lastly, the chemical properties of the drilling fluid are vital for performance and wellbore stability, as they monitor factors like salt formations, PH levels, and corrosion effects.

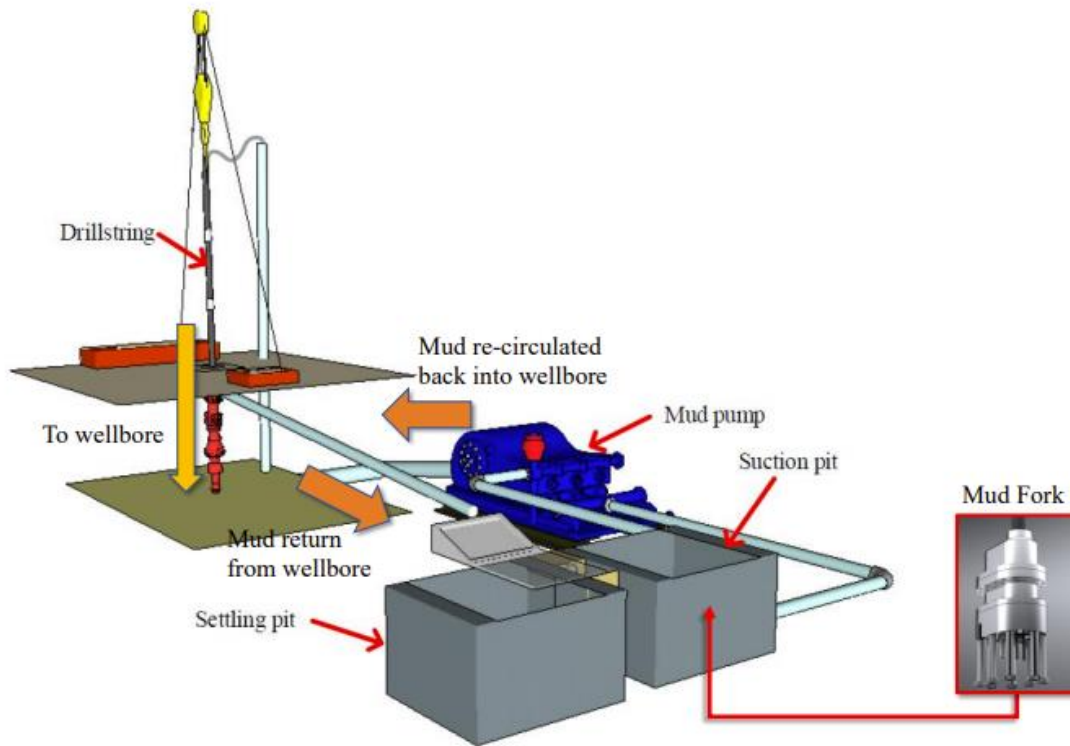


Figure 2.1: A simplified drilling mud circulation system shows mud pumped downhole through the drillstring, returning via the annular space, cleaned at shale shakers, and recirculated from the suction pit, where the mud fork is installed (Miguel et al., 2021).

2.4 Related Previous Work Done

Several studies have been executed to study influence of drilling invasion on relaxometry response.

Van der Zwaag et al. (2001) investigated core plug samples from sandstone and chalk and noticed significant alterations in NMR signal response after displacing 50% of the original pore fluids with drilling fluid filtrate. The results indicated that water-based filtrate invasion caused a shift to shorter T_2 values, leading to an underestimation of free-fluid volumes using standard cut-off values for both sandstone and chalk. Conversely, oil-based filtrate invasion resulted in signals that suggested larger free-fluid volumes than initially present. Additionally, NMR-permeability calculations based on the Timur-Coates Free Fluid model showed variations by up to one order of magnitude, highlighting the substantial impact of drilling fluid invasion on NMR measurements.

Endurance et al. (2019) integrated Nuclear Magnetic Resonance (NMR) logging into the petrophysical evaluation. During their study, in well B, they found out that the NMR spectrum is affected by a significant OBMF invasion. They proposed it was probably caused by prolonged invasion due to slow mudcake buildup in the low permeability rocks. This invasion issue was corrected using fluid substitution techniques and the NMR analysis was integrated into petrophysical evaluation, aiding in selecting optimal intervals for stimulation and correlating fluid sample data from formation testers with NMR-based predictions.

Abdulrauf and Bageri (2020) introduced a novel nuclear magnetic resonance (NMR) technique for evaluating filter cake properties in drilling fluids. This method assesses filter cake thickness, pore volume, porosity, and potentially permeability, along with the amount and particle size distribution of solids invading rock samples. The technique was tested on four identical rock samples subjected to drilling fluid invasion and filter cake deposition, with NMR measurements taken at various stages: before deposition, after deposition, and after removal. The NMR method

provided high accuracy and simultaneous evaluation of multiple filter cake properties, offering a more efficient alternative to conventional techniques that require multiple procedures and can disturb the sample state. Additionally, the method helps evaluate secondary damage from the filter cake removal process.

Bageri et al. (2021) investigated the effectiveness of using nuclear magnetic resonance (NMR) to characterize filter cakes formed by drilling fluids, comparing it with conventional methods like gravimetric and X-ray computed tomography (CT). The study aimed to address the challenges of fluid infiltration into underground rocks during drilling, which can lead to issues like fluid loss, formation damage, and borehole collapse. Fluid loss tests were performed on four consolidated sandstone and carbonate rock samples, with NMR measurements taken before and after filter cake deposition. The results showed significant differences between the NMR method and the conventional methods. Cross-correlation analysis revealed that NMR measurements of filter cake porosity and infiltrated solids had a stronger correlation with cake thickness compared to the other methods, although NMR reported lower porosity values. The study explained why conventional methods tend to overestimate porosity and highlighted the NMR method's accuracy and usefulness for characterizing filter cakes in drilling operations.

Bruno et al. (2021) introduced a new approach to improve the interpretation of nuclear magnetic resonance (NMR) well log data in the Santos Basin's Pre-salt carbonate reservoirs, which are characterized by complex and heterogeneous pore systems. The study addresses the issue of unreal NMR readings caused by drilling mud invasion in caverns and vugs, leading to a shift in T_2 distribution towards lower relaxation times. Analyzing data from 28 wells in the Sapinhoá Field, the authors used special core analyses, routine core analyses, and other data to develop a method involving five key steps: computing new curves, detecting invasion-affected zones, calculating

water saturation using the saturation height model, generating a synthetic NMR free fluid curve, and estimating a synthetic T_2 distribution using the Random Forests algorithm. The approach minimized errors, increased pay zone thickness by up to 10%, and reduced water saturation averages by up to 9%, potentially translating into significant oil recovery. This method enhances petrophysical interpretations in highly heterogeneous carbonate reservoirs, thereby reducing exploration and development risks.

Hany et al. (2021) investigated the impact of mud filtrate on the pore systems of various sandstone rocks, including Berea (Buff and Spider), Bandera Brown, and Parker. Using water-based mud (weighted with barite), the study simulated downhole conditions (300 psi, 200°F, 10 days) to analyze rock-filtrate interactions. Advanced techniques like X-ray diffraction, scanning electron microscopy, and nuclear magnetic spectrometry were employed to observe changes in pore structure. The results revealed significant alterations in the internal topography of the rocks, particularly in pore size distribution. Berea types showed more pronounced changes compared to Bandera Brown and Parker. Although total porosity remained largely unchanged, a general decrease in pore throat radius and permeability was observed, affecting flow characteristics and transforming the porous systems of Berea Buff and Parker from macro-porous to meso-porous.

Sung et al. (2021) studied the impact of oil-based mud filtrate invasion on NMR response characteristics in low-porosity, low-permeability sandstone gas formations in the East China Sea. They observed that the invasion caused the relaxation time of the NMR T_2 spectrum for the free fluid to shift towards slower relaxation times, significantly affecting petrophysical evaluations. To address this, they proposed a three-step method to correct the NMR T_2 spectrum for the effects of invasion. This method involved analyzing differences in T_2 spectrum morphology between oil-based and water-based mud wells, categorizing rocks based on pore structure, and using a

multidimensional matrix method to establish corrected T_2 spectra. The corrected spectra effectively eliminated the influence of oil-based mud filtrate, thereby enhancing the accuracy of NMR logging in permeability calculations.

Duan et al. (2023) proposed a novel technique to evaluate mudcake pore structure, addressing the shortcomings of traditional macro- and micro-evaluation methods used in drilling engineering. They integrated Nuclear Magnetic Resonance (NMR) T_2 relaxation tests with high-pressure mercury injection, fluid isotope tracing, dynamic/static filtration experiments, core permeability tests, and particle-size distribution analyses. Their approach allows for detailed micron-scale evaluation of mudcake formed by static and dynamic filtration, effectively analyzing both the outer mudcake and the intruded core.

Bageri et al. (2024) investigated the effect of hematite concentration on the properties of water-based drilling fluid, addressing challenges associated with drilling deep wells in high-pressure and high-temperature (HPHT) conditions. The study involved adding three different doses of hematite to a constant drilling fluid formulation and evaluating key properties such as density, viscosity, filtration, and filter cake characteristics. Results showed a proportional relationship between hematite concentration and apparent viscosity, plastic viscosity, and yield point, both before and after aging in a hot rolling oven. The yield point-to-plastic viscosity (YP/PV) ratio decreased as hematite concentration increased, while gel strengths at 10 seconds and 10 minutes increased with higher hematite levels. Additionally, the thinness of the filter cake, filtration volume, and filter cake permeability improved with increased hematite concentration, whereas filter cake porosity remained nearly constant. Overall, the study established several correlations between hematite dosage and the examined drilling fluid properties.

Gao et al. (2024) examined the challenges of identifying formation fluids using NMR logging after oil-based mud (OBM) invasion, particularly in heterogeneous conglomerate formations of the Junggar Basin. The study involved core experiments simulating OBM invasion into water- and hydrocarbon-bearing formations under four conditions: water-saturated, OBM displacing water, oil-saturated, and OBM displacing oil. The results showed that OBM invasion alters the NMR T_2 spectrum, with OBM filtrate primarily influencing the movable peak in water layers, while oil layers exhibited a three-peak distribution. The study introduced a fluid identification factor based on two T_2 cutoffs and the T_2 geometric average. Field application verified the method's effectiveness in distinguishing oil layers under OBM drilling, aiding reservoir identification and development.

Jichuan et al. (2024) explored the effects of drilling fluid invasion on pore structure and permeability in the carbonate reservoir of the Dengying Formation, located in the Penglai Gas Field, Sichuan, China, at depths exceeding 6000 meters and temperatures over 150°C. The study highlighted the complex nature of the reservoir's structures. Through core sample testing using NMR and gas permeability measurements, the research found that drilling fluid invasion caused significant blockages in the pores. Additionally, the researchers developed a permeability estimation model that predicted permeability changes.

Suo et al. (2024) investigated the wellbore instability challenges in laminated shale formations, where high clay mineral content and weak planes increase the risk of borehole collapse due to drilling fluid invasion. The study involved displacement experiments and nuclear magnetic scanning to assess fluid invasion depths over time. Key findings revealed that as intrusion time increased, pore sizes expanded, leading to micro-cracks and potential rock failure. A new mathematical model was developed to account for both end-face and striated fluid intrusion,

accurately predicting invasion depths based on time and layer characteristics. The results showed that invasion depth increased logarithmically with time, slowed in later stages, and decreased with a greater number of laminated layers.

From previous studies, it is evident that the current research lacks a standardized approach to establish a consistent connection between drilling fluid properties and their impact on NMR response. Existing literature falls short in discussing T_2 bulk fluid characteristics, potentially leading to discrepancies in their results and NMR response. This study aims to address how drilling fluid characteristics, host rock properties, and additives/particles affect NMR responses, particularly concerning pore size distribution and relaxation times.

CHAPTER 3

METHODOLOGY

3.1 Background Statement

The study was meticulously designed to investigate the impact of drilling fluid bulk relaxation on nuclear magnetic resonance (NMR) interpretation, which is critical for accurate formation evaluation in the oil and gas sector. To achieve this objective, we employed three distinct approaches to vary the bulk relaxation properties of the drilling fluid. First, we altered the concentration of viscosifier to assess how different levels of viscosity influence the bulk relaxation characteristics of the fluid. By systematically varying these concentrations, we aimed to establish a clear correlation between viscosity and NMR T_2 responses, providing insights into the rheological behavior of the drilling fluid under varying conditions.

Second, we adjusted the percentage of perlite in the drilling fluid formulation to examine its influence on the fluid's physical properties and its subsequent effect on NMR T_2 measurements. The inclusion of perlite, known for its lightweight and insulating properties, allows for better control over fluid dynamics and may significantly affect NMR signal responses during invasion scenarios.

Third, we varied the concentration of iron-rich additives (hematite) to investigate their impact on surface relaxivity and overall NMR measurements. Iron additives are crucial in enhancing the density of the drilling fluid, and understanding their influence on NMR responses is essential for accurate petrophysical evaluations, particularly in complex reservoir conditions.

Lastly, we validate our experimental procedure of using rock as a filter during the filtration experiment against the common practice in the literature, where a standard filter paper is used instead. This comparison was designed to elucidate the differences in NMR responses produced

by these two methodologies, highlighting how experimental design can significantly influence the interpretation of NMR data.

This systematic and multifaceted approach was crucial for unraveling the influence of drilling fluid invasion on NMR responses. By varying key parameters and employing different experimental setups, we aimed to provide a comprehensive understanding of how bulk relaxation mechanisms in drilling fluids affect the accuracy and reliability of NMR interpretations. Ultimately, this investigation seeks to enhance our understanding of the complex interactions occurring during fluid invasion, improving the interpretive framework for NMR data in the context of formation evaluation and reservoir characterization, which is vital for optimizing drilling strategies and ensuring successful hydrocarbon extraction.

3.2 Desk Study

The desk review necessitates the review of key pieces of literature, both written and unpublished, relevant to the research project. Our work builds on the work of (Duan et al., 2023; Bageri et al., 2021; Hany et al., 2021; and Adebayo et al., 2020).

3.3 Laboratory Procedures

This section aims to discuss the process used in the laboratory to achieve the stated objectives in this research.

3.3.1 Mud-Filtration Experiment

a. The fluid loss experiment procedure using rock as filter

A specialized filtration loss setup (Figure 3.1) was specifically built to house roughly 5 cm long rock samples. This device consists of a stainless-steel cylinder equipped with two removeable end caps featuring flow-through and pressure ports for fluid flow and pressure monitoring. The test

cell's outlet was subjected to a backpressure of 200 psi. Prior to testing, the rock sample was saturated with a 3% potassium chloride solution (KCl) and inserted into the cell, secured within a hollow rubber sleeve. The drilling mud prepared was then transferred onto the core sample's surface, forming a vertical column. Subsequently, heat was gradually applied until reaching temperature of 66 °C, and gas (i.e. nitrogen) was introduced over the column of the drilling fluid to generate a hydrostatic pressure across the sample which is 500 psi, resulting in a differential pressure of 300 psi. Pressure and temperature were closely monitored and maintained till the test completion. Concurrently, at the outlet the fluid loss was observed. The experiment concluded after 30 minutes, adhering to normal API time for fluid loss tests (API, 2003). Following the test, the temperature and pressure were gradually reduced, and the rock was delicately retrieved to preserve the deposited filter cake. The sleeve ensured filtration loss occurred solely in the axial direction, facilitating filter cake deposition on the sample's cross-sectional area. Once retrieved, the sample, featuring the filter cake on top, was promptly transferred to a sealed glass vessel for NMR relaxometry experiments. The filter cake formed on the rock surface was carefully removed using a scraper upon conclusion of the NMR measurements. Subsequently, the sample's surface area was carefully cleaned with identical brine to eliminate residual filter cake, and NMR measurements were repeated. The T_2 bulk relaxation of the filtrate collected during the experiment was also measured.

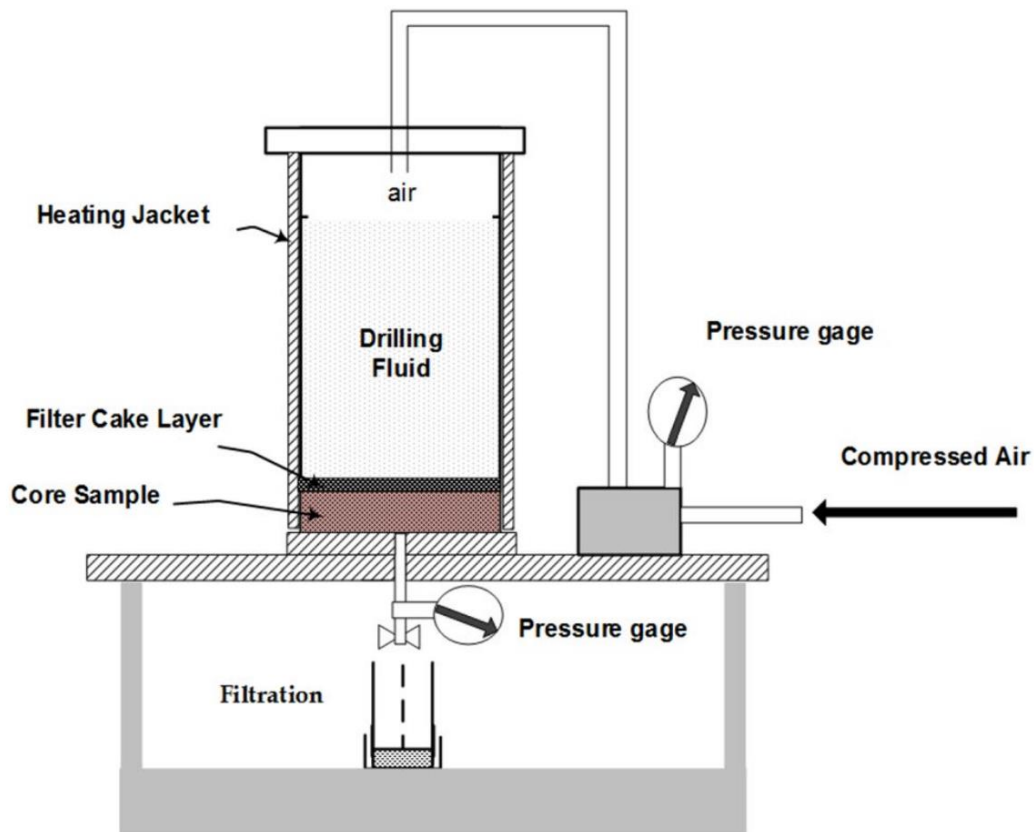


Figure 3.1: A high-temperature high pressure fluid loss system using rock as filter.

b. Filter Paper fluid-loss experiment

The HPHT filtration properties of drilling fluids were evaluated under simulated downhole conditions using an HPHT filter press (Figure 3.2). To perform the test, the drilling mud prepared was transferred onto the filter paper's surface, forming a vertical column. Subsequently, heat was transferred onto the filter paper's surface, forming a vertical column. Subsequently, heat was gradually applied until reaching temperature of 66 °C, and gas (i.e. nitrogen) was introduced over the column of the drilling fluid to generate a hydrostatic pressure across the sample which is 500 psi, resulting in a differential pressure of 300 psi. Pressure and temperature were closely monitored and maintained till the test completion. Concurrently, at the outlet the fluid loss was observed. The experiment concluded after 30 minutes, adhering to normal API time for fluid loss tests (API,

2003). Following the test, the temperature and pressure were gradually reduced, and the filter paper was delicately retrieved to preserve the deposited filter cake.



Figure 3.2: *A high-temperature high pressure fluid loss system using filter paper*

3.3.2 Core Flooding Test

The core flooding system used in this study, as illustrated in Figure 3.3, consists of several key components: an injection pump to introduce fluid into the core, an overburden pressure pump and backpressure regulator to control pressure, an accumulator for fluid storage, a core holder to secure the core within the system, a sample collector for gathering filtrate, and a data acquisition and recording system. The permeability of the core samples was assessed at various flow rates under single-phase and steady-state flow conditions using KCl brine, employing a liquid permeability

measurement device. Darcy's single-phase flow equation was applied to calculate liquid permeability. Following the permeability measurements, two pore volumes (PV) of filtrate collected from the filter press fluid loss experiment (as shown in Figure 3.2) were injected into the cores at a rate of 0.5 cc/min. A backpressure of 1000 psi was maintained at the core's outlet to ensure a consistent pore pressure of 1000 psi. After injection, both inlet and outlet valves were closed for 18 hours to allow the injection process to stabilize. After the conclusion of the experiment, the cores were then carefully extracted, and the rock samples were subjected to NMR measurements.

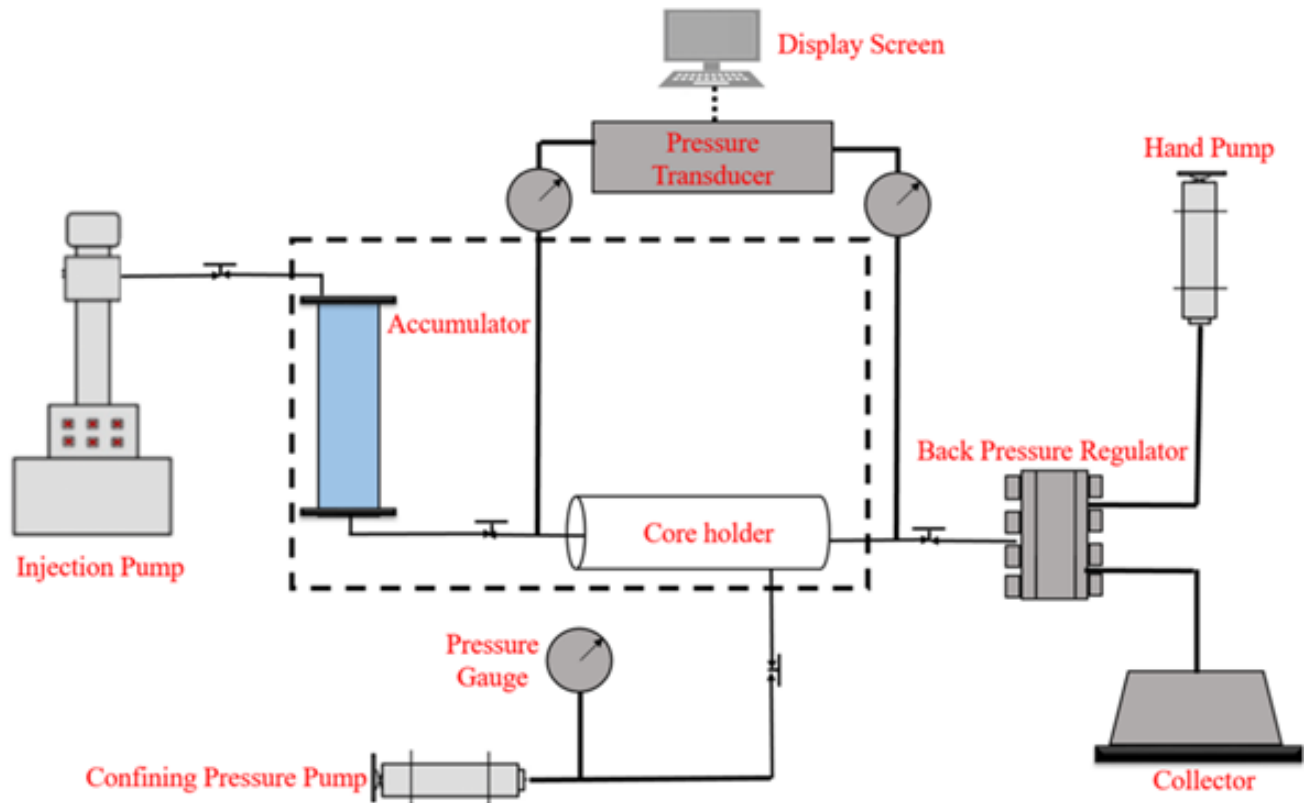


Figure 3.3: Core flooding Schematic Diagram (After Jaber et al., 2022)

3.3.3 SEM – EDX Analysis

The morphology and particle shapes of hematite and perlite were determined using a scanning electron microscope equipped with an energy dispersive X-ray analyzer (SEM-EDX) as shown in Figure 3.4. Specifically, we employed a Field Emission Scanning Electron Microscope (FE-SEM), which offers ultra-high-resolution imaging capabilities, achieving a remarkable resolution of 1 nm at 15 kV. This advanced tool was instrumental in examining the additive powders, allowing us to capture detailed images of their surface features. By utilizing beam deceleration, we minimized charging effects, which is crucial for obtaining clear images of non-conductive samples like perlite. The FE-SEM provided both secondary electron imaging to analyze surface topography and backscattered electron imaging for assessing un-etched surfaces. Coupled with energy dispersive X-ray spectroscopy (EDS), we conducted microchemical analysis to identify the composition of various microstructural features, including precipitates and phases within the hematite and perlite samples. We used the Gemini SEM 450 model, which is equipped with a field emission gun and a 5-axis stage, enabling us to perform multi-quadrant and mixed imaging. This setup, along with various detectors and an infrared camera, facilitated comprehensive high-resolution material characterization of hematite and perlite, enhancing our understanding of their properties and potential applications.



Figure 3.4: SEM – EDX device

3.3.4 Micro CT Scan

The Xradia (Zeiss) Versa XRM-500 is a high-resolution 3D X-ray microscopy system designed for non-destructive imaging and characterization of materials. It uses X-rays to penetrate the sample and capture detailed internal structures without the need for physical sectioning. The versatility of the Versa series allows for imaging under various conditions, such as varying magnifications and energy levels, making it an ideal choice for analyzing the extent of hematite invasion after filtration experiment in this study.

The PerGeos software, formerly developed by FEI (now under ThermoFisher Scientific), is an advanced tool for digital rock physics and image processing. It supports a wide range of capabilities, including artifact correction to address issues such as noise, beam hardening, and ring

artifacts that can distort the final image. PerGeos also features sophisticated filtering techniques to enhance image quality, facilitating the identification of different phases or materials within the sample.

For segmentation, PerGeos utilizes machine learning algorithms, thresholding, watershed techniques, and other advanced methods to differentiate various features in the images. The software allows users to create 2D and 3D visualizations with customizable color maps and rendering options, providing a clearer view of the internal structure. This is particularly useful for analyzing pore spaces, fractures, and mineral distributions in geological samples. Furthermore, the software's analytical tools enable quantitative measurements of features like porosity, grain size, and connectivity, making it an essential tool for digital rock analysis and petrophysical studies.



Figure 3.5: Micro CT Scanner

3.3.5 Laser Particle Size Analyzer

Laser Particle Size Analysis (LPSA) is a particle size measurement technique first developed in the 1970s (Agrawal et al., 1991). It has since become a standard test method, referenced in both ASTM (B822-20) and ISO (13322-1:2014) guidelines. In this study, particle size distribution was determined using the wet dispersion unit of the ANALYSETTE 22 Nano Tec plus instrument, equipped with an aqueous liquid module (ALM). A schematic representation of an LPSA device is provided in Figure 3.6. The technique works by measuring the particle size distribution of loose powders through the diffraction patterns generated by laser light. It operates on the principle that particles of specific sizes diffract light at predictable angles, with the diffraction angle inversely related to the particle diameter (Di Stefano et al., 2010). For PSD measurement, a parallel beam of monochromatic light with a wavelength of 750 nm is focused using Fourier optics and directed through the sample cell. The diffracted light is then detected by an array of 126 photodiode sensors positioned at an angle of roughly 35° from the optical axis (Beckman Coulter, 2011), allowing for the measurement of the size of the suspended particles.

The ANALYSETTE 22 Nano Tec plus instrument's aqueous liquid module is designed for analyzing loose particles, which eliminated the need for special sample preparation for the additives in this study. The module was filled with deionized water to prevent any contamination or particle charging. If a sample contained organic material, it was removed beforehand using hydrogen peroxide, as organic matter in LPSA analysis can shift the particle size distribution (PSD) towards larger particles due to adhesion and clumping (Allen and Thornley, 2004). Ultrasonic treatment was not applied before or during analysis since it can bias the results towards finer particles by potentially breaking them apart (Ballard and Beare, 2013). Samples were added gradually to the loading chamber of the aqueous liquid module until the obscuration level,

indicating the proportion of laser light blocked by particles, reached 20% (though Beckman Coulter (2011) recommends a range of 8-12%). The particles were circulated in a closed loop to ensure a representative measurement of the spherical particles (as depicted in Figure 3.6). Each sample underwent three consecutive 1-minute analyses (for a total of 3 minutes). Results were reported as volumetric percentages of the sample within each size range. The size ranges, or bins, were distributed logarithmically from 0.5 μm to 2000 μm , with the logarithmic midpoint of each bin used to calculate PSD descriptors.

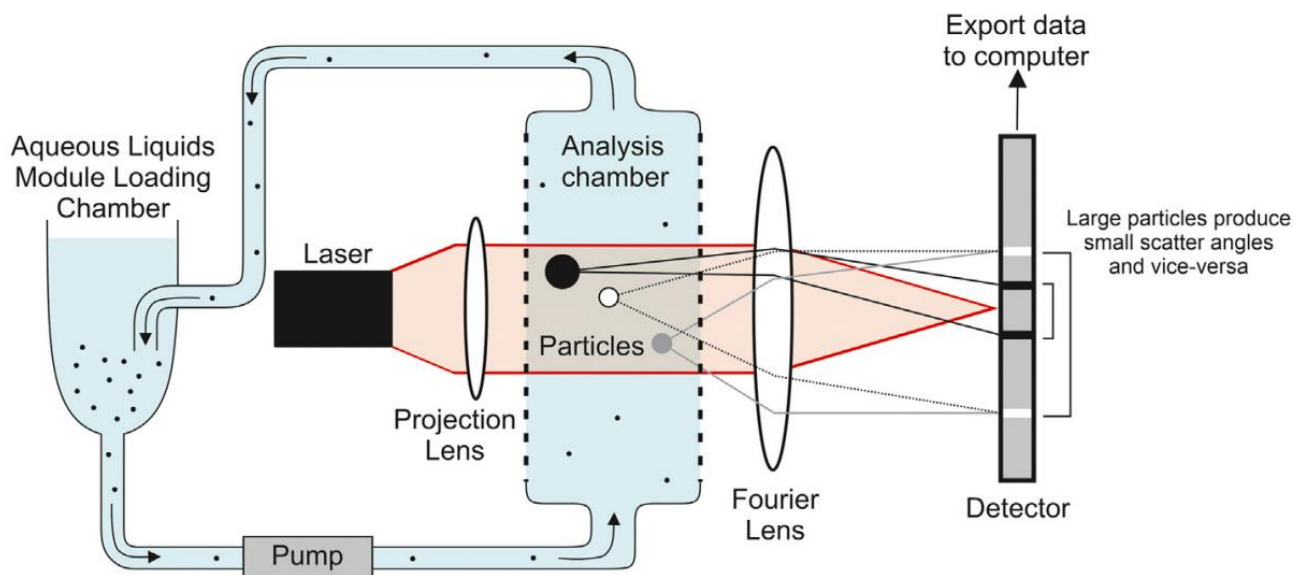


Figure 3.6: A schematic of an LPSA device shows particles suspended in de-ionized water circulating around the observation cell. As laser light passes through, particles scatter the light at angles measured by the detector, with larger particles scattering at smaller angles. (Adapted from Ballard and Beare, 2013; Beckman Coulter, 2011.)

3.3.6 NMR theory and set-up

In NMR relaxometry, T_2 relaxation refers to the transverse relaxation time and it is obtained utilizing a pulse sequence known as CPMG, named after its inventors (Carr and Purcell, 1954; Meiboom and Gill, 1958). The NMR transverse relaxation time (T_2) comprises three main terms: bulk relaxation time (T_{2B}), surface relaxation time (T_{2S}), and diffusion relaxation time (T_{2D}), according to the following relationship (Coates et al., 2009; Dunn et al., 2002):

$$\frac{1}{T_2} = \frac{1}{T_{2B}} + \frac{1}{T_{2S}} + \frac{1}{T_{2D}} \quad (1)$$

In geological materials like sedimentary rocks, surface-controlled (fast) diffusion processes primarily govern NMR relaxation (Brownstein and Tarr, 1979). Moreover, T_{2B} is relatively high for most original reservoir fluids, so the term $1/T_{2B}$ can be considered negligible. In the above conditions, the overall T_2 is dominated by the surface relaxation mechanism which can be expressed (Brownstein and Tarr, 1979; Kenyon, 1997; Kleinberg et al., 1994) as shown in equation (2):

$$\frac{1}{T_2} \approx \rho \left(\frac{S}{V} \right) \quad (2)$$

where, ρ is the surface relaxivity, and S/V is the surface area of pore–volume ratio. The surface relaxivity refers to the efficiency with which pore surfaces in a rock induce relaxation of fluid magnetization, affecting NMR relaxation times.

The mean- T_2 permeability model was also used here to estimate permeability, as it assumes a strong correlation between porosity, pore-body and pore-throat size, and pore connectivity (Timur, 1969; Seevers, 1966; Ahmed et al., 1991). In this model, permeability is expressed as:

$$k_{SDR} = C \times T_{2gm}^2 \times \phi^4 \quad (3)$$

Here, ϕ represents the NMR effective porosity (MPHI), T_{2gm} denotes the geometric mean of the T_2 distribution, and C is a formation-dependent parameter.

Consequently, the NMR reading gives information about pore size distribution. Previous works often overlooked the effects of drilling fluid bulk relaxation on NMR interpretation of the invaded reservoir rocks. Bulk relaxation refers to the fluid's intrinsic relaxation properties, which are influenced by factors such as viscosity. The drilling fluid can be very viscous at times, and as its viscosity increases, T_{2B} decreases. As a result, T_{2B} may no longer be neglected in relation to the surface relaxation effects. This means that NMR T_{2S} measurements may no longer solely reflect the pore structure, as Equation (2) becomes less accurate, potentially distorting pore size interpretations. We investigate here whether or not and to what extent replacing the original fluid with drilling fluid (which has a lower T_{2B}) can impact the T_2 interpretation for pore size distribution.

A GeoSpec rock core analyzer (figure 3.7), operating at a low magnetic field of 0.05 Tesla (2 MHz) and manufactured by Oxford Instruments, was employed to measure the T_2 relaxation times of the samples. The Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence was used with augmented scanning constraints, including a Tau value of 0.10 ms, a signal-to-noise ratio of 200.0, and a recycle delay of 11,250.0 ms. Each scan took an average of 10 minutes for each core. The NMR device featured an accessible software package, Green Imaging Technology, which facilitated the data enhancement. NMR measurements were observed at three distinct phases: (1) an initial NMR profile (with rock and the original 3% KCl brine only), (2) an NMR profile post filter cake deposition and mud invasion (rock + filter cake + mud filtrate), and (3) an NMR profile after the removal of the filter cake (rock + mud filtrate). Each sample was first saturated with 3% KCl brine, and the baseline NMR T_2 profile was recorded. To measure bulk fluid relaxation for

the drilling fluid and filtrate, the fluid was placed in a large container to eliminate surface relaxation effects, then exposed to a homogeneous magnetic field and subjected to a CPMG pulse sequence.

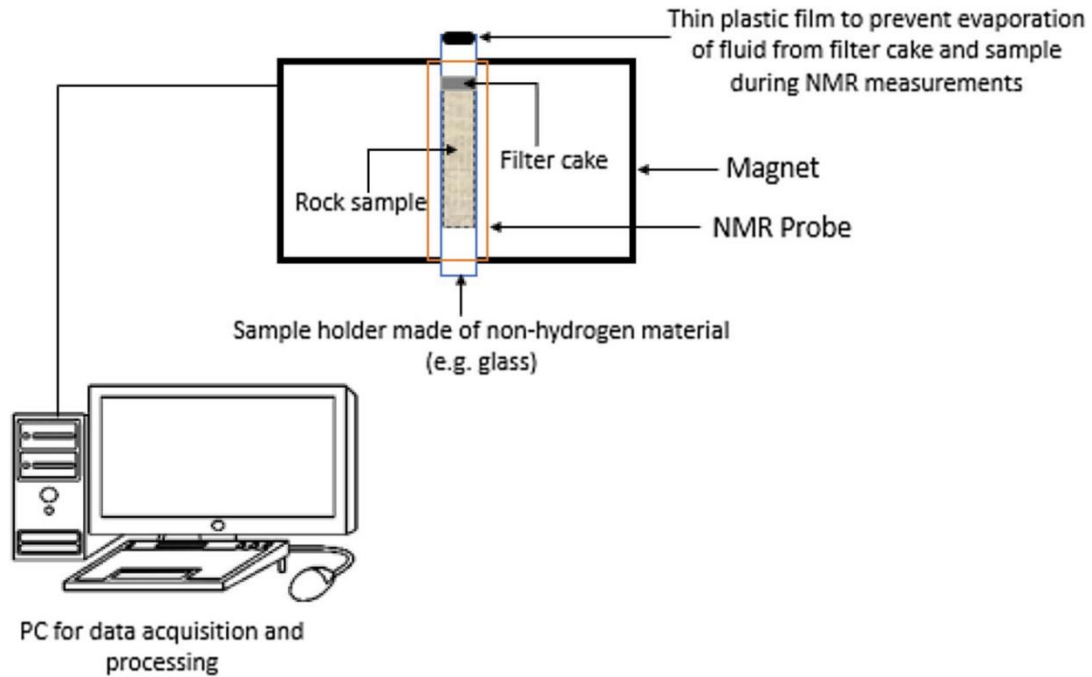


Figure 3.7: NMR setup for rock sample with filter cake on it

3.4 Evaluation Procedure

The evaluation procedure is designed to study how the NMR T_2 in the rock is altered due to invasion by drilling fluids focusing on the influence of the drilling fluid bulk relaxations, due to variable particle invasion and XC polymer additive. The following point summarizes the experimental procedures:

1. **Testing the basic parameters.** The drilling fluid was formulated and evaluated for its bulk relaxation properties, which served as a reference for subsequent NMR measurements. Core samples were then analyzed for essential characteristics, including length, diameter, weight, porosity, and permeability.

2. **Saturating cores with the formation water.** Each pair of cores was labeled according to permeability: high-permeability samples were labeled from cores A to H, while low-permeability samples were labeled from cores I to N. The cores were oil-washed, dried, and vacuumed before being saturated with 3% KCl brine to simulate formation water conditions.
3. **Obtaining T_2 base measurement.** NMR- T_2 tests was conducted on both classes of limestone samples to measure the T_2 relaxation times of the rocks saturated with the initial formation water, providing baseline data.
4. **Performing static filtration experiments of drilling fluids using rock as filter.** Static filtration experiments were carried out with the drilling fluid to form a relatively complete mudcake on the rock surfaces. The objective was to assess how the mud invasion process affects the NMR response of the samples.
5. **Recording filtrate volume collection with time:** During the filtration experiments, the volume of filtrate released was measured over time. This data helped in understanding the dynamics of fluid invasion and mudcake formation.
6. **Measuring the thickness of the outer mudcake:** The thickness of the filter cake formed after the filtration experiment was measured using a high-resolution caliper to quantify the extent of mud deposition on the core surface.
7. **Obtaining T_2 measurement after filtration experiment.** NMR measurements were conducted in three stages: first, a baseline NMR profile of the rock saturated with 3% KCl brine; second, an NMR profile after filter cake deposition and mud invasion (including rock, filter cake, and mud filtrate); and third, an NMR profile after the filter cake had been removed, leaving only the mud filtrate in the rock. Additionally, NMR measurements were

performed on the filtrate collected during the filtration experiments to analyze changes in fluid relaxation properties.

- 8. Static filtration experiments of drilling fluids using standard filter paper.** Static filtration experiments were conducted with the drilling fluid to form a mudcake on the surface of standard drilling filter paper. The objective was to observe how this experimental setup influences the NMR T_2 response compared to using rock samples for filtration.
- 9. Collection of filtrates released during the experiment.** The filtrate produced during the filtration experiments was collected in a flask.
- 10. Injecting filtrate into the core samples:** The collected filtrate was then injected into the rock samples using a core flooding setup to simulate field conditions. This step aimed to validate the effectiveness of the rock-fluid system in simulating field conditions as opposed to a filter paper set - up.
- 11. Obtaining T_2 measurement after filtration experiment.** NMR measurements were conducted in two stages: first, a baseline NMR profile of the rock saturated with 3% KCl brine; second, an NMR profile after the injection of the filtrate in the rock to evaluate the effects of fluid invasion on the relaxation properties.

3.5 Drilling Fluids

For the first approach, we focused on assessing the impact of XC polymer (viscosifier) additive on bulk T_2 shifts. In this phase, the drilling fluid was a 3% KCl solution with varying concentrations of XC polymer additive, as detailed in Table 3.1. At this stage, we did not add any particles to isolate the effect of the XC polymer on the NMR response from that of the particle additive. In the second approach, the viscosity and thus the bulk relaxation of the drilling mud was varied by changing the concentration of the additive particle, specifically perlite. In this case, particles were

introduced into the drilling fluid (Table 3.2). The viscosifier concentration was maintained at 0.5 lb, which is the most frequently used concentration in drilling fluid formulation (Bageri et al., 2021; Abdulrauf et al., 2020). In the third approach, the bulk relaxation of the drilling mud was varied by changing the concentration of the additive particle, specifically iron. In this case, iron additives were introduced into the drilling fluid (Table 3.3). For the last phase, we validated the effectiveness of our experimental procedure using rock as filter as compared to the common procedure of using filter paper in the literature. The drilling fluid used in this phase is same as shown in table 3.2.

Table 3.1. *Drilling fluid formulation with variable XC polymer concentration*

Chemical composition	Unit	Baseline Brine #1.1 Description	Drilling Fluid #1.2	Drilling Fluid #1.3	Drilling Fluid #1.4
Water	bbbl	0.60	0.60	0.60	0.60
XC polymer	lb	0.00	0.50	2.00	5.00
KCl	lb	30.0	30.0	30.0	30.0

Table 3.2. *Drilling fluid formulation with variable additive particles (Perlite) concentration*

Chemical composition	Unit	Baseline Drilling Fluid #2.1 Description	Drilling Fluid #2.2
Water	bbbl	0.60	0.60
Bentonite	lb	5	5
XC polymer	lb	0.50	0.50
KCl	lb	30.0	30.0
Barite	lb	100.0	100.0
Additive (Perlite)	lb	0.00	2.50

Table 3.3. *Drilling fluid formulation with variable additive particles (Hematite) concentration*

Chemical composition	Unit	Baseline Drilling Fluid #3.1 Description	Drilling Fluid #3.2
Water	bbbl	0.60	0.60
Bentonite	lb	5	5
XC polymer	lb	0.50	0.50
KCl	lb	30.0	30.0
Barite	lb	100.0	100.0
Additive (Hematite)	lb	0.00	80.0

3.5.1 Drilling Fluid Characterization

The structural characteristics of the perlite additive employed in this study were thoroughly analyzed using particle size distribution (PSD) measurements and scanning electron microscopy (SEM). As illustrated in Figure 3.8, the PSD results obtained from the wet dispersion unit, ANALYSETTE 22 Nano Tec plus, revealed a median particle size (D50) of 62.39 μm . The particle size range extended from 20.30 μm (D10) to 122.68 μm (D90), indicating a broad distribution of particle sizes. SEM imaging (Figure 3.9), demonstrated that the perlite particles exhibited a distinct flake-like morphology. Additionally, chemical composition analysis via SEM-EDS, summarized in Table 3.4, confirmed that silica constitutes approximately 65–80% of the perlite's overall composition.

In parallel, the particle size distribution (PSD) of the hematite used in this investigation is shown in Figure 3.10. The analysis revealed a median particle size (D50) of 9.29 μm , with a size range from 1.45 μm (D10) to 33.00 μm (D90), reflecting a wide distribution. High-resolution SEM images, presented in Figure 3.11, indicated that the hematite particles exhibit an irregular shape with sharp edges. Energy-dispersive X-ray spectroscopy (EDS) analysis of the hematite, summarized in Table 3.5, revealed that iron (Fe) is the primary component, constituting 60 wt%. Additional elements identified included oxygen (O) at 20.9 wt%, carbon (C) at 17.9 wt%, and calcium (Ca) at 0.5 wt%.

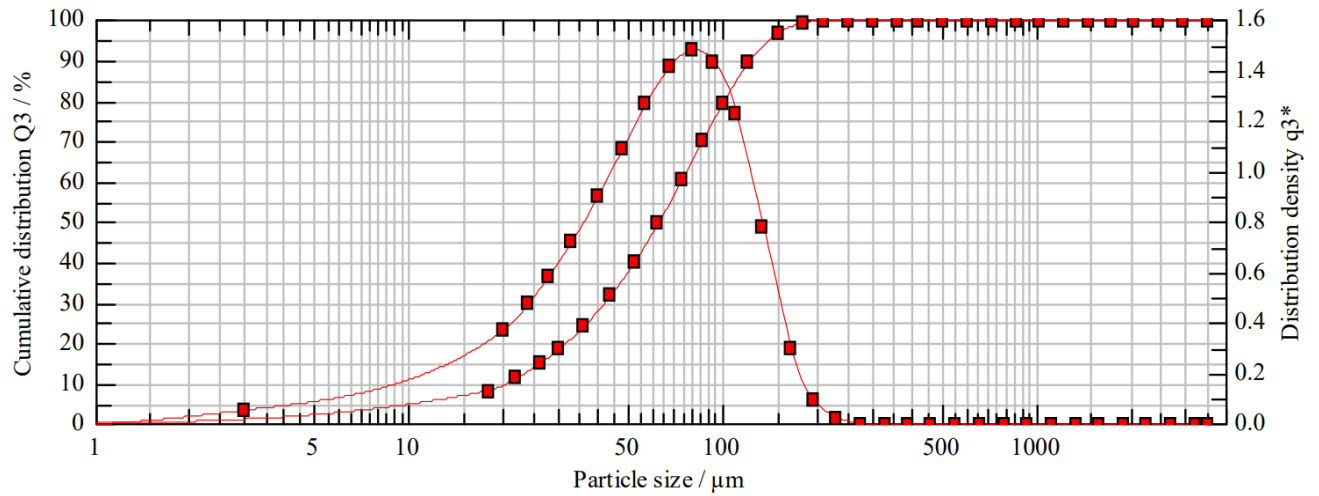


Figure 3.8: Particle size distribution perlite

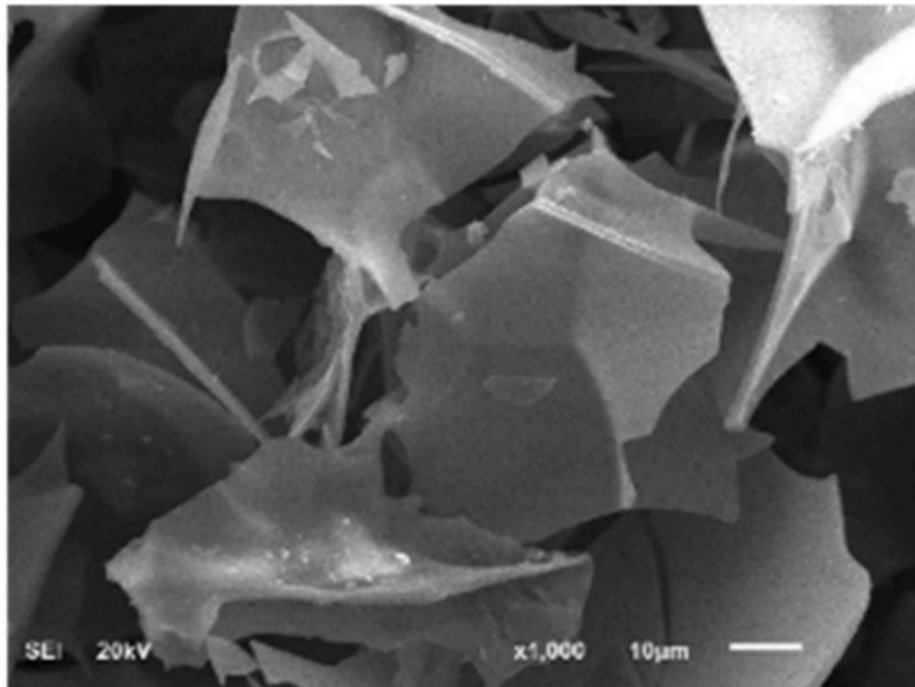


Figure 3.9: High – resolution SEM for perlite particles

Table 3.4. Perlite particles composition (SEM-ESD)

Composition	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O
Range (wt%)	65 - 80	10 - 18	2 - 5	2 - 6

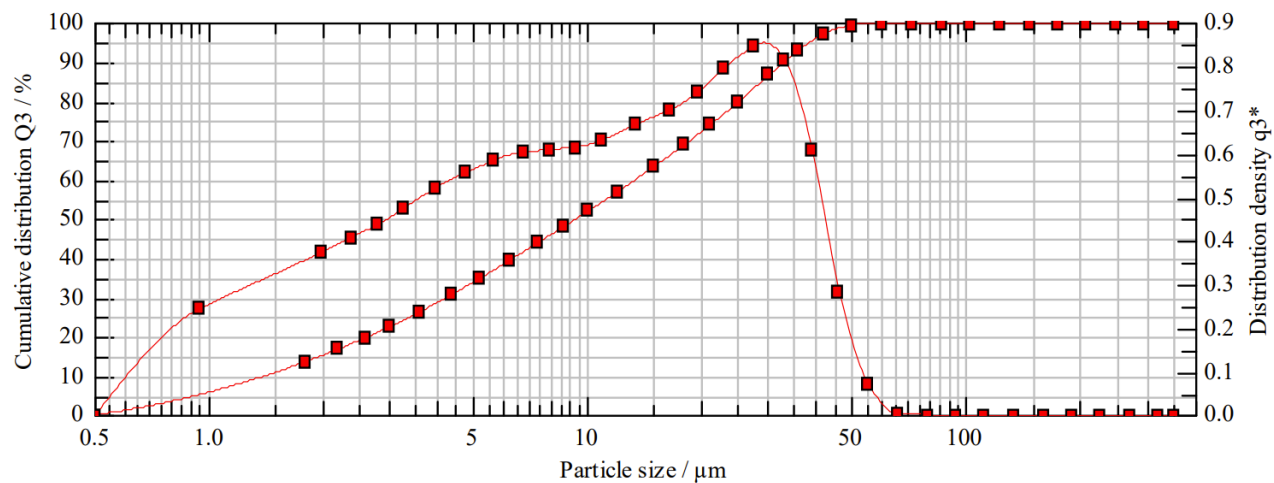


Figure 3.10: Particle size distribution hematite

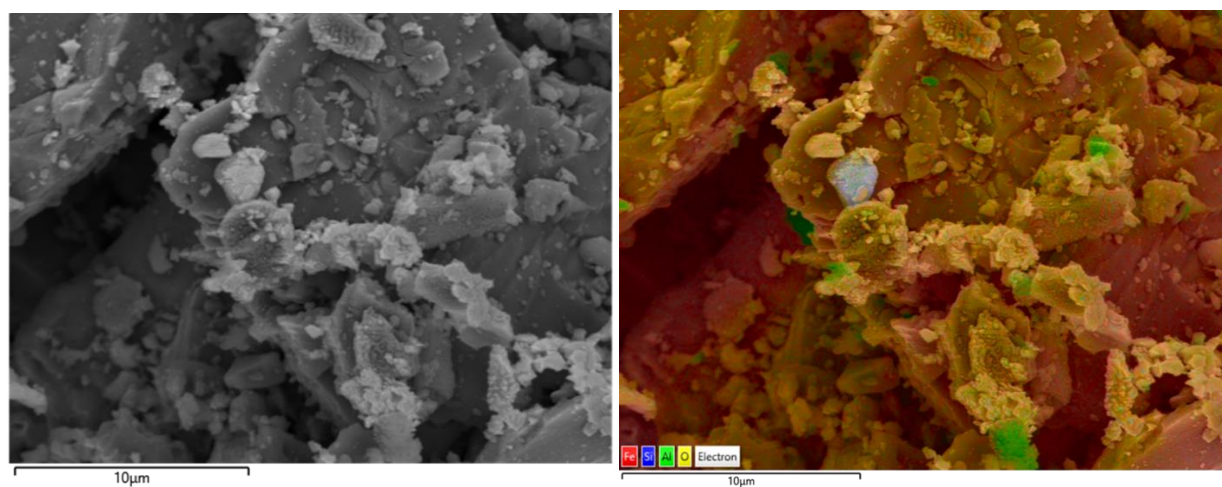


Figure 3.11: High – resolution SEM for hematite particles

Table 3.5. Hematite particles composition (SEM-ESD)

Composition	Fe	O	Si	Al
Range (wt%)	75	23	0.7	0.6

3.6 Rock samples

In this study, rock samples were categorized into two groups based on their permeability. The first group consisted of permeable rocks with larger pores, while the second group comprised tight rocks with microporosity. For the permeable group, which had a permeability range of 90 to 110 mD, an Indiana limestone sample from the Salem Formation, sourced from Kocurek Industries, Inc., was cut into nine identical pieces, each measuring approximately 4.2 to 5 cm in length. Similarly, the Buda and Leuder core samples, sourced from Kocurek Industries, representing low-permeability carbonate rocks with permeabilities ranging from 1 to 2 mD, were originally sized at 8 inches (20.32 cm) by 1.5 inches (3.8 cm) and were divided into four identical pieces of the same length range for each sample. The high-permeability samples were designated as A, B, C, D, E, F, G, and H, while the low-permeability samples were labeled I, J, K, L, M, and N as detailed in Table 3.6.

To test the impact of variable viscosifier concentrations on the NMR signal when invading rocks, we used samples characterized by similar pore structures (sister cores from a given group) for each viscosifier concentration. NMR measurements were employed to ensure that all sister samples exhibited nearly identical T_2 distributions. Figures 3.12 – 3.15 show the T_2 distributions for sister/identical samples from each of the groups (low versus high permeability) used with different drilling fluids (shown in Tables 3.1 - 3.3). Samples A, B, and C were used to investigate the impact of increasing drilling fluid viscosity on the NMR signal for permeable rocks, while samples I and J were used to study the same effect in tight rocks. Each sample was dedicated to a specific viscosity during the mud filtration experiments, as a new sample was required for each test to avoid complications related to sample cleaning. For the second test, samples D and E were utilized to assess the impact of added perlite particles in the high-permeability group, whereas samples K and L were used to address the same issue in the low-permeability group. For the third test, samples F

and G were utilized to validate the effectiveness of our experimental procedure of using rock as filter rather than filter paper used in literatures. Lastly, sample H was utilized to assess the impact of added iron particles in the high-permeability group, whereas samples M and N were used to address this issue in the low-permeability group.

Table 3.6. *Rock samples used in this study*

Sample ID	Sample Type	Diameter (cm)	Length (cm)	Porosity (%)
Sample A	Indiana	3.80	5.00	19.60
Sample B	Indiana	3.80	5.00	20.60
Sample C	Indiana	3.80	5.00	20.60
Sample D	Indiana	3.80	4.60	20.20
Sample E	Indiana	3.80	4.52	20.60
Sample F	Indiana	3.80	5.00	19.00
Sample G	Indiana	3.80	5.00	19.30
Sample H	Indiana	3.80	5.00	20.20
Sample I	Buda	3.80	5.00	16.80
Sample J	Buda	3.80	5.00	16.70
Sample K	Buda	3.80	4.20	16.60
Sample L	Buda	3.80	5.04	16.50
Sample M	Leuder	3.80	4.20	13.82
Sample N	Leuder	3.80	5.04	13.92

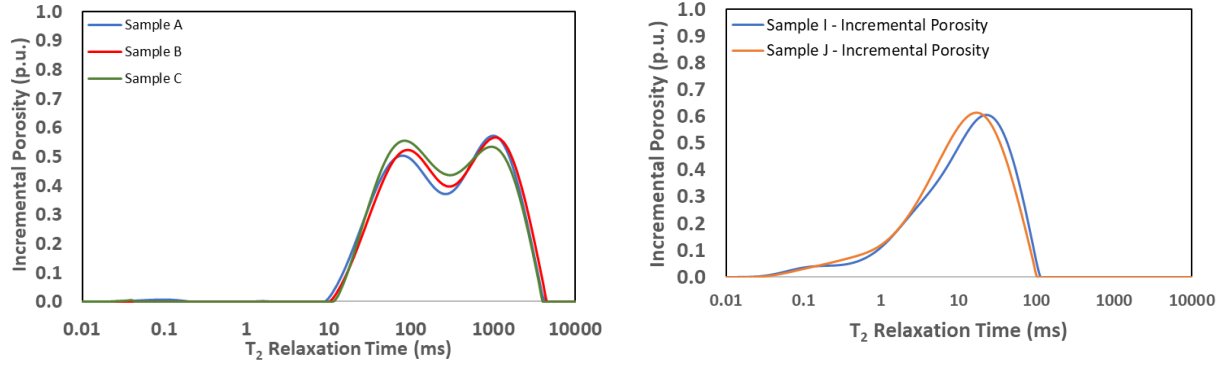


Figure 3.12: NMR T_2 relaxation for samples used for the XC polymer additive. Continuous lines show the probability distribution function (PDF) of the pores. Left (highly permeable), Right (Low permeability)

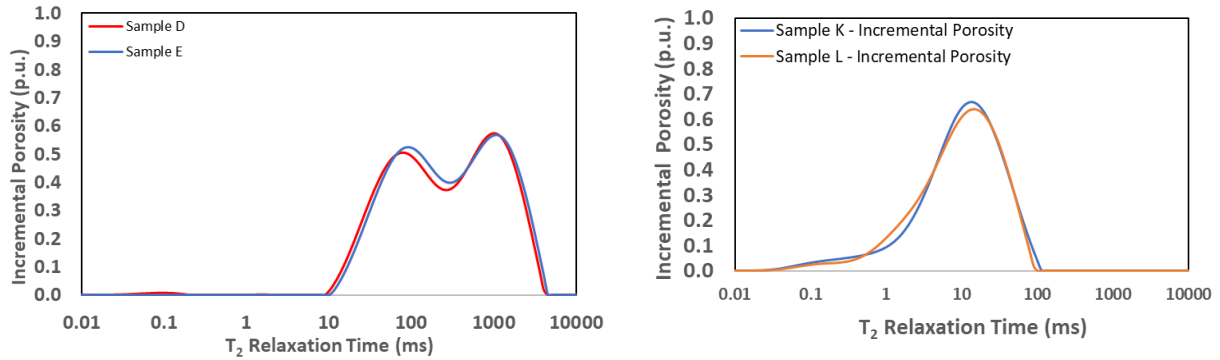


Figure 3.13: NMR T_2 relaxation for the samples used for the perlite additive. Continuous lines show the probability distribution function (PDF) of the pores. Left (highly permeable), Right (Low permeability)

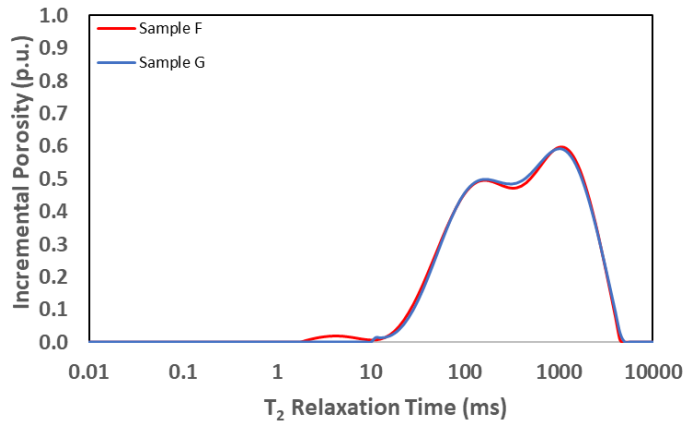


Figure 3.14: NMR T_2 relaxation for the samples used for the static rock-fluid interaction using filter paper. Continuous lines show the probability distribution function (PDF) of the pores. The samples are the permeable samples.

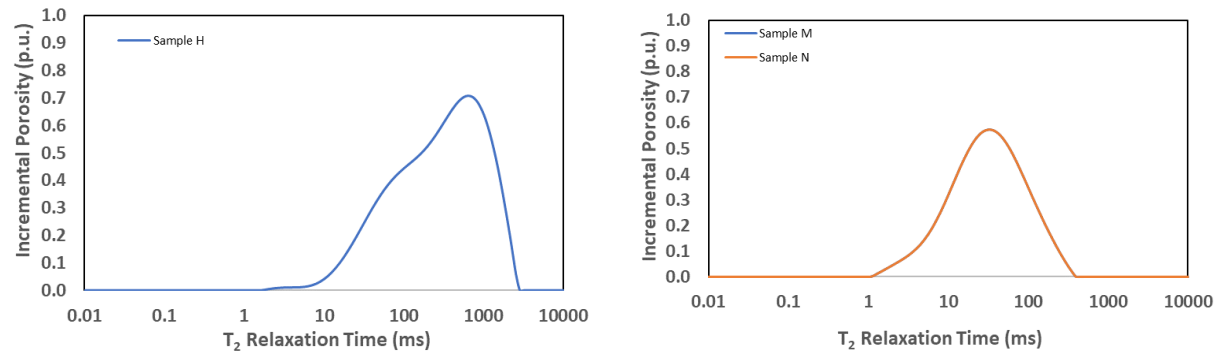


Figure 3.15: NMR T_2 relaxation for the samples used for iron additive. Continuous lines show the probability distribution function (PDF) of the pores. Left (highly permeable), Right (Low permeability)

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Impact of viscosifier on NMR T_2 relaxometry response

Figures 4.1 and 4.3 illustrate the T_2 distribution of rock samples at two stages: 100% water saturation and post-mud filtrate invasion. Figure 4.1 shows the results for the permeable Indiana limestone samples, while Figure 4.3 demonstrates the same for the tighter Buda limestone samples. The green curve represents the T_2 relaxation at 100% water saturation, reflecting the entire pore size distribution, while the red curve illustrates the T_2 relaxation after the sample was invaded by drilling fluid. There was no proper mud-cake formation in this experiment due to the absence of particle additives in the drilling fluid (Figure 4.4).

When using a 0.5g XC polymer concentration, it is evident that the bulk relaxation of the drilling fluid does not significantly impact the T_2 relaxation time post-invasion (Figure 4.1a). The NMR signal corresponding to the microporosity remains predominantly unchanged, while the alteration of the macroporosity signal might be attributed to the 1.6 p.u. reduction in porosity (Table 4.1, sample A), as the viscous polymer can plug some pores when filtered out of the drilling fluid. The smaller impact of the 0.5 XC polymer drilling fluid, compared to the higher concentration fluids, on the overall NMR signal can be attributed to its higher T_{2B} of the drilling fluid which is comparable to that of brine (as shown in Figure 4.2b).

As the concentration of the XC polymer in the drilling fluid increases from 0.5g to 2g and 5g (Figure 4.1c – 4.1e), the T_2 distribution of the permeable samples becomes significantly distorted. The NMR signal from the macropores is almost diminished, while the incremental porosity within micropores (peak around 100 ms) has increased. The reduction in porosity for these samples (Table 4.1) can contribute to the observed trends (i.e. reduction of macroporosity) but cannot fully explain them. This is because the reduction in porosity did not exceed 3 p.u., while the macropores

originally contributed to about half of the pore volume (about 10 p.u. as per Table 4.1). That is, the disappearance of the macroporosity signal in sample C, for example (Figure 4.1e), cannot be solely attributed to the complete blockage of such macropores, as the porosity reduction was much smaller than the macropores fraction. This suggests that another factor must be contributing to the observed results, and we propose that this factor is the drilling fluid bulk relaxation.

Supporting this argument, one can notice the decrease in T_{2B} as the XC polymer concentration increases (Figure 4.2). The drilling fluid T_{2B} is around 1000 ms for the 5g XC polymer (Figure 4.2d), which is comparable to the T_2 peak corresponding to the macropores in the original water-saturated sample (green curve in Figure 4.1e). In this case, the NMR T_2 signal will no longer be solely indicative of the pore structure, as T_{2B} cannot be neglected anymore (it is comparable with T_{2S}). The reduction in T_{2B} leads to faster overall relaxation, causing some relaxation within the macropores to overlap with the signal from micropores, thus resulting in an increased amplitude for the microporosity peak (Figure 4.1e).

Unlike the permeable samples, the NMR signal in the tight Buda samples did not show significant changes after the drilling fluid invasion (regardless of the polymer concentration), as demonstrated in Figure 4.3. It is important to mention that the filtrate volume collected in the tight samples exceeded one pore volume (Figure A.1 in the appendix), indicating that the drilling fluid did invade the sample. Nevertheless, this invasion did not seem to alter the original T_2 distribution in the rocks.

In this context, surface relaxivity remains the dominant relaxation mechanism, particularly in tighter formations like the Buda limestone, where the pore sizes are significantly smaller than the macropores within the Indiana limestone. In this case, the drilling fluid T_{2B} (at least 1000 ms) remains much higher than the T_2 peak of the original Buda samples (19.95 ms), and consequently,

bulk relaxation has minimal impact on the T_2 of the invaded sample. Conversely, in more permeable formations like the Indiana limestone (Fig. 4.1), the larger pore sizes mean that the T_2 relaxation time of the sample is comparable to the T_{2B} of the drilling fluid. In this case, the bulk relaxation of the drilling fluid has a more pronounced effect, leading to a noticeable decrease in the overall T_2 relaxation time of the sample after invasion. This is particularly evident in the macropores, where the influence of bulk relaxation can cause a shift in the T_2 distribution (Fig. 4.1c and 4.1e), resulting in a distorted interpretation of the pore size within the rock.

Table 4.1. *Rock and their petrophysical properties before and after invasion of drilling fluids #1.2 to 1.4*

Sample ID	Rock Type	Porosity	Pore volume	Porosity	Pore volume
		before (%)	before (cc)	after (%)	after (cc)
Sample A	Indiana	20.6	11.14	19.0	10.32
Sample B	Indiana	20.6	11.14	18.0	10.06
Sample C	Indiana	19.6	11.13	16.7	9.45
Sample I	Buda	16.7	8.25	15.9	8.19
Sample J	Buda	16.8	9.78	15.6	9.15

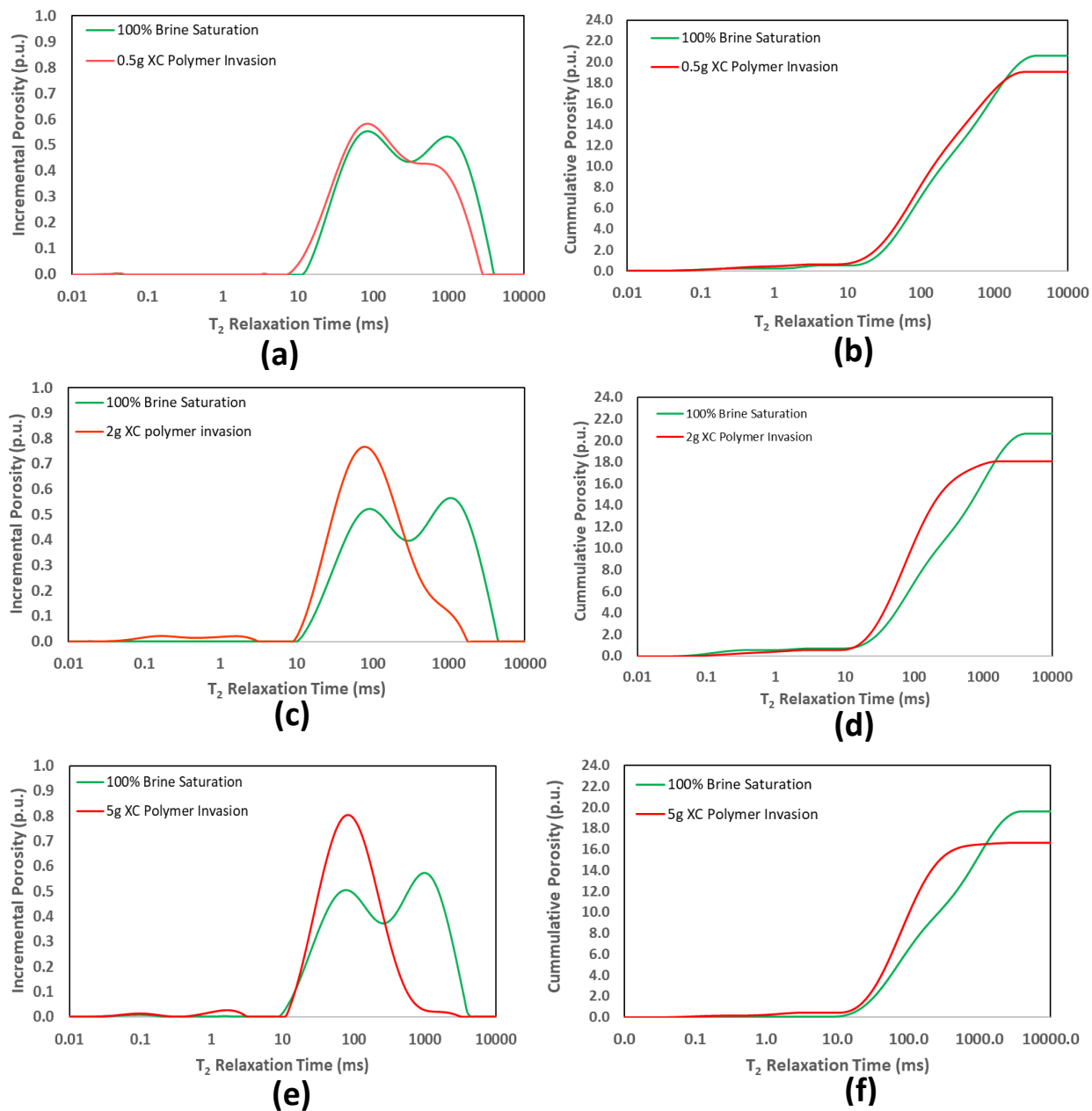


Figure 4.1: NMR T_2 distribution (incremental porosity) before & after invasion for the permeable samples (plots A, C, E). The corresponding cumulative relaxation distribution is shown in plots B, D and F.

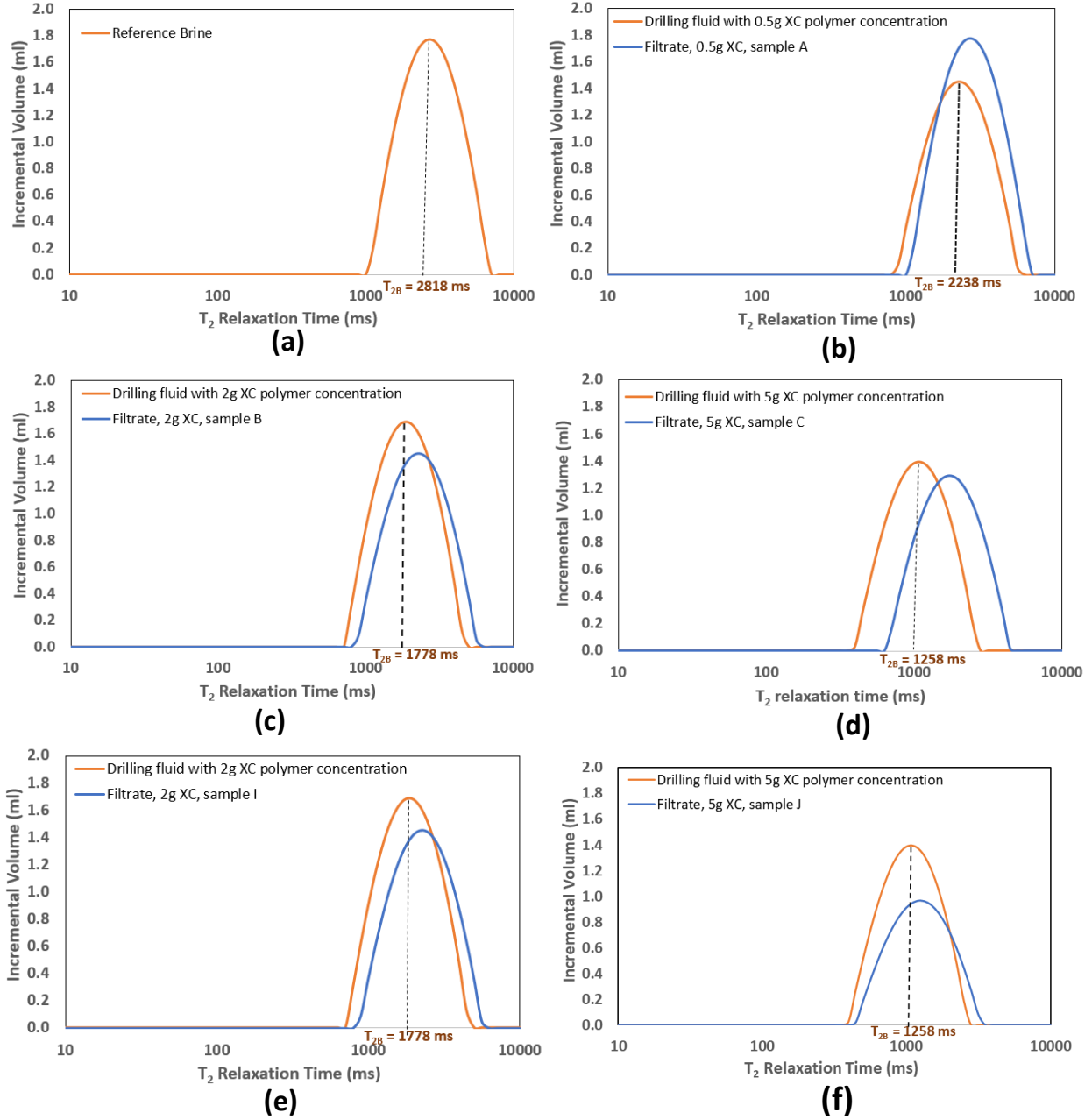


Figure 4.2: NMR T_2 relaxation showing the bulk fluid relaxation (T_{2B}) for the original brine (a) as well as the drilling fluid before (orange) and after (blue) the filtration experiment for the permeable (A - C) and tight (I - J) samples. The increase in the filtrate T_{2B} compared to the original drilling fluid is due to filtering out the viscous component from the fluid during the experiment.

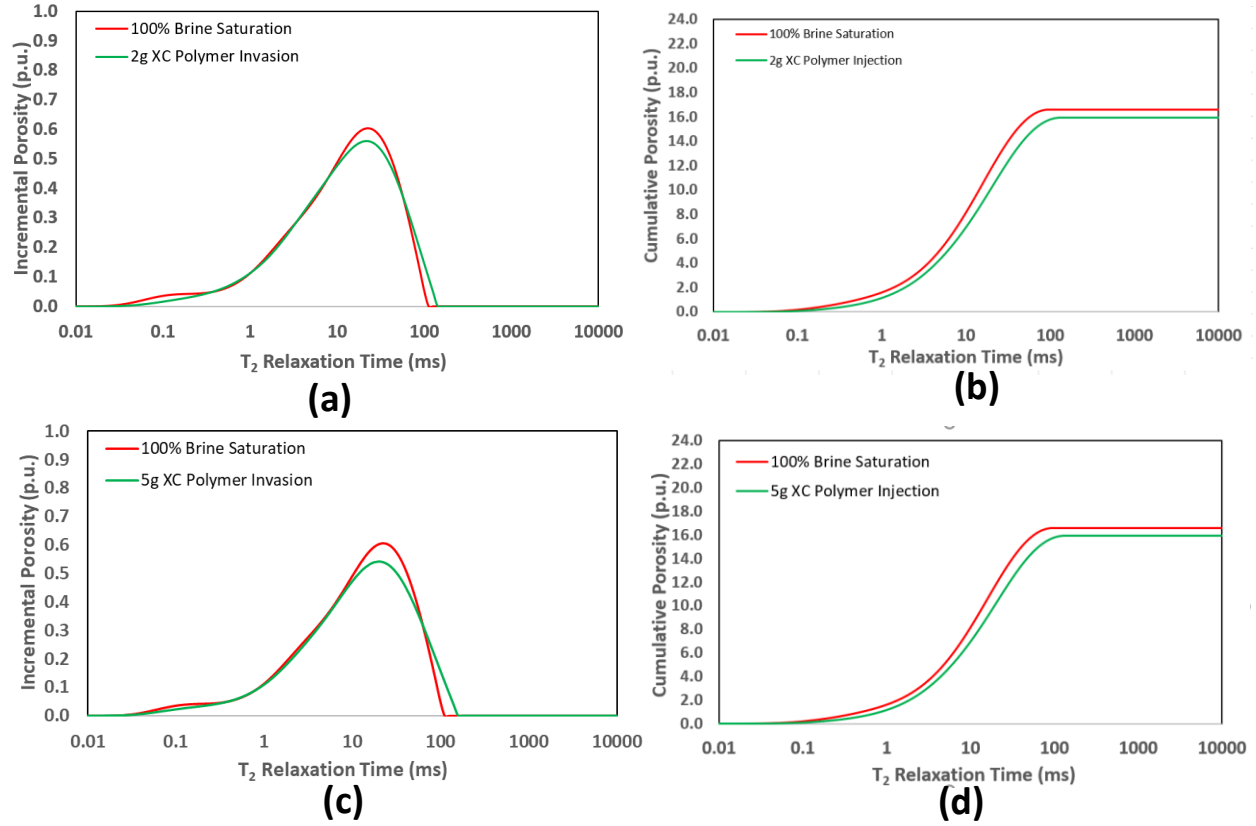


Figure 4.3: NMR T_2 distribution (incremental porosity) before and after the invasion for the tight samples (plots a, c). The corresponding cumulative relaxation distribution is shown in plots b, and d



Figure 4.4: An example of poor filter cake during filtration experiment due to absence of particle additives

4.2 Impact of particles additive (perlite) on NMR T_2 relaxometry response

The use of particles as additives in the drilling fluid (Table 3.2) resulted in the formation of a well-developed filter cake. The NMR T_2 distribution curves in Figure 4.5 and 4.7 correspond to measurements taken at three different states. The green curve represents the T_2 relaxation at 100% water saturation, serving as a reference for the original pore size distribution. The orange curve depicts the T_2 relaxation after the sample was invaded by drilling fluid (as described in Table 3.2), with the upstream filter cake in place, while the blue curve represents the T_2 relaxation after the filter cake was removed.

When using the baseline drilling fluid (fluid #2.1, Table 3.2), it is evident that the bulk relaxation of the drilling fluid impacts the T_2 relaxation time post-invasion (Figure 4.5a). The reduction in total porosity did not exceed 1 p.u. (Table 4.2), yet the signal from macropores decreased significantly, with an overall shift of the T_2 distribution to shorter times (Figure 4.5a). Comparing the results in Figure 4.5 with those in Figure 4.1a (same 0.5g XC polymer concentration but without particle additives), it is clear that the addition of particles has had a more significant impact on the NMR signal. For example, the signal from micropores did not change much when using only 0.5g XC polymer concentration (Fig. 4.1a), but the addition of bentonite and barite (drilling fluid #2.1) altered the T_2 distribution even for the microporosity system (Figure 4.5a).

Comparing both the orange and blue curves in Figure 4.5a suggests that the signal where $T_2 < 10$ ms in the orange curve corresponds to relaxation within the microporous filter cake. The addition of perlite (drilling fluid #2.2, Table 3.2) resulted in a thicker filter cake (Table A.1 in the appendix), leading to a more pronounced signal from the filter cake (Figure 4.5c) and a larger difference between the orange and blue curves. The more significant impact of drilling fluids #2.1 and 2.2 (with added particles) on the NMR signal compared to drilling fluid #1.2 (same XC polymer

concentration but without particles) can be explained by the much smaller T_{2B} of the drilling fluids when particles are added.

Figure 4.6 shows the T_{2B} of the drilling fluids before and after the filtration experiments. The peak T_{2B} for drilling fluids #2.1 and 2.2 is about 80 ms, which is smaller than the original T_2 values dominated by surface relaxivity when the sample was saturated with brine. This applies to both the micro- and macropores, as the original corresponding peaks (green curves in Figures 4.5a and 4.5c) were about 100 ms and 1000 ms, respectively. This results in faster relaxation, causing an overall shift of T_2 to shorter times. Because of the very small T_{2B} of the drilling fluid, with and without the perlite additive (Figure 4.5), the NMR T_2 signal will no longer be solely indicative of the pore structure, as T_{2B} cannot be neglected. The reduction in T_{2B} leads to faster overall relaxation, causing some relaxation within the macropores to overlap with the signal from micropores, thus resulting in relatively an increased amplitude for the microporosity peak (Figure 4.5).

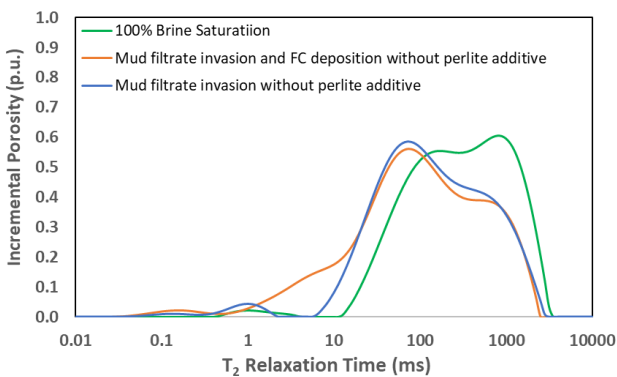
This shift in T_2 to the left due to bulk relaxation of the drilling fluid also led to an overestimation of formation damage when using the mean- T_2 model to estimate permeability from the NMR data (Figure 4.5c and 4.5d). Specifically, the model indicated a 48.1% reduction in permeability, attributing the leftward shift caused by bulk relaxation to a decrease in pore size. However, laboratory measurements revealed a smaller reduction of 32.5%, highlighting the discrepancy between the model's assumptions and experimental observations.

For the tight Buda samples, the impact of drilling fluid on the NMR signal is again minimal (Figure 4.7). There is only a slight reduction in porosity (by less than 1.5 p.u., Table 4.2), but the overall T_2 distribution shape remains similar. The filtrate volume (Fig. A.2 in the appendix) and the porosity reduction (Table 4.2) due to invasion were not significantly different for both permeable

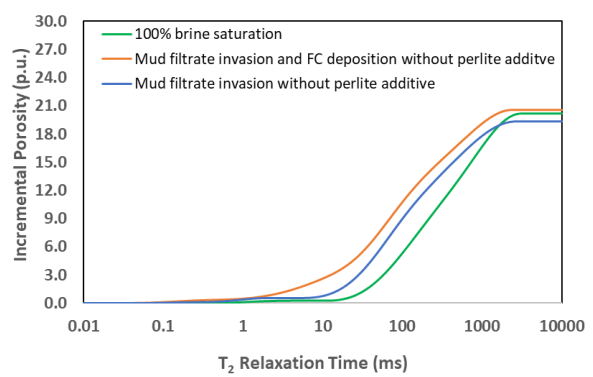
and tight samples, yet the permeable samples experienced more significant NMR alteration. This can be explained by the smaller pore size of the tight Buda samples, where the surface area-to-volume ratio is much higher, making surface relaxivity the primary factor influencing the relaxation time. The dominant peak of the original Buda sample saturated with brine (dominated by surface relaxation) is around 10 ms, which is still lower than the T_{2B} of the drilling fluid (Figure 4.6). Therefore, surface relaxation remains the dominant mechanism of relaxation even after the invasion of the tight samples. Figure 4.8 and 4.9 shows the samples used with the filter cake formed during the filtration experiments.

Table 4.2. Rock and their petrophysical properties before and after invasion for the experiments involving added particles (drilling fluids # 2.1 and 2.2).

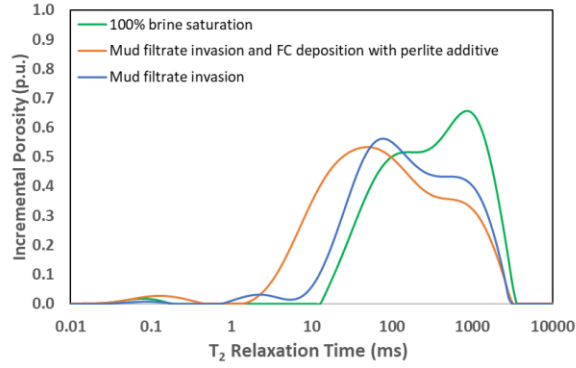
Sample ID	Rock Type	Porosity	Pore volume	Porosity	Pore volume
		before (%)	before (cc)	after (%)	after (cc)
Sample D	Indiana	20.2	11.32	19.40	10.85
Sample E	Indiana	20.6	11.69	19.30	10.99
Sample K	Buda	16.6	8.65	15.70	8.20
Sample L	Buda	16.5	8.44	14.80	7.60



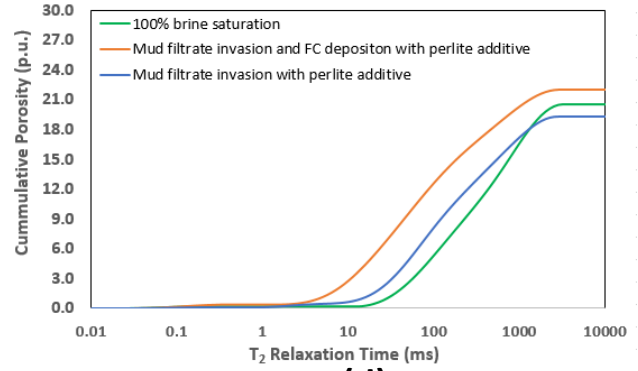
(a)



(b)

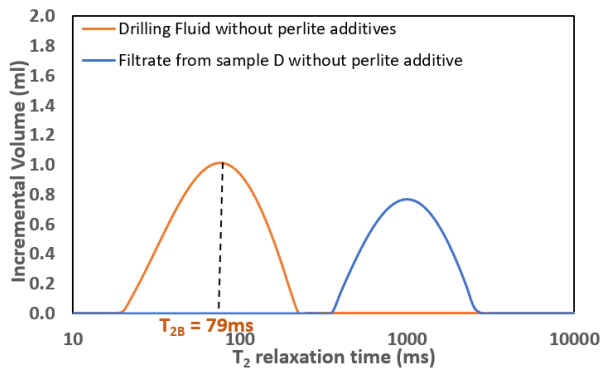


(c)

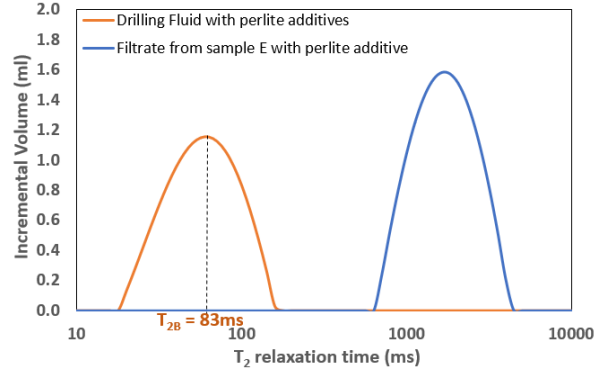


(d)

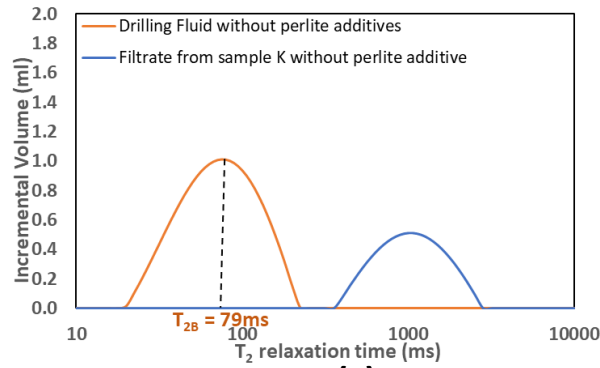
Figure 4.5: NMR T_2 incremental and cumulative distribution for sample D (upper row) with barite and bentonite added but without perlite; and sample E (lower row) with perlite used as additive. Green curves correspond to the original signal before invasion, the orange is after invasion with filter cake (FC) in place, while blue curve is for the measurements after FC removal.



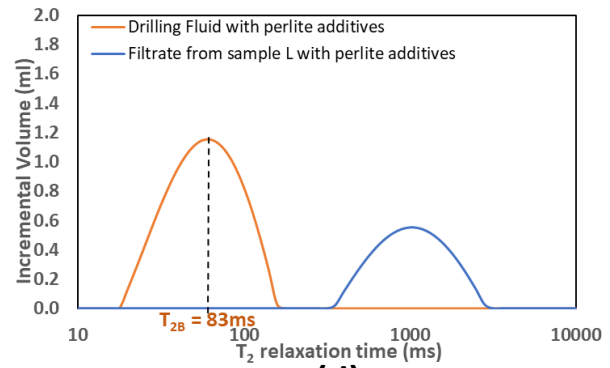
(a)



(b)



(c)



(d)

Figure 4.6: NMR T_2 relaxation showing the bulk fluid relaxation (T_{2B}) for the drilling fluid before (orange) and after (blue) the filtration experiment for the permeable (a-b) and tight (c-d) samples. The increase in the filtrate T_{2B} compared to the original drilling fluid is due to filtering out the viscous component from the fluid during the experiment.

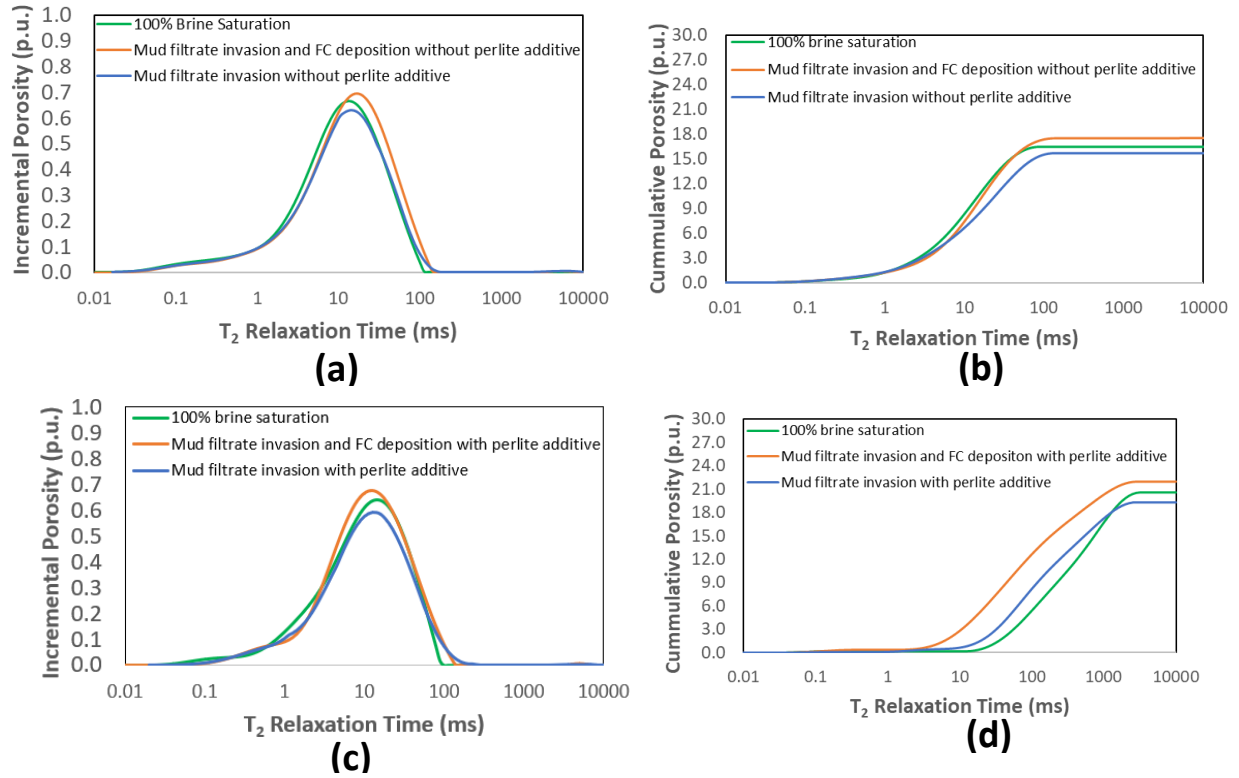


Figure 4.7: NMR T_2 incremental and cumulative distribution for sample H (upper row) with barite and bentonite added but without perlite; and sample I (lower row) with perlite used as additive. Green curves correspond to the original signal before invasion, the orange is after invasion with filter cake (FC) in place, while blue curve is for the measurements after FC removal.

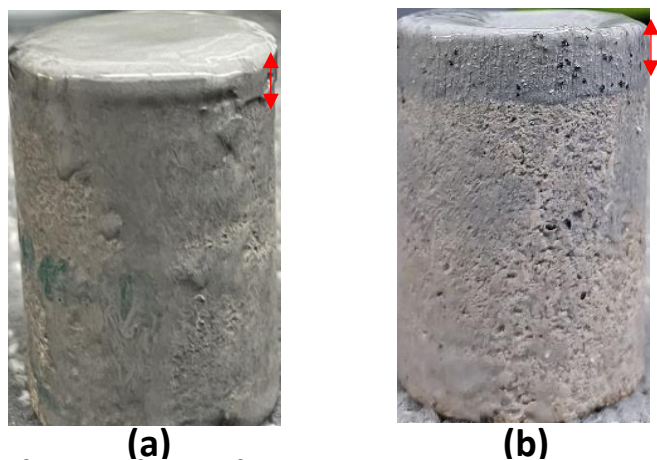


Figure 4.8: The filter cake formed after the filtration experiment (a) Sample D (b) Sample E

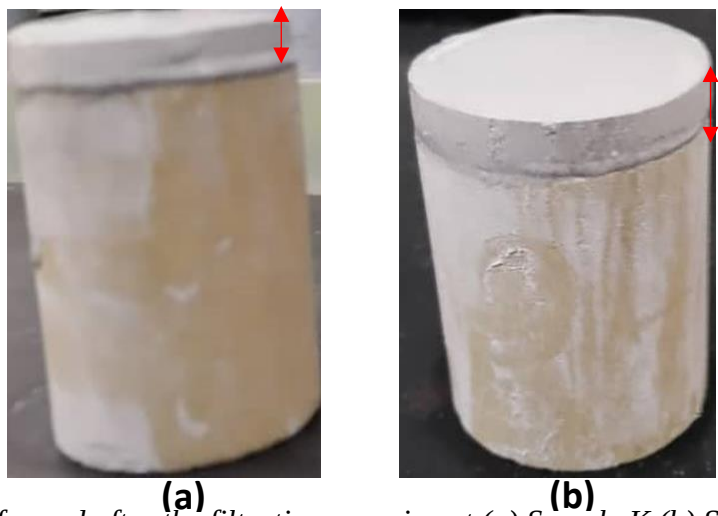


Figure 4.9: The filter cake formed after the filtration experiment (a) Sample K (b) Sample L

4.3 Validation of the usage of rock as filter during filtration experiment

Figure 4.10 illustrate the T_2 distribution of rock samples under two different conditions: (i) filtrate injection into the rock after performing the filtration test using standard filter paper, and (ii) after using rock as a filter. In these figures, the green curve represents the T_2 relaxation at 100% water saturation, indicating the entire pore size distribution of the rock. The blue curve depicts the T_2 relaxation after the sample was invaded by drilling fluid without a filter cake, using the rock as the filtration medium. Meanwhile, the red curve shows the T_2 relaxation following the injection of the filtrate collected from the filter paper setup during the filtration experiment, which simulates the process of mud invasion. Figure 4.10a presents the result when drilling fluid #2.1 was used, while Figure 4.10b shows when drilling fluid #2.2 was used (table 3.2).

In Figure 4.10a, which compares the T_2 relaxation time distribution of rock sample F under different invasion conditions using the baseline drilling fluid (fluid #2.1 from Table 3.2), a distinct contrast is observed between the filter paper setup and the rock-fluid setup. For the rock-fluid setup, represented by the blue curve, there is a noticeable leftward shift in the T_2 relaxation time after mud filtrate invasion. This shift occurs because the drilling fluid directly affects the invaded rock's relaxation properties due to the presence of bulk relaxation effects, which reduce the T_2 values. The presence of solid particles and additives in the mud filtrate contributes to this change by altering the pore structure or fluid properties within the rock. In contrast, the red curve, which represents the filter paper setup, shows a different trend. The filter paper acts as a sieve, effectively removing most of the solid particles and additives from the drilling fluid, resulting in a cleaner filtrate. When this cleaner filtrate is injected into the rock sample, it causes only a minor change in the T_2 relaxation time because there are no significant alterations to the pore structure or fluid characteristics within the rock. This indicates that the filtrate's influence on the rock's NMR response is minimal when the solid components are filtered out.

Figure 4.10b exhibits a similar trend for rock sample G, which was tested using drilling fluid #2.2 (with perlite added, as noted in Table 3.2). In the case of the rock as the filter medium (blue curve), the addition of perlite results in a pronounced leftward shift in the T_2 relaxation time, again due to the bulk relaxation effect of the drilling fluid. The only difference is more distortion and greater filter cake was formed due to this added additive (refer to section 4.2). The overall trend is consistent regardless of the fluid's composition, as the solid particles in the drilling fluid affect the invaded rock's NMR response. However, for the filter paper setup (red curve), there is no significant difference in the T_2 response with or without perlite. This similarity arises because the filter paper effectively removes most of the solid components from the drilling fluid before injection, resulting in a filtrate that lacks substantial additives and thus has a minimal impact on the rock's T_2 distribution.

For both scenarios, the permeability reduction estimated from NMR was significantly lower for the filter paper compared to the rock filter. When using drilling fluid without perlite, the filter paper showed a permeability reduction of only 0.96%, while the rock filter exhibited a much higher reduction of 32.5%. Similarly, for drilling fluid containing perlite, the permeability reduction from NMR model for the filter paper was 0.95%. This discrepancy underscores the differing permeability responses between filter paper and rock filters.

The use of rock as a filter in filtration experiments provides a more realistic representation of subsurface conditions compared to using filter paper. When rock serves as the filtration medium, the drilling fluid invades the actual pore spaces, interacting directly with the rock's natural pore structure, fluid distribution, and mineral composition. This interaction influences the NMR T_2 relaxation behavior, reflecting the true effects of mud invasion, including bulk relaxation and changes in pore fluid properties due to the presence of additives and particulates. In contrast, filter

paper filtration does not accurately simulate this process, as it acts merely as a barrier that removes most of the solid particles and additives, resulting in a cleaner filtrate with minimal interaction with the pore structure. Consequently, using filter paper overlooks the complex mechanisms involved in real mud invasion, such as particle bridging, pore-throat blockage, and the alteration of fluid-rock interactions, which are critical factors influencing the actual reservoir conditions during drilling operations. Table 4.3 shows the petrophysical properties of the rocks before and after the filtrate injection into the rock. Sample D and E were used as comparison since they share similar pore structure. Their petrophysical properties before and after invasion when rocks were used as filter during the experiment was shown in table 4.2 (refer to section 4.2).

Table 4.3. Rock and their petrophysical properties before and after injection for the experiments involving filter press set-up (drilling fluids # 2.1 and 2.2).

Sample ID	Rock Type	Porosity	Pore volume	Porosity	Pore volume
		before (%)	before (cc)	after (%)	after (cc)
Sample F	Indiana	19.00	8.47	19.20	8.50
Sample G	Indiana	18.80	8.20	18.60	8.13

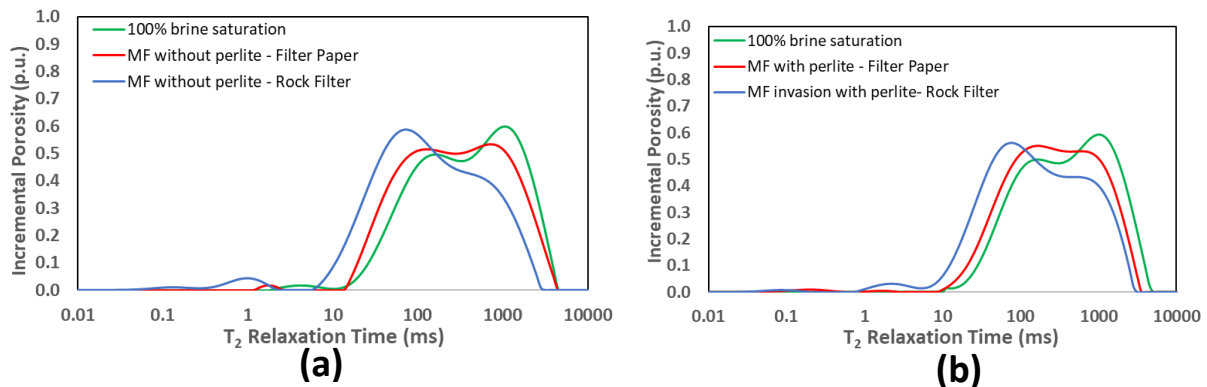


Figure 4.10: NMR T_2 incremental distribution for sample F (left) with barite and bentonite added but without perlite; and sample G (right) with perlite used as additive. Green curves correspond to the original signal before invasion, the red is the mud filtrate injection in the rock after filtering with filter paper, while blue curve is for the measurements when the rock is used as a filter.

4.4 Impact of particles additive (Hematite) on NMR T_2 relaxometry response

The use of particles as additives in the drilling fluid (Table 3.3) resulted in the formation of a well-developed filter cake. The NMR T_2 distribution curves in Figure 4.11 and 4.13 correspond to measurements taken at three different states. The green curve represents the T_2 relaxation at 100% water saturation, serving as a reference for the original pore size distribution. The orange curve depicts the T_2 relaxation after the sample was invaded by drilling fluid (as described in Table 3.3), with the upstream filter cake in place, while the blue curve represents the T_2 relaxation after the filter cake was removed.

The results indicate that the use of hematite-containing drilling fluid (#3.2, Table 3.3) significantly impacts the NMR T_2 relaxation times and porosity of the sample. The addition of hematite increases the magnetic susceptibility, leading to accelerated relaxation and a shift in the T_2 distribution toward shorter times, as seen in Figure 4.11. This shift, particularly for T_2 values below 1 ms, is associated with the formation of a microporous filter cake, which is more evident when hematite is present. The absence of this low T_2 signal in samples without hematite (Figure 4.5c) underscores the influence of hematite on the NMR response. Additionally, a notable reduction in total porosity, by about 5.1 porosity units, is observed, which may be attributed to the presence of magnetic gradients between the pore and the grain's surface due to hematite invasion as it can be seen the invasion is only at the topmost part of the sample, as shown in Figure 4.12. The hematite presence really led to underestimation of porosity as the porosity measured in the laboratory was 18.87% as opposed to NMR porosity of 14.9 p.u. Also, permeability reduction was about 61% for NMR and 31% reduction was measured in laboratory showing the hematite drilling fluid bulk relaxation was low enough and this reduction was accounted for as pore reduction in the permeability estimate equation. Figure 4.13 shows the drilling fluid bulk relaxation with hematite and the filtrate bulk relaxation and it is noticeable that the rock act as a filter to limit particle

invasion into the sample. Figure 4.14 shows the sample used with filter cake formed after the filtration experiment.

The impact of drilling fluids on the NMR T_2 distribution of tight Buda samples is minimal, primarily due to the small pore sizes and high surface area-to-volume ratio, which make surface relaxivity the dominant relaxation mechanism. The T_2 distribution for the original brine-saturated sample shows a peak around 30 ms, indicating surface relaxation as the main influence, which persists even after mud filtrate invasion. The slight reduction in porosity (less than 0.6 p.u.) after invasion suggests limited fluid movement into the pore space because of the rock's low permeability. The addition of hematite to the drilling fluid significantly affects the NMR response, as seen in the low T_2 signal (below 1 ms) in Figure 4.14c, attributed to the filter cake formed during the filtration process. This signal is absent in Figure 4.14b, where the filter cake is formed without hematite, indicating that the presence of hematite increases magnetic susceptibility and accelerates relaxation, resulting in shorter T_2 values. Figure 4.16 shows the sample with the filter cake formed after the filtration experiment for both without hematite (a) and with hematite (b).

Table 4.4. Rock and their petrophysical properties before and after injection for the experiments involving filter press set-up (drilling fluids # 3.1 and 3.2).

Sample ID	Rock Type	Porosity	Pore volume	Porosity	Pore volume
		before (%)	before (cc)	after (%)	after (cc)
Sample H	Indiana	20.00	10.46	14.90	7.84
Sample P	Leuders	13.70	6.50	13.30	7.07
Sample Q	Leuders	13.00	6.00	12.50	6.69

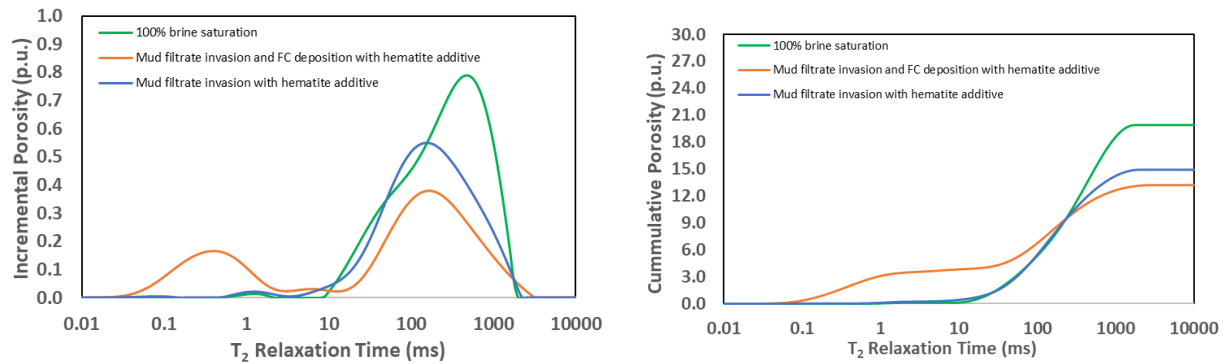


Figure 4.11: NMR T_2 incremental and cumulative distribution for sample H with barite and bentonite added but without hematite. Green curves correspond to the original signal before invasion, the orange is after invasion with filter cake (FC) in place, while blue curve is for the measurements after FC removal.

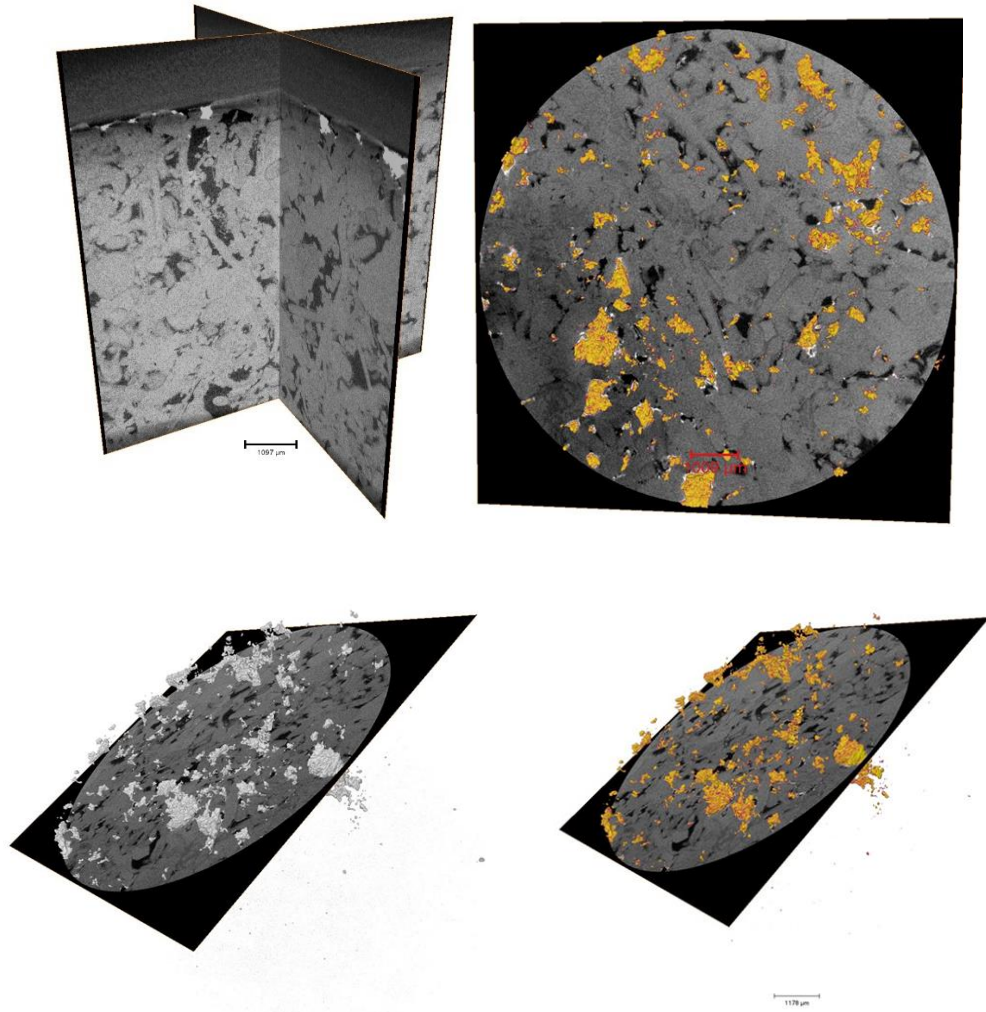


Figure 4.12: CT scans of the sample used for hematite invasion experiments, shown from different angles. The images highlight the distribution and extent of hematite invasion within the sample, particularly at the top, where significant clogging and porosity reduction are observed

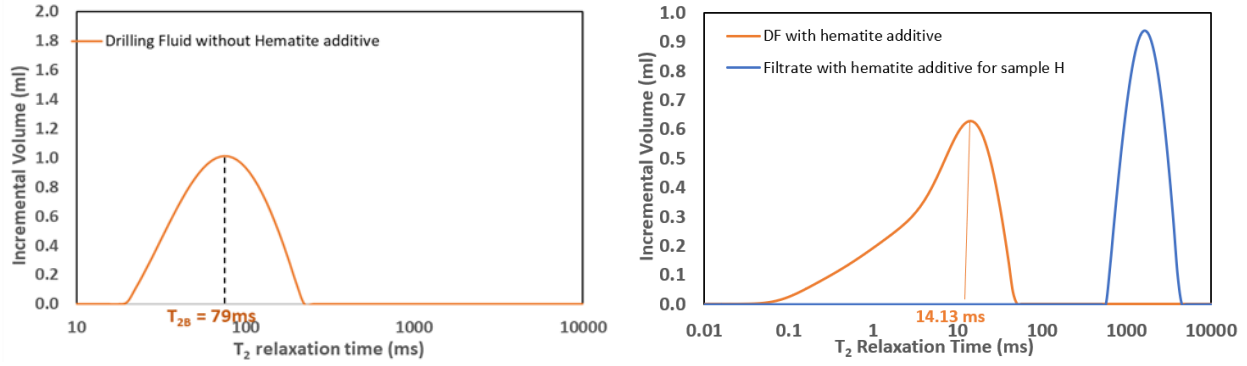


Figure 4.13: NMR T_2 relaxation showing the bulk fluid relaxation (T_{2B}) for the drilling fluid before (orange) and after (blue) the filtration experiment for the permeable (a-b) and tight (c-d) samples. The increase in the filtrate T_{2B} compared to the original drill drilling fluid is due to filtering out the viscous component from the fluid during the experiment.

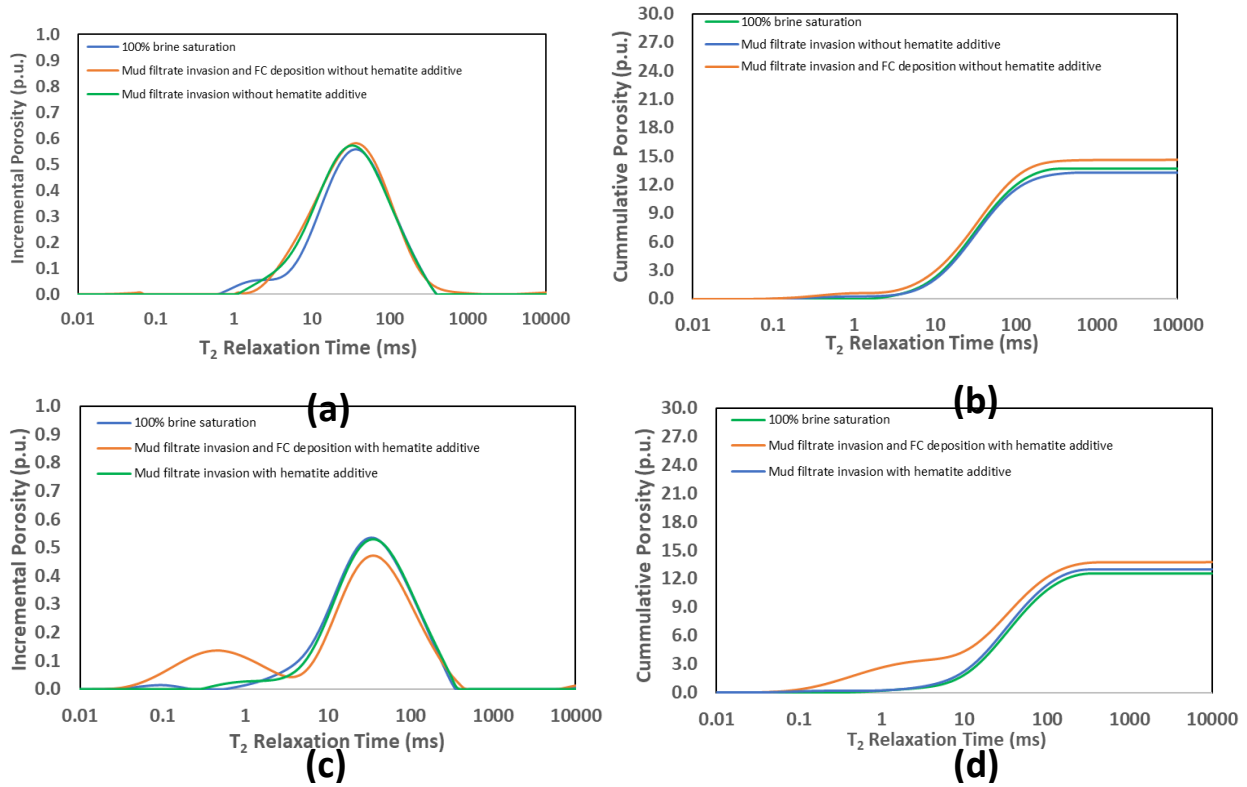
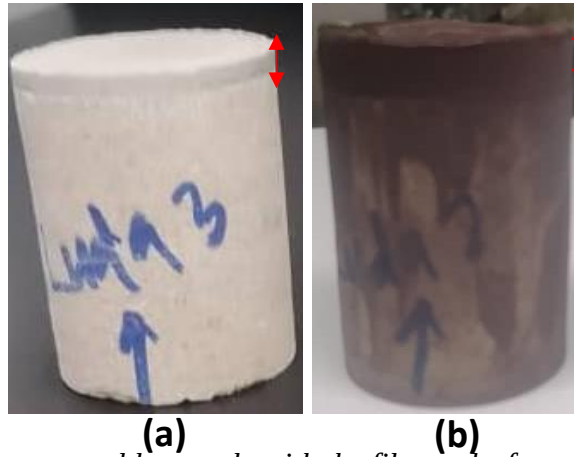


Figure 4.14: NMR T_2 incremental and cumulative distribution for sample M with barite and bentonite added but without hematite. Green curves correspond to the original signal before invasion, the orange is after invasion with filter cake (FC) in place, while blue curve is for the measurements after FC removal.



Figure 4.15: The high permeable sample with the filter cake formed after the filtration experiment



(a)

(b)

Figure 4.16: The low permeable sample with the filter cake formed after the filtration experiment. (a) the filter cake formed after using drilling fluid 3.1 without hematite in table 3.3 (b) the filter cake formed after using drilling fluid 3.2 with hematite in table 3.3

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

This study investigated how different drilling fluid properties, including bulk relaxation and the presence of various additives, influence NMR T_2 relaxometry responses in near-wellbore conditions. The focus was on understanding the impact of these factors on pore size distribution and relaxation mechanisms in both permeable and tight limestone samples. The findings reveal that the bulk relaxation properties of drilling fluids can significantly affect T_2 values and their distributions, particularly in rocks with larger pore sizes. Drilling fluids with lower T_2 bulk relaxation times resulted in noticeable shifts in the T_2 distribution, especially within the macroporosity range. This shift was more evident in permeable samples like Indiana limestone, where the bulk relaxation effects of the drilling fluid could not be disregarded due to the similarity between T_{2B} and T_{2S} . The bulk relaxation effects in these formations led to a reduction in the T_2 signal corresponding to macropores and an apparent increase in the signal from micropores due to overlapping relaxation contributions. Conversely, the impact of drilling fluids on tight Buda limestone samples was minimal, as surface relaxivity dominated the relaxation mechanism. This dominance is attributed to the smaller pore sizes and higher surface area-to-volume ratios, which limited the influence of the bulk relaxation properties of the drilling fluid.

The role of particulate additives in the drilling fluids (e.g., bentonite, barite, perlite, and hematite) was also significant in shaping the NMR T_2 responses. When additives were introduced, particularly at higher concentrations, the formation of well-developed filter cakes was observed. These filter cakes, characterized by lower T_{2B} values than the original brine-saturated rock, resulted in an overall shift of T_2 distributions to shorter times, indicating faster relaxation. The addition of particles altered both the pore structure and fluid properties within the invaded rocks,

resulting in a more pronounced reduction in the macroporosity signal. The impact of hematite was particularly notable; its magnetic susceptibility accelerated relaxation processes, leading to a shift toward shorter T_2 times and the appearance of low T_2 signals below 1 ms. These effects underscore the substantial role that drilling fluid composition plays in NMR responses, influencing not only porosity reduction but also the interpretation of pore size distribution.

Experiments involving different filtration setups further illustrated the significance of filtration media in simulating mud invasion conditions. When filter paper was used as the filtration medium, it effectively removed most solid components from the drilling fluid, resulting in minimal changes to the T_2 distribution after filtrate injection. In contrast, using the rock itself as the filter medium allowed for direct interaction between the drilling fluid and the natural pore structure, providing a more realistic representation of subsurface conditions. The rock-filtration setup revealed more significant shifts in T_2 values due to bulk relaxation effects and changes in pore fluid properties, which were not captured using filter paper. This highlights the complexity of mud invasion processes, including particle bridging, pore-throat blockage, and fluid-rock interaction alterations that are critical to understanding real reservoir conditions during drilling.

In conclusion, this study demonstrated that the bulk relaxation properties and additives in drilling fluids significantly influence NMR T_2 relaxometry responses, with varying impacts depending on the rock permeability and pore size distribution. Permeable rocks with larger pore sizes are more susceptible to bulk relaxation effects, leading to distorted NMR responses and misinterpretations of pore size distribution, while tight formations remain dominated by surface relaxivity. The inclusion of particulate additives like hematite can further complicate the relaxation behavior by accelerating T_2 relaxation due to magnetic susceptibility effects. These findings underscore the

importance of accounting for drilling fluid properties when interpreting NMR data in the near-wellbore region.

5.2 Recommendations

Based on the findings, it is recommended to carefully consider the properties of drilling fluids when designing mud invasion experiments or interpreting NMR data in reservoir characterization. For formations with larger pore sizes, special attention should be given to the T_2 bulk relaxation time of the drilling fluid, as fluids with lower T_{2B} can significantly affect the T_2 distribution and lead to misinterpretation of pore structures. Additionally, using realistic filtration setups such as rock filtration rather than filter paper is recommended to better simulate subsurface conditions and capture the complex interactions between mud and rock. When particulate additives are used in drilling fluids, it is advisable to assess the potential impact on magnetic susceptibility and relaxation behavior, as these factors can accelerate relaxation and alter T_2 distributions. Future studies should utilize this understanding during corrections for drilling invasion in regions where NMR is logged when developing their models to accurately predict NMR responses under different invasion scenarios. The recommendations are summarized below:

1. When interpreting NMR logging data in formations invaded by particle-laden drilling fluids, it is crucial to account for the impact of the bulk relaxation time (T_{2B}) of the drilling fluid. Advanced modeling techniques or correction algorithms should be developed to differentiate between the relaxation effects caused by the drilling fluid and the true pore structure of the formation.
2. Laboratory studies aiming to simulate real-world drilling fluid invasion should prioritize rock-fluid interaction setups over filter paper setups. This will provide more accurate

representations of how drilling fluids, especially those with particle additives, interact with the formation and affect NMR measurements.

3. In tight formations where surface relaxivity dominates, drilling fluid invasion may have minimal impact on the NMR signal and reduction in signal could be attributed to porosity reduction. However, in permeable formations, NMR measurements are more susceptible to alteration by particle-laden drilling fluids leading to bulk relaxation effects which should be accounted for to get the true pore size distribution.
4. In areas where the drilling fluid contains iron additives, NMR logging should be avoided, or the impact must be critically considered in the corrections.
5. To improve the accuracy of formation evaluation, the industry could explore the development of drilling fluids with controlled T_{2B} values that minimize interference with NMR measurements. This would allow for more reliable interpretation of the pore size distribution and fluid properties in both permeable and tight formations.

By considering these findings, it is possible to enhance the accuracy of NMR-based formation evaluation in complex drilling environments, ultimately leading to better reservoir characterization and decision-making in subsurface operations.

5.3 Limitation of study

1. The study primarily relies on controlled laboratory experiments, which may not fully replicate the complexities and variability of in-situ conditions encountered in actual drilling environments. Factors such as temperature, pressure, and fluid dynamics in the field can differ significantly from those in the laboratory.

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Appendix

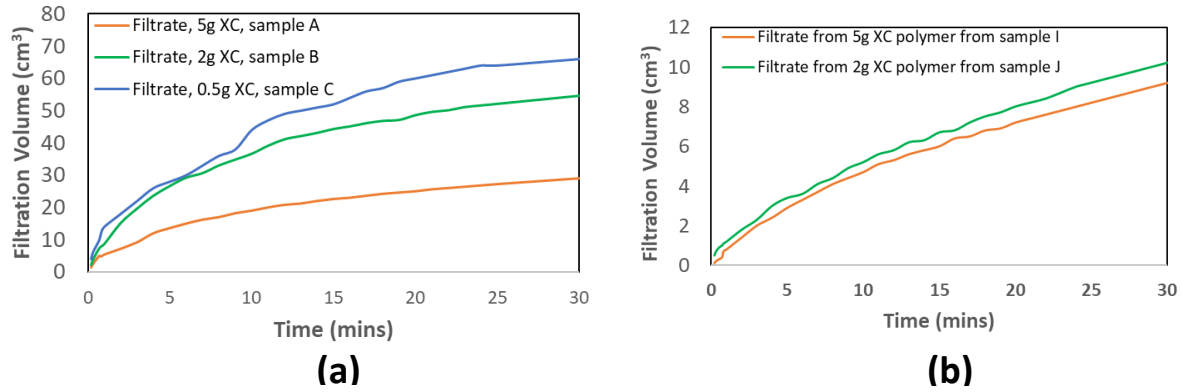


Figure A.1: API fluid loss volume versus time with varied XC polymer concentration (a) for the permeable sample; (b) for the tight samples.

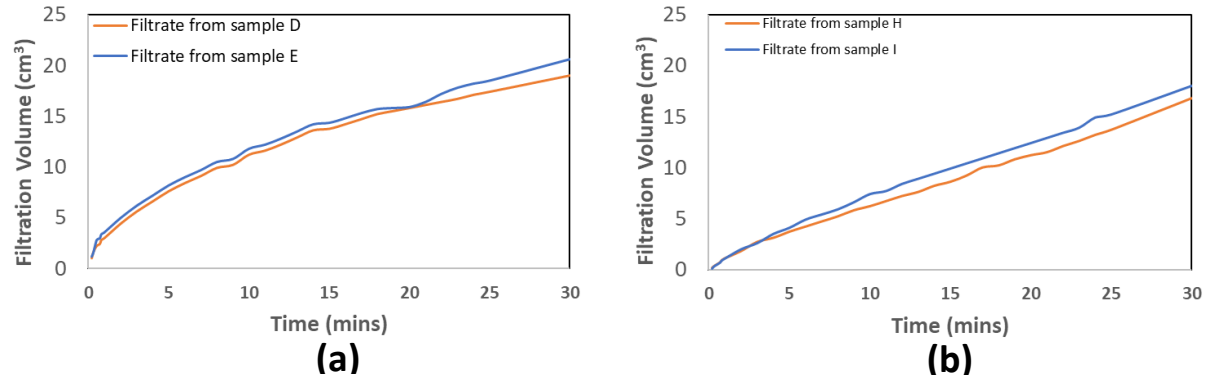


Figure A.2: API fluid loss volume versus time with varied perlite concentration (a) for the permeable sample; (b) for the tight samples.

Table A.1: Filter cake thickness for sample D and H with barite and bentonite added but without perlite; and sample E and I with perlite used as additive.

Sample ID	Length before and after FC deposition		FC thickness
Sample D	4.14	5.00	0.86
Sample E	5.02	6.01	0.99
Sample K	4.60	5.34	0.74
Sample L	4.52	5.46	0.94

Vitae

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Journal Publications/Poster Presentations

1. Fauzan, A., Ammar E, Abdulrauf A., Mohamed, M., and Badr, B (2024). A Systematic Investigation of Drilling Fluid Bulk Relaxation Impact on NMR Interpretation for Formation Evaluation. Journal of Petroleum (Under Review).
2. Fauzan, A. (2024). Petrophysical evaluation of an X field in Niger Delta. A poster presentation at the Seismic Petrophysics Symposium organized by SPWLA at Khobar, Saudi Arabia in October.