



## Full Length Article

## Mitigating shale swelling in deep drilling: Novel natural deep eutectic solvent vs hybrid additives system

Muhammad Hammad Rasool<sup>a,b,\*</sup> , Maqsood Ahmad<sup>a,\*\*</sup>, Numair Ahmed Siddiqui<sup>a</sup>,  
Syahrir Ridha<sup>b,\*</sup>, Azam Khan<sup>c</sup>, Husnain Ali<sup>d</sup> 

<sup>a</sup> Petroleum Geosciences Department, Universiti Teknologi PETRONAS, Bandar Seri Iskandar 32610, Malaysia

<sup>b</sup> Petroleum Engineering Department, Universiti Teknologi PETRONAS, Bandar Seri Iskandar 32610, Malaysia

<sup>c</sup> Department of Petroleum and Gas Engineering, University of Engineering and Technology, Lahore, Pakistan

<sup>d</sup> Department of Chemical and Biological Engineering, Hong Kong University of Science & Technology (HKUST), Hong Kong

## ARTICLE INFO

## Keywords:

Drilling fluid  
Green additives  
NADES  
Shale inhibition

## ABSTRACT

Shale instability during shale drilling poses significant challenges that require effective additives to control swelling and enhance water-based drilling fluids. This study investigates the effectiveness of various shale inhibitors, both individually and in combination, and compares them to the latest innovation i.e, Natural Deep Eutectic Solvents (NADES) as a promising alternative. Various additives including Potassium Chloride (KCl), 1-Ethyl-3-methylimidazolium chloride. ([EMIM]Cl), SiO<sub>2</sub> nanoparticles, amine terminated polyetheramine (ATPE), Okra mucilage, Choline Chloride:Urea Deep Eutectic Solvent (DES), and Citric acid:Glycerine Natural Deep Eutectic Solvent (CA NADES) and their combinations were subjected to rigorous examination to delineate their impact on shale stability and drilling fluid properties. Notably, CA NADES reduced mudcake thickness by 42.8 %, filtrate volume by 40.3 %, and linear swelling by 76.1 %, while improving shale recovery by 51.7 %. Among the additive combinations, SET B (0.5 % KCl + 0.5 % ATPE) and SET G (0.5 % KCl + 0.5 % [EMIM]Cl) demonstrated particularly effective performance. Surface tension measurements revealed favorable interfacial properties, X-ray diffraction analysis confirmed effective intercalation, and zeta potential assessments indicated improved colloidal stability. Overall, these findings highlight the critical role of optimized additive formulations in mitigating shale instability and enhancing drilling fluid performance, offering promising strategies for more efficient and reliable drilling operation.

## 1. Introduction

Shale inhibition stands as a critical aspect of drilling operations, particularly in formations rich in shale deposits [1]. Shale, a type of sedimentary rock composed primarily of clay minerals, poses significant challenges during drilling due to its propensity to swell and collapse upon contact with water in drilling fluids [2]. This phenomenon, known as shale instability, can lead to wellbore instability issues such as stuck pipe, lost circulation, and wellbore collapse, all of which can substantially increase drilling costs and jeopardize operational safety [3].

The significance of shale inhibition lies in its role in mitigating these challenges and ensuring the smooth progression of drilling operations.

By employing effective shale inhibitors, operators can prevent or minimize shale swelling and collapse, thereby stabilizing the wellbore and facilitating efficient drilling processes [4]. Shale inhibitors achieve this by interacting with the clay minerals present in shale formations, altering their physical and chemical properties to inhibit swelling and maintain the integrity of the wellbore. Moreover, shale inhibition is crucial for optimizing drilling fluid performance [5]. Drilling fluids, also known as drilling muds, serve multiple functions in the drilling process, including lubrication, cooling, and carrying drilled cuttings to the surface. However, if not properly managed, drilling fluids can exacerbate shale instability issues by interacting with shale formations [6]. By incorporating effective shale inhibitors into drilling fluid formulations,

\* Correspondence to: Petroleum Engineering Department and Institute of Sustainable Energy & Resources (ISER), Universiti Teknologi PETRONAS, Bandar Seri Iskandar 32610, Malaysia.

\*\* Corresponding author.

E-mail addresses: [Muhammad\\_19000949@utp.edu.my](mailto:Muhammad_19000949@utp.edu.my), [hammad.rasool@utp.edu.my](mailto:hammad.rasool@utp.edu.my) (M.H. Rasool), [maqsood.ahmad@utp.edu.my](mailto:maqsood.ahmad@utp.edu.my) (M. Ahmad), [Syahrir.ridha@utp.edu.my](mailto:Syahrir.ridha@utp.edu.my) (S. Ridha), [azamkhan@uet.edu.pk](mailto:azamkhan@uet.edu.pk) (A. Khan).

<https://doi.org/10.1016/j.deepre.2025.100173>

Received 17 February 2025; Received in revised form 14 March 2025; Accepted 20 March 2025

Available online 24 March 2025

2949-9305/© 2025 The Author(s). Publishing services by Elsevier B.V. on behalf of KeAi Communications Co. Ltd This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

operators can tailor the fluid properties to the specific challenges posed by shale formations, thereby enhancing drilling efficiency and minimizing operational risks [7].

Shale inhibitors can be broadly classified into chemical inhibitors and mechanical inhibitors, each functioning through distinct mechanisms to mitigate shale instability [8]. Chemical inhibitors include (i) inorganic inhibitors, such as salts, metal oxides, and silicates, which reduce shale hydration by altering ion exchange processes; (ii) organic inhibitors, comprising polymers, amines, and macromolecules that enhance shale stability through adsorption and encapsulation mechanisms; (iii) ionic liquids, including imidazolium-based ionic liquids, deep eutectic solvents (DES), and natural deep eutectic solvents (NADES), which modify shale surface properties and suppress hydration; and (iv) natural biobased materials, such as okra mucilage, pomelo peel powder, and potato peel, which offer eco-friendly and sustainable alternatives for shale inhibition [9–12].

On the other hand, mechanical inhibitors primarily function by physically reinforcing the shale structure. These include (i) nanomaterials, such as silica, metal oxides, and nanocomposites, which reduce shale permeability and strengthen its framework, and (ii) polysaccharides, including cellulose and starch, which form a protective film over shale surfaces to prevent water infiltration [13,14]. The integration of chemical and mechanical inhibitors provides a comprehensive approach to addressing shale swelling and instability in drilling operations.

Among chemical inhibitors and particularly among inorganic inhibitors, potassium chloride (KCl) is a widely used inorganic salt in industry that operates through ion exchange with clay minerals. By substituting potassium ions for other cations within the clay lattice, KCl disrupts the hydration forces between clay layers, ultimately contributing to improved wellbore stability and reduced drilling fluid invasion into the formation. In the category of organic inhibitors, several types of amines are popular among leading petroleum services companies as shale inhibitors. Various research groups also explored various organic shale inhibitors and achieved excellent shale inhibition with improved drilling fluid properties. Chen et al. (2017) utilized Amine-tartaric salt (ATS-4) [15] which exhibited excellent compatibility with the modified starch in water-based drilling fluids and showed excellent shale inhibition. Moreover, J. Zhang et al. [16], R. Zhang et al. [17], Song et al. [18], Du et al. [19] utilized ammonium-lauric salt (ALS-2) [16], Ammonium-malic salts (AMS-9), Polyammonium (DEP-7), Piperazine-based polyether Gemini quaternary ammonium salts (QAs) [20], Monomeric amine (DTHDB) [19] and Polyhydroxy organic amine (THEED) [21] respectively in category of organic amine based shale inhibitors which achieved improved shale inhibition and compatibility with other drilling fluid additives. Recently, efficacy of polyetheramine/diamine as shale inhibitors have been investigated by various research groups such as Bai et al. [22], Zhong et al. [23,24], Tian et al. [25], Bat et al. [26], Abbas et al. [27], Li et al. [28], Zhou et al. [29], and is found to be an efficient and compatible drilling fluid additive for shale stability.

Due to toxicity of amines and low thermal stability of polymers, researchers started exploring greener alternatives and that's when ionic liquids came into the picture. Rahman et al. (2021) investigated the efficacy of tetramethylammonium chloride (TMACl) and 1-ethyl-3-methylimidazolium chloride (EMIM-Cl) in drilling mud, observing reductions in linear swelling by 23.40 % and 15.66 %, respectively [30]. Khan et al. (2021) explored the use of a Trihexyltetradecyl phosphonium bis(2,4,4-trimethyl pentyl) phosphonate-based ionic liquid, reporting a 12.3 % decrease in shale inhibition compared to water [31]. In another study, Huang et al. (2020) utilized ionic liquids specifically, 1-hexyl-3-methylimidazolium bromide and 1,2-bis(3-hexylimidazolium1-yl) ethane bromide with Na-Bt pellets demonstrating reductions in shale swelling of 86.43 % and 94.17 %, respectively [32]. Yang et al. (2017) employed 1-Vinyl-3-dodecylimidazolium bromide and 1-Vinyl-3-tetradecylimidazolium bromide, yielding reductions in shale swelling of

16.91 % and 5.81 %, respectively [33]. Ofei et al. (2017) utilized 1-butyl-3-methylimidazolium chloride (BMIM-Cl) in water-based mud (WBM), resulting in a reduction in mudcake thickness of up to 50 % and decreased YP/PV across all considered temperatures, thereby enhancing drilling fluid hydraulics [34]. Furthermore, Yang et al. (2017) investigated the effects of 1-Vinyl-3-ethylimidazolium bromide, achieving a 31.62 % reduction in shale swelling with a 40.60 % shale recovery rate [33]. Lastly, Luo et al. (2017) utilized 1-octyl-3-methylimidazolium tetrafluoroborate, observing an 80 % reduction in shale swelling [35]. Ionic liquids have certain advantages over traditional organic and inorganic solvents but their efficacy faded when more sustainable class of ionic liquids came into the picture as shale inhibitors as recent research declared ILs as toxic, costly and majorly non bio-degradable [36–38]. That is when greener alternative of Ionic liquids i.e, DES came into the picture.

In the realm of utilizing Deep Eutectic Solvents (DES) as shale inhibitors in drilling mud, various researchers made significant advancements. Jia et al. (2019) explored the effectiveness of DES compositions including Propoanoic acid ChCl (1:1), 3-phenyl propanoic acid ChCl (1:2), and 3-mercapto propanoic acid + Itaconic acid + ChCl (1:1:2), achieving bentonite swelling inhibition rates of 68 %, 58 %, and 58 % respectively [39]. Following this, Rasool et al. (2021a, b) conducted experiments utilizing Glycerine: Potassium Carbonate DES in a 2:1 ratio, resulting in 87 % swelling inhibition of shale samples [5,40]. Ma (2021) investigated Urea: ChCl based DES, achieving 67 % inhibition of shale swelling [41]. Rasool et al. (2022a) introduced a novel approach using a combination of Potassium carbonate-based DES and Poly (2-ethyl-2-Oxazoline) hydroxyl terminated polymer in drilling mud, achieving 76 % swelling inhibition [42]. However, the environmental credentials of DES were questioned with the emergence of natural deep eutectic solvents (NADES), which resemble DES in chemical and definitional terms. The key disparity lies in the formulation of NADES through naturally occurring hydrogen bond donors and acceptors, such as salts like KCl and  $\text{CaCl}_2$  [43,44]. Notable research conducted by Rasool et al. (2023a) demonstrated the efficacy of Ascorbic acid-based NADES as a shale inhibitor in drilling mud, yielding 77.7 % inhibition of shale swelling and an 87 % improvement in shale recovery [45]. Similarly, Calcium Chloride-based NADES, KCl based NADES and Epsom salt based NADES showed promising results as a drilling mud additive, exhibiting excellent shale inhibition properties [1,46,47]. In realm of chemical inhibitors, Natural deep eutectic solvents, derivatives of DES, are the latest solution and there has been research conducted utilizing various combination of NADES.

Lately, apart from NADES, the incline of researchers is also shifting towards using bio-based natural product which one may seem as a 'trash' but researchers have proved otherwise with their results. Lately, Okra mucilage has surfaced as an efficient bio based shale inhibitor due to its ability to interact with clay surface and modify its hydration properties [48]. Apart from this various researchers also utilized Mulberry leaf extract, Glycyrrhiza glabra root extract, Pomelo peel extract, Tribulus terrestris extract (TTE), Korean red ginseng root extract, Equisetum arvense leaf extract, Henna extract as shale inhibitors, however, these additives still remain controversial in industrial perspective [49, 50].

Nano-particles serve as prominent mechanical inhibitors by physically interacting with clay minerals and modifying their surface properties. Nanoparticles adsorb onto clay surfaces, creating a protective layer and plugs micro pores that impedes water absorption and clay swelling. Their high surface area and reactivity enable them to reinforce the shale structure and enhance wellbore stability. Among nano particles, various research groups utilized silica nano particles as shale inhibitors and found effective results. For instance, Xu et al. [51] and Yang et al. (2017) utilized silica Nano-particles [52], Polyethylene glycol grafted nano-silica composite (PEGNS) [51], respectively and their shale inhibition is mainly due to blocking shale pores, creating a clogged membrane and modifying the wettability of clay surface.

This research aims to explore the efficacy of popular shale inhibitors across various classes, individually and in combination (sets) with a comparative analysis against the latest class of shale inhibitors, namely Natural Deep Eutectic Solvent (NADES). Specifically, this study will focus on utilizing Citric Acid:Glycerine based NADES, selected based on predetermined screening criteria [53]. Various parameters including drilling fluid properties, linear swelling, and shale recovery will be meticulously assessed. Furthermore, to unravel the underlying mechanisms, Zeta potential, surface tension, and d-spacing of treated and untreated bentonite wafers will be meticulously measured. Ultimately, based on the findings, the most effective combination of shale inhibitors will be recommended as an optimal drilling fluid additive for shale inhibition.

## 2. Materials and methods

### 2.1. Materials

Glycerine 99 USP has been purchased from EvaChem, Selangor, Malaysia. Okra has been purchased from local market in Seri Iskander, Malaysia. KCl, EMIM[Cl]<sup>>98</sup> % (1-Ethyl-3-methylimidazolium chloride), Polyetheramine (Trimethylolpropane tris[poly(propylene glycol), amine terminated] ether -ATPE, Choline Chloride 99 %, Silica, nanoparticle dispersion in water (<30 nm (DLS), triethoxypropylaminosilane functionalized) have been procured from Sigma Aldrich, Malaysia.

### 2.2. Methods

#### 2.2.1. In-house synthesis/processing of additives

KCl, ATPE and [EMIM]Cl were used as it is. Si-NP was procured as a dispersion, therefore, sonication was not needed, so it was used in essence. Okra powder was produced using a dry grinding process. Initially, the okra was extensively washed with water and subsequently dried for 3–4 days at temperatures ranging from 40 °C to 45 °C. Once dried, the okra was pulverized into a fine powder using a grinder.

Choline Chloride and Urea based DES was prepared according to the literature with 1:2 molar ratio at 60 °C while Citric Acid: glycerine NADES is the novelty of this work and has been prepared as deliberated in our previous work using 1:4 molar ratio at 70 °C following M.H. screening criteria for the selection of Hydrogen Bond Donor (HBD) and Hydrogen Bond Acceptor (HBA).

In addition to performance enhancements, the study emphasizes the eco-friendly attributes of the Citric Acid:Glycerine NADES. Citric acid is a naturally occurring organic acid, abundantly available in citrus fruits, and glycerine is a renewable byproduct of biodiesel production. Both compounds are inherently biodegradable and exhibit low toxicity. By synthesizing NADES from these raw materials, the present work not only achieves effective shale inhibition but also aligns with the growing demand for greener drilling fluid formulations. This green profile makes Citric Acid:Glycerine NADES a promising alternative to conventional shale inhibitors, which often raise environmental concerns.

#### 2.2.2. Drilling fluid preparation

The water based drilling mud was prepared using API 13 B-1 standards, and the following components were combined in specified quantities: 22.5 g of Na-bentonite, 0.26 g of sodium carbonate, 0.26 g of NaOH, and 350 milliliters of water. 1 % concentration of all additives were used using design of experiment tabulated in Table 1.

**2.2.2.1. Drilling fluid properties.** The study investigated the YP/PV and filtration properties of drilling mud formulations containing different additives, all at a consistent 1 % concentration. YP/PV is considered as the best indicator of mud rheology. Utilizing a FANN Viscometer, the mud's viscosity at varying rotational speeds (3 rpm, 6 rpm, 300 rpm, and

**Table 1**  
Design of Experiment for the study.

Sample type	Concentration	Aging	Category
Base	x	No aging and	Individual
KCl	1 %	aging at 100 °C	
[EMIM]Cl		and 150 °C at	
SiO <sub>2</sub> nanoparticles (Si-NP)		1000 psia for	
Polyetheramine (ATPE)		24 h	
Okra mucilage (O.M)			
Choline Chloride:Urea			
DES (CC DES)			
Citric acid:Glycerine			
NADES (CA NADES)			
Base+KCl+ [EMIM]Cl	0.5 % KCl + 0.5 % EMIM[Cl]		SET A
Base+KCl+ATPE	0.5 % KCl + 0.5 % ATPE		SET B
Base+KCl+ (Si-NP)	0.5 % KCl + 0.5 % Si- NP		SET C
Base+KCl+O.M	0.5 % KCl + 0.5 % O. M		SET D
Base+KCl+CC DES)	0.5 % KCl + 0.5 % CC DES		SET E
Base+KCl+CA NADES	0.5 % KCl + 0.5 % CA NADES		SET G
Base+KCl+ [EMIM]	0.33 %KCl+ 0.33 % [EMIM] Cl+ 0.33 % ATPE		SET AB
Base+KCl+ [EMIM]	0.25 %KCl+ 0.25 % [EMIM]Cl+ 0.25 % ATPE+ 0.25 %Si-Np		SET ABC
Base+KCl+ATPE+CC	0.33 %KCl+ 0.33 % ATPE+ 0.33 %CC DES		SET ABE
Base+KCl+ATPE+CC	0.25 %KCl+ 0.25 % ATPE+ 0.25 %CC DES+ 0.25 %Si-NP		SET ABCE
Base+KCl+ATPE+CC	0.20 %KCl+ 0.20 % ATPE+ 0.20 %CC DES+Si-NP+ 0.20 % Okra		SET ABCEF
Base+KCl+Si-Np+CA	0.33 %KCl+ 0.33 % Si-Np+ 0.33 %CA NADES		SET ACG

600 rpm) before and after subjecting it to elevated aging temperatures (100 °C and 150 °C) were measured. This procedure simulated the aging process using rolling oven in aging cells at 1000 psia and 100 °C and 150 °C typically experienced during drilling operations hot HT wells and allowed for the assessment of key rheological parameters such as Yield Point (YP) and Plastic Viscosity (PV). Additionally, the High Pressure High Temperature (HPHT) filtration test was conducted to evaluate the filtration properties of the drilling fluids under extreme conditions (pressure: 1000 psia, temperature: 400 °C). By measuring filtrate volume and mud cake thickness, this test provided insights into the additives' performance in mitigating filtration loss and maintaining wellbore stability in challenging drilling environments. In this study, mudcake thickness was measured using a digital vernier caliper with the least count of 0.01 mm. This instrument ensured precise and consistent measurements, contributing to the reliability of our data on drilling fluid performance.

Moreover, in this research, drilling fluid formulations were prepared using a five-spindle multimixer that conforms to American Petroleum Institute Specification 13 A, ensuring that all spindles rotate at 11,500 RPM ± 300 RPM. The fluids were mixed at this specified speed for 40 minutes to achieve a thorough and uniform dispersion of additives within the base fluid. Temperature was maintained at room temperature, as our laboratory is well insulated, ensuring stable ambient conditions throughout the mixing process.

### 2.2.3. Bentonite wafer preparation and Shale Inhibition Testing

**2.2.3.1. Linear swelling test.** In shale swelling investigations, many researchers favor bentonite wafers due to their similarity to shale in composition, both containing the 'smectite' group responsible for swelling. Obtaining shale 'true' core samples poses challenges as coring renders shale highly unstable, resulting in cores that are not entirely shale, often containing sandstone and limestone layers. Furthermore, conducting swelling inhibition experiments directly on shale outcrops is impractical, as they typically lack the smectite group crucial for shale swelling.

To address these challenges, this study employs refurbished bentonite pellets approximately 2.54 cm in diameter. These pellets are formed by compressing 11.5 g of Na-bentonite powder at 1600 psi using a hydraulic press. Before introducing the pellets into the Linear Swell Meter (LSM) environment, their thickness is precisely measured. Subsequently, the pellets are immersed in drilling mud samples, including both the base sample and samples containing inhibitor-based mud. The change in pellet thickness is then measured by the linear swell meter over a period of 24 hours, with measurements taken at intervals of 60 seconds. The utilization of the Grace HPHT Linear Swell Meter (M4600) marks a significant advancement in measuring the swelling phenomenon directly through the monitoring of sample thickness alterations. This study leverages the LSM to evaluate the inhibitory effects of a water-based drilling fluid on shale swelling. Comprising two integral components, namely the Wafer Compactor and the Linear Swell Meter (Model: M4600), the LSM facilitates the preparation of bentonite wafers using the Grace core/wafer compactor and subsequently conducts swelling tests to provide real-time data on swelling behavior.

**2.2.3.2. Shale recovery test.** The shale recovery test, alternatively known as the shale immersion or hot rolling dispersion test, serves to investigate the dispersion behavior of shale, which directly correlates with its stability. Initial steps involve separating shale cuttings by filtering through a 6 BSS mesh sieve and placing them on a 10 mesh sieve. These cuttings are then mixed with both the base fluid and NADES-based drilling fluids in aging cells and subjected to hot rolling in an oven to ensure thorough mud-cutting contact. Following a 16-h period, the cuttings are removed from the mud, resulting in shale disintegration and a subsequent decrease in cutting weight. Analysis of shale recovery entails filtering the cuttings from the mud using a finer mesh screen (40 mesh), followed by comprehensive washing, and drying in an oven. The recovered cuttings are then compared to ascertain the percentage of shale cuttings recovered from the initial weight. Shale outcrop samples from Niah, District of Miri, Sarawak, Malaysia, have been utilized, with their clay composition quantified via XRD to assess suitability for dispersion and shale recovery testing.

The clay mineralogy of the shale samples employed in this research is detailed below:

The shale sample consists of five types of clay minerals depicted by XRD results: Illite, Kaolinite, Chlorite, Vermiculite, and Mica.

- Illite comprises 18 % of the total clay mineral composition.
- Kaolinite is the most abundant, constituting 31 % of the sample.
- Chlorite makes up 22 % of the clay mineral content.
- Vermiculite accounts for 10 % of the total composition.
- Mica is present in the sample at a percentage of 19 %.

### 2.2.4. Characterizations

To understand the underlying mechanism of modification in drilling fluid properties and shale inhibition traits of various additives, Surface tensions, Zeta Potential and d-spacing will be estimated of modified and base bentonite wafers/mud.

**2.2.4.1. Surface tension.** Surface tension plays a critical role in

understanding the mechanism of additives in shale inhibition. Capillary action, driven by surface tension, facilitates the infiltration of water cations into the micropores of shale, affecting its stability. To assess the surface tension of drilling fluid samples, an Interfacial Tensiometer (IFT) is employed. By measuring the surface tension at the interface between the fluid and another medium, the IFT provides insights into interfacial behavior and the fluid's effectiveness in overcoming cohesive forces. Through this analysis, this study aims to understand how drilling fluids interact with shale formations.

**2.2.4.2. d-spacing analysis via XRD.** X-ray diffraction (XRD) analysis offers valuable insights into the intercalation of inhibitors into bentonite layers, further elucidating the mechanism of shale inhibition. By examining d-spacing, calculated by Bragg's equation, which represents interlayer spacing between clay layers, XRD reveals changes induced by inhibitors. This analysis conducted using D2 phaser functioning at 40 mA and 45 kV, with Cu-K $\alpha$  radiation ( $\lambda = 1.54059 \text{ \AA}$ ), enables researchers to investigate the interaction between inhibitors and clay mineral structures, shedding light on their effectiveness in modifying clay properties.

**2.2.4.3. Zeta potential.** Zeta potential measurement using the Malvern Zetasizer Nano ZSP instrument offers further understanding of the electrical charge and stability of colloidal particles in drilling mud. By analyzing Zeta Potential values, researchers gain insights into particle behavior within the fluid, which directly impacts shale stability. This analysis helps assess the likelihood of particle aggregation or dispersion, providing critical information for optimizing drilling fluid performance. Integrating data from surface tension, XRD, and Zeta potential measurements offers a comprehensive understanding of how additives interact with shale formations, ultimately enhancing shale inhibition strategies in drilling operations.

The standard equipment and apparatus used in this study have been illustrated in Fig. 1.

### 2.2.5. Data reliability and robustness

All experimental except d-spacing (XRD) measurements were conducted in triplicates to ensure data reliability and reproducibility. The results are presented as the  $\pm$  standard deviation (SD) in tabulated form for all additives (individually and in sets) at all non aging and aging temperatures. Standard deviation values were calculated to quantify variability within the dataset and are included in all reported results.

For clarity and consistency, SD values are presented in tabulated form rather than as error bars in graphical representations to avoid clutter in the figures. This approach ensures better visualization of trends while maintaining transparency regarding data variability.

## 3. Results and discussions

### 3.1. YP and PV ratio (YP/PV) analysis

The analysis of Yield Point to Plastic Viscosity (YP/PV) ratios is crucial for gauging the effectiveness of drilling fluid additives in optimizing hydraulic performance. When the YP/PV ratio falls within the range of 0.75–1 lb<sub>m</sub>/100ft<sup>2</sup>/cp, it indicates a balanced rheological profile that is conducive to superior hydraulic efficiency and overall operational performance during drilling activities. This range, depicted in red dashed line in Fig. 2, signifies an optimal balance between the fluid's yield point, which determines its resistance to flow, and its plastic viscosity, which influences the ease of fluid movement. When these properties align within the specified range, the drilling mud exhibits ideal characteristics for maintaining wellbore stability, facilitating effective cuttings transport, and promoting efficient hydraulic fracturing. Consequently, maintaining YP/PV ratios within this range is imperative for ensuring optimal hydraulic performance and overall operational

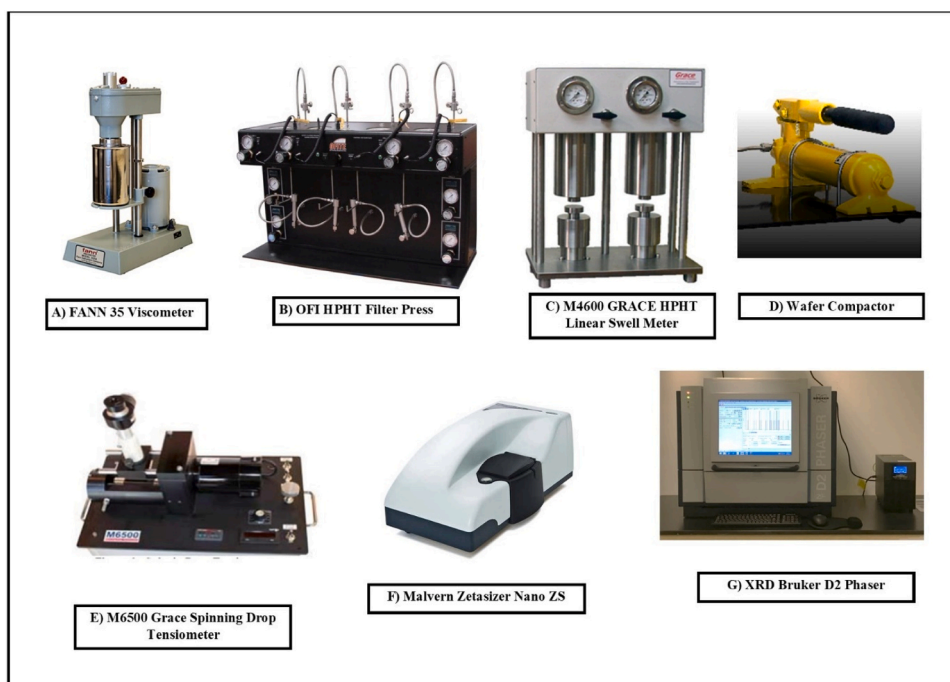


Fig. 1. Equipment utilized in this study A) FANN 35 Viscometer, B) OFI HPHT Filter Press C) M4600 Grace HPHT Linear Swell Meter D) Wafer Compactor E) M6500 Grace Spinning Drop Tensiometer F) Malvern Zetasizer Nano SZ G) XRD Bruker D2 phaser.

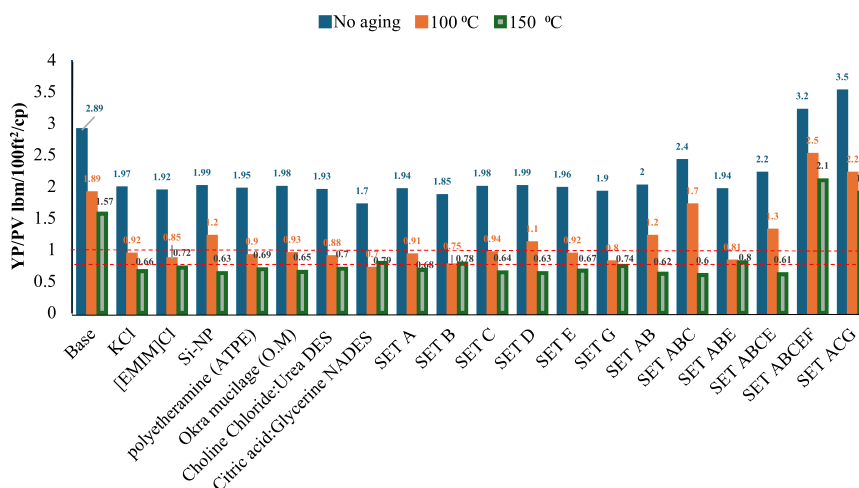


Fig. 2. YP/PV ratios of various additives reported for non-aged and aged samples at 100 °C and 150 °C.

success in drilling endeavors.

In this study, both individual additives and additive combinations (sets) are selected to determine their YP/PV ratios, a systematic approach is adopted to discern their impact on drilling fluid rheology. Initially, individual additives are subjected to testing under non-aging and aging scenarios. Each additive's YP/PV ratio is calculated to assess its performance independently. Following this, additive combinations, or sets, are formulated based on predetermined compositions, incorporating a combination of individual additives. These sets undergo similar testing procedures to evaluate their collective influence on drilling fluid behavior. By comparing the YP/PV ratios of both individual additives and additive combinations, researchers can identify synergistic effects and determine the most effective formulations for achieving desired rheological properties.

Among the individual additives, Citric acid:Glycerine NADES (CA NADES) stands out as the top performer, securing the 1st position overall. With YP/PV ratios of 1.7, 0.7, and 0.79 for non-aging, aging at

100 °C, and aging at 150 °C conditions, respectively, CA NADES consistently demonstrates promising rheological behavior across different scenarios closest to the optimum range. Following closely behind, [EMIM]Cl, Choline Chloride:Urea DES (CC DES), and Polyetheramine (ATPE) exhibit the 2nd, 3rd, and 4th best performances, respectively, maintaining favorable YP/PV ratios closest to the optimum range for both aged and non-aged samples as shown in Fig. 2. However, SiO<sub>2</sub> nanoparticles, Okra mucilage (O.M), and KCl show overall improvements in the YP/PV ratios, but their performance ranks as the least favorable among all individual additives.

When assessing the performance of sets based on YP/PV ratios, distinct trends emerge, revealing varying degrees of effectiveness in optimizing drilling fluid rheology. Leading the rankings is SET B (Base+KCl+ATPE), securing the top position among all sets. With consistently favorable YP/PV ratios across different conditions, SET B demonstrates promising rheological behavior, positioning it as a standout combination for enhancing drilling fluid performance.

Following closely behind is SET G (Base+KCl+CA NADES), which exhibits favorable YP/PV ratios and secures the 2nd position among the sets. Its performance indicates potential in improving fluid behavior and hydraulic efficiency during drilling operations. In contrast, sets like SET ABE (Base+KCl+ATPE+CC DES) and SET A (Base+KCl+EMIM[Cl]) gave overall 3rd position, indicating combination of DES with KCl and amines with DES can be good drilling fluid additives. Meanwhile, SET C (Base+KCl+Si-NP) and SET E (Base+KCl+CC DES) showed an average performance. On the other hand, combinations like SET ABCE (Base+KCl+ATPE+CC DES+Si-NP) and SET ABCEF (Base+KCl+ATPE+CC DES+Si-NP+Okra) demonstrated negative performance resulting into YP/PV ratios too high which result in the operational difficulties. This shows, additives which may show superior performance individually may not give the same results if added altogether.

Overall top three best performing additives in terms of favourable mud hydraulics and cutting transportation ability are Citric Acid:Gly NADES, SET B (0.5 % KCl + 0.5 % ATPE) and set G (0.5 % KCl + 0.5 % [EMIM]Cl) respectively which shows even addition of KCl in small concentration can enhance the performance of amine and ionic liquids. This comprehensive evaluation enables the identification of combinations that contribute most effectively to enhancing drilling fluid performance and operational efficiency. The mechanism behind working of these additives will be elaborated further in the subsequent Sections 3.6–3.8.

### 3.2. Mudcake thickness

Analyzing mudcake thickness provides valuable insights into the effectiveness of drilling fluid additives in controlling filtration and formation damage during drilling operations. Mudcake thickness serves as an indicator of the ability of the drilling fluid to form a protective barrier on the wellbore wall, preventing fluid invasion into the formation while facilitating efficient drilling. Ideally, a mudcake thickness within the desired range ensures optimal wellbore stability and minimizes fluid loss into the formation, thereby enhancing drilling efficiency and reducing operational challenges. In this study, both individual additives and additive combinations (sets) were evaluated to determine their impact on mudcake thickness. The thickness of the mudcake formed by each additive or combination was measured under non-aging and aging conditions to assess their effectiveness in controlling fluid invasion and formation damage. By comparing the mudcake thicknesses of individual additives and additive combinations, this study could identify formulations that effectively mitigate fluid invasion and maintain wellbore integrity.

The analysis of mudcake thickness reveals distinct trends in the performance of individual additives and additive combinations. Among the individual additives, CA NADES exhibited the most favorable performance reducing mudcake thickness to 42.85 %, consistently forming

thinner mudcakes across different scenarios. Following closely behind were [EMIM]Cl and Choline Chloride DES which resulted into a 25 % reduction in mudcake thickness each. Okra mucilage (O.M) and nanoparticles gave average performance, when assessing the performance of additive combinations (sets), SET B (Base+KCl+ATPE), emerged as the top performer with 39.28 % reduction in mudcake thickness, forming thinner mudcakes consistently across different conditions. Following closely behind SET G (Base+KCl+CA NADES), which showed 35.71 % reduction in mudcake thickness. Meanwhile, SET ABCEF and ACG gave negative performance as shown in Fig. 3.

Conversely, NADES, SET B and SET G were top three additives in reducing mudcake thicknesses, indicating potential challenges in controlling fluid invasion and formation damage while set ABCEF and ACG were the worst performing additives. Overall, the analysis highlights the significance of selecting additive combinations that effectively mitigate fluid invasion and maintain optimal mudcake thickness, thereby enhancing drilling efficiency and reducing operational challenges.

### 3.3. Filtrate volume

Filtrate volume in drilling fluids serves as a critical parameter indicative of fluid loss and invasion into the formation, directly influencing wellbore stability and drilling efficiency. Lower filtrate volumes signify better control over fluid loss, leading to improved wellbore integrity and reduced formation damage. In this study, the effectiveness of various additives in reducing filtrate volume was evaluated across different conditions.

Among the additives tested, CA NADES exhibited remarkable efficacy in reducing filtrate volume, consistently outperforming other additives across all conditions. With a reduction of approximately 40.32 %, 43.33 %, and 38.71 % in filtrate volume compared to the base sample under non-aging, 100 °C, and 150 °C conditions, respectively, CA NADES demonstrated superior performance in controlling fluid loss and invasion into the formation. Following closely behind, [EMIM]Cl highlighted significant effectiveness in reducing filtrate volume, particularly under higher temperature conditions. With a reduction of approximately 29.63 %, 30.00 %, and 29.03 % in filtrate volume compared to the base sample under non-aging, 100 °C, and 150 °C conditions, respectively, [EMIM]Cl proved to be a reliable additive for mitigating fluid loss during drilling operations. ATPE was the third best performing additive in terms of filtrate volume.

Among the sets examined, SET B emerges as the top performer, displaying the highest reduction in filtrate volume across all conditions. With reductions of approximately 37.04 %, 36.67 %, and 38.71 % under non-aging, 100 °C, and 150 °C conditions, respectively, SET B demonstrates substantial effectiveness in mitigating fluid loss and invasion into the formation as shown in Fig. 4. Following closely behind, SET G also demonstrates notable reductions in filtrate volume, with reductions of

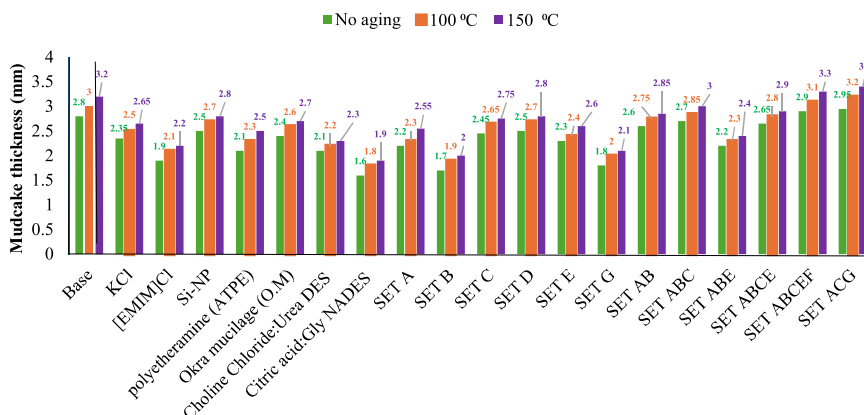


Fig. 3. Mudcake thickness of various additives based mud reported for non-aged and aged samples at 100 °C and 150 °C.

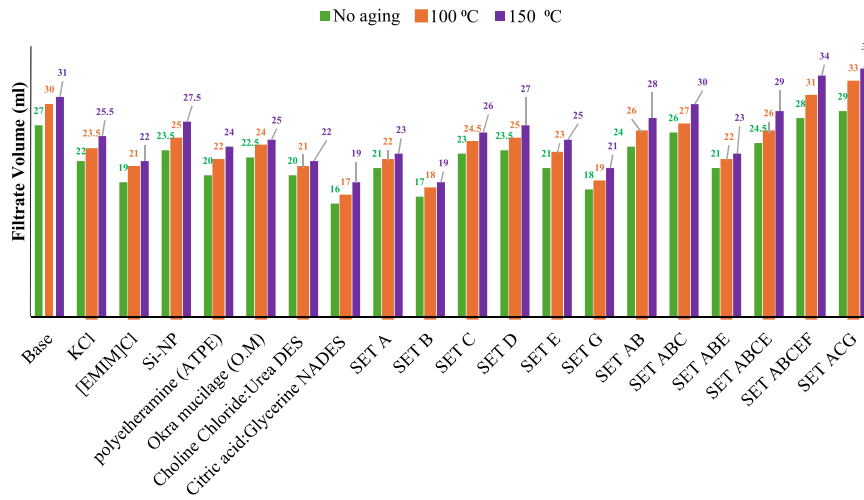


Fig. 4. Filtrate volume of various additives based mud reported for non-aged and aged samples at 100 °C and 150 °C.

approximately 33.33 %, 36.67 %, and 32.26 % under non-aging, 100 °C, and 150 °C conditions, respectively. These findings highlight the robust fluid loss control properties of both SET B and SET G, positioning them as top-performing additive combinations in drilling fluid formulations. Conversely, SET ACG exhibits relatively poorer performance, with an increase in filtrate volume compared to the base sample. With reductions of approximately -38.10 %, -50.00 %, and -41.94 % under non-aging, 100 °C, and 150 °C conditions, respectively, SET ACG demonstrates deficiencies in fluid loss control capabilities, leading to elevated filtrate volumes. This outcome underscores the importance of thorough evaluation and optimization of additive combinations to ensure effective fluid loss control and formation damage mitigation in drilling operations. The deficient performance of SET ACG and SET ABCEF in reducing filtrate volume could be attributed to several factors. One possible explanation is the composition and interaction of the additives within the set. It is plausible that the combination of additives in SET ACG may not have synergized effectively to control fluid loss. Certain additives within the set may have conflicting properties or may not interact optimally with one another, leading to reduced efficacy in mitigating fluid invasion into the formation.

Overall, NADES, SET B and SET G gave top-tier performances which are in accordance with the mudcake thickness results. Further elaboration of the mechanism has been carried out in the subsequent sections.

### 3.4. Linear clay swelling

Clay swelling presents a formidable challenge in shale drilling operations, impacting wellbore stability and overall drilling efficiency. Effective management of clay swelling in shale is paramount to mitigate risks associated with formation damage and wellbore instability. Among the additives evaluated for their ability to reduce clay swelling, Citric acid:Glycerine NADES emerges as the top-performing additive across all temperatures, exhibiting the highest reduction in linear swelling compared to the base sample. At 25°C, Citric acid:Glycerine NADES demonstrates a remarkable percentage reduction of 76.1 %, followed by [EMIM]Cl with 65.7 % and Choline Chloride:Urea DES with 62.7 % at 25°C as shown in Fig. 5. These additives exhibit significant effectiveness in controlling clay swelling at lower temperatures, contributing to improved wellbore stability. At elevated temperatures of 100 °C and 150 °C, Citric acid:Glycerine NADES maintains its dominance, highlighting the highest reduction in linear swelling compared to the base sample, with impressive percentage reductions of 79.1 % and 73.6 %, respectively. [EMIM]Cl and Choline Chloride:Urea DES also maintain their effectiveness across all temperatures, further emphasizing their importance in mitigating formation-related challenges and optimizing drilling operations in shale formations.

Among sets, SET B and SET G showed dominance with 68.7 % and 59.7 % reduction in linear swelling at 25°C. SET B and SET G maintained the same dominance at elevated temperatures as well. SET C and SET E gave optimized and also similar performance at all temperatures as

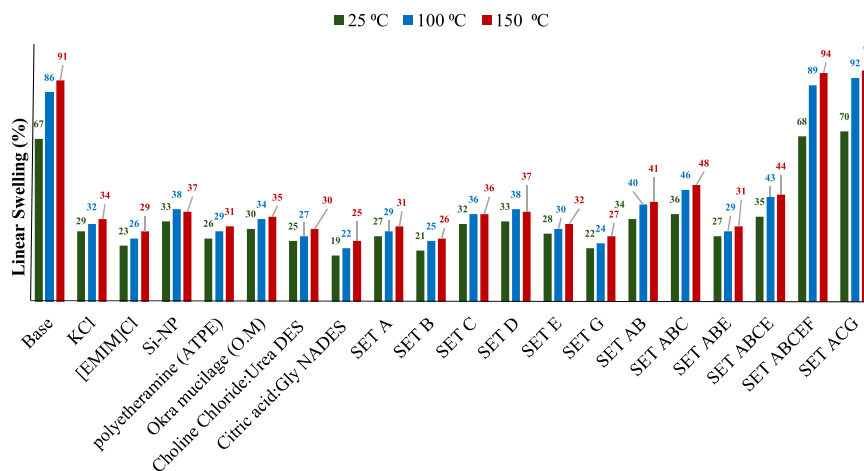


Fig. 5. Linear Swelling of bentonite clay resulted by various additives based mud reported at 25 °C,100 °C and 150 °C.

shown in Fig. 5. However, SET ABCEF and SET ACG, gave negative improvement in clay swelling. Overall, CA NADES, SET B and SET G were the best performing inhibitors in terms of improving clay swelling, and thus can be regarded as potential shale inhibitors. The more discussion, to elucidate the working of these additives has been carried out in the subsequent sections.

### 3.5. Shale recovery

Shale recovery is a critical aspect of drilling operations, particularly in formations where shale constitutes a sizable portion of the rock matrix. When shale formations are encountered during drilling, they tend to destabilize upon contact with water-based drilling fluids. Shale is inherently hydrophilic, meaning it has an affinity for water, and as such, when exposed to water-based mud, it absorbs moisture, causing it to swell and disintegrate. This process, known as hydration, leads to the release of fine particles and clay minerals from the shale matrix, which can then disperse into the drilling fluid. As a result, shale recovery becomes challenging as shale disintegration can lead to wellbore instability, increased drilling fluid viscosity, bit balling and other operational issues. Therefore, assessing shale recovery is crucial for understanding the effectiveness of drilling fluid additives in mitigating shale destabilization and improving overall drilling efficiency.

Similarly to the results of linear swelling, CA NADES resulted in the most shale recovery which shows it has neutralized the charge on clay surface by making a strong hydrogen bond with it, stabilizing it against hydration. CA NADES resulted in 51.68 % improved recovery, [EMIM] Cl gave 46.9 % recovery while DES gave 45.5 % recovery at 25 °C as shown in Fig. 6. The same trend can be observed at elevated temperatures for these three additives mentioned above. Following the pursuit, in the individual category Si-NP was the worst performing while KCl, ATPE and O.M gave average results. In the category of sets, SET B and SET G resulted into 50 % and 48.1 % improved shale recovery at 25 °C. Predictably, same as other drilling fluid properties, SET ACG and SET ABCEF gave worst performing results for shale recovery as also depicted in Fig. 6.

### 3.6. Surface tension

Surface tension plays a crucial role in shale inhibition mechanisms through its influence on capillary pressure and subsequent effects on shale hydration and swelling. Capillary pressure refers to the pressure difference across the interface between two immiscible fluids in a porous

medium, such as drilling fluid and formation fluids within shale formations. This pressure gradient is influenced by surface tension, among other factors. When the surface tension is high, as in the case of drilling fluids with additives that increase surface tension, the capillary pressure between the fluid and shale surfaces also tends to be higher. This elevated capillary pressure can enhance the invasion of drilling fluid into the shale matrix, leading to increased shale hydration and swelling. On the other hand, additives that reduce surface tension effectively lower capillary pressure. By decreasing the surface tension of the drilling fluid, these additives weaken the capillary forces, thus mitigating the invasion of drilling fluid into the shale pores. Consequently, the hydration and swelling of shale formations are minimized. Shale formations are prone to instability when exposed to drilling fluids with high invasion tendencies, leading to issues such as wellbore collapse, stuck pipe, and lost circulation. Additives that effectively inhibit shale hydration and swelling by affecting capillary pressure indirectly help mitigate these risks, enhancing drilling efficiency and wellbore integrity.

In examining the percentage reductions in surface tension compared to the base sample, varying degrees of alteration in interfacial properties across different additives were observed. Citric acid:Glycerine NADES emerges as the most impactful additive, with a substantial reduction in surface tension of approximately 20.2 %. Following closely behind are SET B and SET G, exhibiting notable reductions of about 18.8 % and 17.4 %, respectively as shown in Fig. 7. These additives demonstrate significant potential in modifying the drilling fluid’s interfacial characteristics, which can play a crucial role in enhancing shale stability and mitigating drilling challenges associated with shale hydration and swelling. Conversely, additives such as SET ACG and SET ABCEF show minimal reductions in surface tension, suggesting lesser influence on interfacial properties compared to others in the study. The results of surface tension follow the trend observed for clay swelling and shale recovery results.

In summary, the surface tension of drilling fluids indirectly impacts capillary pressure, which in turn influences shale hydration and swelling behavior. Additives that lower surface tension play a crucial role in mitigating capillary pressure, thereby inhibiting shale hydration, and swelling and promoting wellbore stability during drilling operations.

### 3.7. Zeta Potential

The zeta potential, which reflects the magnitude of the electrostatic charge at the interface between the clay particles and the surrounding fluid, plays a crucial role in determining the interaction between these

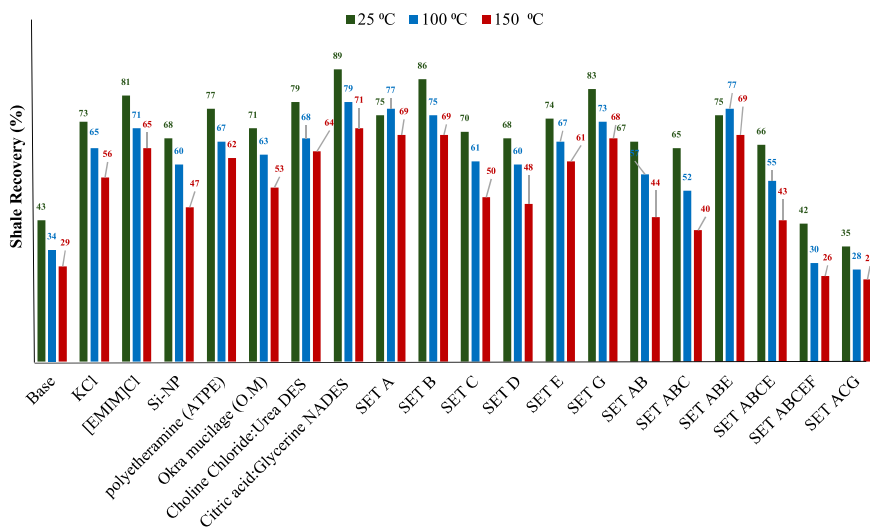


Fig. 6. Shale recovery test/hot rolling dispersion test of Miri, Sarawak shale resulted by various additives based mud reported at 25 °C, 100 °C and 150 °C after hot rolling at 1000 psia for 24 h.

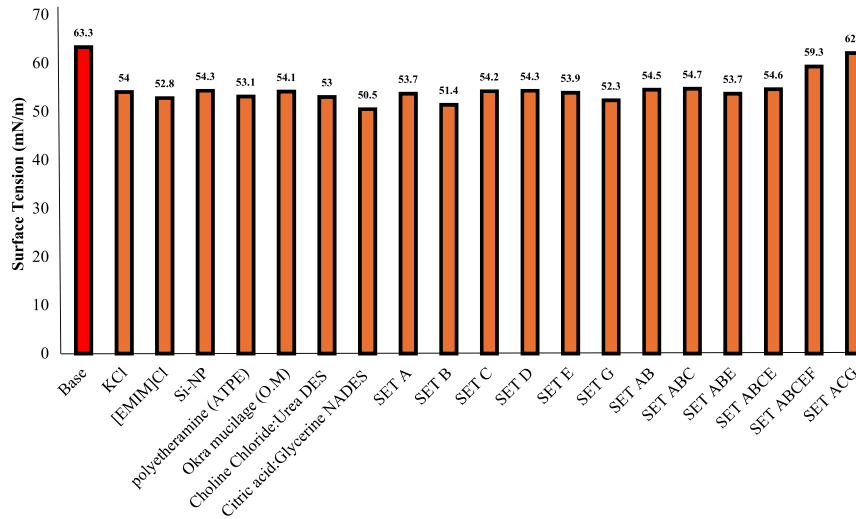


Fig. 7. Surface tension of drilling mud resulted by various additives reported at 25 °C.

particles. When an additive reduces the zeta potential of the drilling mud, it effectively diminishes the strength of the electrostatic repulsion between the clay particles. This reduction in repulsion allows the particles to approach each other more closely, leading to the formation of aggregates or flocs. As a result, the aggregation of clay particles reduces their individual mobility and tendency to disperse, ultimately enhancing shale stability. Furthermore, the mechanism by which additives lower the zeta potential involves their interaction with the clay surfaces. These additives may adsorb onto clay particles, altering their surface properties and modifying the distribution of surface charges. This adsorption process can effectively neutralize or shield the charges on the clay surfaces, leading to a decrease in the zeta potential of the system and thus stabilizing clay against hydration.

Moreover, additives that lower the zeta potential tend to decrease the thickness of the electrical double layer (EDL) surrounding the clay particles. A thinner EDL implies a weaker repulsive force between the particles, facilitating their aggregation and minimizing the likelihood of clay swelling and dispersion. By promoting particle aggregation and reducing swelling tendencies, these additives contribute to the overall stabilization of shale formations.

Among the additives, Citric acid:Glycerine NADES exhibits the highest reduction, with approximately 46.5 % lower ZP compared to the base sample. This significant reduction underscores the strong interaction of NADES with clay particles, leading to improved shale stability. KCl demonstrates a notable reduction of approximately 28.6 % compared to the base ZP, indicating its potential in improving shale

stability. Similarly, [EMIM]Cl exhibits a reduction of around 35.9 %, suggesting its effectiveness in mitigating electrostatic forces and enhancing shale inhibition as shown in Fig. 8. SiO<sub>2</sub> nanoparticles (Si-NP) display a reduction of approximately 19.4 % compared to the base ZP, indicating moderate effectiveness in promoting shale stability. Polyetheramine (ATPE) and Okra mucilage (O.M) also show substantial reductions of about 33.3 % and 28.2 %, respectively, emphasizing their roles in enhancing shale inhibition properties. Choline Chloride:Urea DES demonstrates a reduction of around 33.2 %, further highlighting its potential as a shale stabilizer. Moving to additive sets, SET B stands out with a substantial reduction of about 42.6 % compared to the base ZP, indicating its efficacy in enhancing shale inhibition. Similarly, SET ABC shows a significant reduction of approximately 41.9 %, suggesting synergistic effects among the additives in promoting shale stability.

### 3.8. d-spacing (XRD)

The d-spacing measurements obtained from XRD provide valuable insights into the intercalation of additives within the clay layers of the shale, indicating the potential improvement in drilling fluid properties. A lower d-spacing value of drilling mud suggests additive has successfully expelled the water out from clay layers, indicating enhanced interaction and potential enhancement in drilling fluid performance. Analyzing the results, it is evident that several additives lead to a reduction in d-spacing compared to the base sample. Notably, Citric acid:Glycerine NADES exhibits the lowest d-spacing value among all

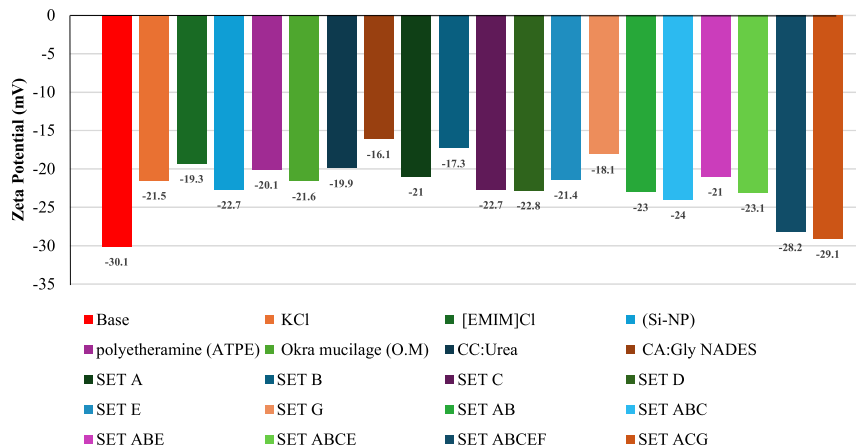


Fig. 8. Zeta Potential of of drilling mud resulted by various additives reported at 25 °C.

additives, indicating effective intercalation into the clay layers. The reduction in d-spacing for NADES signifies its strong affinity for the clay surfaces and efficient expulsion of water molecules, contributing to improved shale stability and drilling fluid performance, with a reduction of approximately 32.7 % compared to the base sample as shown in Fig. 9.

Similarly, SET B, comprising a combination of various additives, also demonstrates a significant reduction in d-spacing compared to the base sample, with a decrease of approximately 27.4 %. This suggests that the combination of additives in SET B effectively interacts with the clay layers, leading to improved properties of the drilling fluid. Additionally, SET G, consisting of other additives, also shows a notable reduction in d-spacing, with a decrease of approximately 30.6 %, indicating favorable intercalation and potential enhancement in drilling fluid performance. Conversely, additives like SiO<sub>2</sub> nanoparticles (Si-NP) and polyetheramine (ATPE) show relatively higher d-spacing values compared to the base sample, with increases of approximately 0.8 % and 3.4 %, respectively. While these additives still contribute to some degree of intercalation, their effectiveness in modifying the clay structure may be comparatively lower than other additives.

Furthermore, SET ABCEF and SET ACG exhibit the highest d-spacing values among all additives and sets, with increases of approximately 9.8 % and 15.1 %, respectively, indicating less effective intercalation or even possible agglomeration within the clay layers. This suggests that despite the inclusion of multiple additives, these combinations may not be as efficient in modifying the clay structure to enhance drilling fluid properties. Considering the reduction in d-spacing, additives leading to lower values, such as Citric acid:Glycerine NADES and certain combinations like SET B and SET G, are likely to contribute to improved shale stability and overall drilling fluid performance. The reduction in d-spacing reflects the enhanced intercalation of additives within the clay layers, facilitating better control over shale swelling and overall drilling fluid behavior.

### 3.9. Data reliability

The SD values for all measured properties remained within an acceptable range across all experimental conditions, ensuring the reliability and reproducibility of the data. For most formulations, low SD values indicate minimal variability between triplicate measurements, demonstrating the consistency of the experimental procedures.

Across different temperatures, a slight increase in SD was observed at higher temperatures (100 °C and 150 °C), particularly for rheological properties (PV, YP) and filtration characteristics (mudcake thickness,

filtrate volume). This variation is expected due to thermal effects on fluid stability, which can lead to minor fluctuations in viscosity, fluid loss, and shale interactions. Despite this, the SD values remained within acceptable limits, confirming that temperature-induced variations were not significant enough to compromise data reliability as shown in Tables 2a 2b.

For shale stability tests, linear swelling and shale recovery exhibited slightly higher SD values compared to rheological properties, particularly in formulations with multiple additives (e.g., SET ABCEF, SET ACG). This can be attributed to complex interactions between inhibitors and shale particles, leading to minor inconsistencies in swelling reduction and dispersion control. However, the overall SD values remained low, ensuring that observed trends are representative of actual performance differences rather than experimental errors.

Furthermore, for zeta potential and surface tension measurements, SD values were consistently low across all formulations at 25 °C, confirming stable electrochemical interactions within the fluid systems as shown in Tables 3a 3b. The low variability in these measurements suggests that surface-active additives provided uniform effects on fluid properties.

Overall, the reported SD values support the statistical robustness of the results, ensuring that observed differences between formulations are meaningful and not due to random experimental variations. The use of triplicate measurements and controlled testing conditions further reinforces the reliability of the findings.

## 4. Summary and discussion

Shale instability poses a significant challenge in drilling operations, primarily due to the presence of clay minerals in shale formations. Clay mineral, such as montmorillonite, is hygroscopic in nature, meaning they have a strong affinity for water molecules. When exposed to drilling fluids, these clay minerals can absorb water and undergo hydration, leading to swelling and destabilization of the shale matrix. This swelling behavior can cause various issues, including borehole instability, well-bore collapse, and increased drilling fluid filtration.

To mitigate shale instability, various additives are incorporated into drilling fluids to inhibit clay hydration and swelling. Each additive interacts with the clay minerals through different mechanisms, ultimately leading to improved shale stability. For instance, KCl acts as a shale inhibitor by exerting electrostatic forces of attraction on clay minerals, effectively reducing their ability to absorb water molecules and swell. This mechanism helps maintain the integrity of the shale formation and prevents borehole instability. Similarly, other additives like [EMIM]Cl,

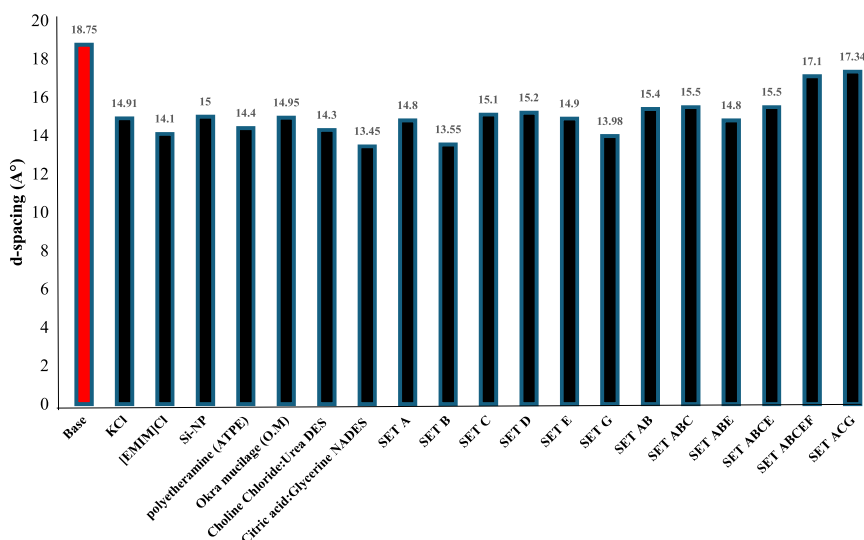


Fig. 9. d-spacing of modified bentonite wafers resulted by various additives reported at 25 °C.

**Table 2a**  
Standard Deviation of experiments conducted for 'individual' additives.

Category	Additive/Set	Temperature (°C)	Linear Swelling (%) ± SD	Shale Recovery (%) ± SD	YP (lb/100 ft <sup>2</sup> ) ± SD	PV (cP) ± SD	Mudcake Thickness (mm) ± SD	Filtrate Volume (mL) ± SD
Individual	Base	25	± 1.2	± 0.05	± 0.3	± 0.4	± 0.1	± 0.8
		100	± 1.5	± 0.02	± 0.4	± 0.5	± 0.2	± 1.0
		150	± 1.8	± 0.1	± 0.9	± 1.2	± 0.2	± 1.2
	KCl	25	± 1.0	± 0.5	± 0.3	± 0.4	± 0.1	± 0.7
		100	± 1.3	± 0.6	± 0.4	± 0.5	± 0.2	± 0.9
		150	± 1.5	± 0.7	± 0.9	± 0.8	± 0.2	± 1.0
	[EMIM]Cl	25	± 1.1	± 0.6	± 0.3	± 0.4	± 0.1	± 0.8
		100	± 1.4	± 0.7	± 0.4	± 0.5	± 0.2	± 0.9
		150	± 1.6	± 0.8	± 0.9	± 0.6	± 0.2	± 1.1
	SiO <sub>2</sub> Nanoparticles (Si-NP)	25	± 1.0	± 0.5	± 0.3	± 0.4	± 0.1	± 0.7
		100	± 1.3	± 0.6	± 0.4	± 0.7	± 0.2	± 0.9
		150	± 1.5	± 0.7	± 1.2	± 1.4	± 0.2	± 1.0
	Polyetheramine (ATPE)	25	± 1.2	± 0.6	± 0.3	± 0.4	± 0.1	± 0.8
		100	± 1.4	± 0.7	± 0.4	± 0.5	± 0.2	± 0.9
		150	± 1.7	± 0.8	± 1.1	± 0.6	± 0.2	± 1.1
	Okra Mucilage (O.M)	25	± 1.1	± 0.6	± 0.3	± 0.4	± 0.1	± 0.8
		100	± 1.4	± 0.7	± 0.4	± 0.5	± 0.2	± 0.9
		150	± 1.6	± 0.8	± 0.5	± 0.6	± 0.2	± 1.1
	Choline Chloride:Urea DES	25	± 1.2	± 0.7	± 0.3	± 0.4	± 0.1	± 0.9
		100	± 1.5	± 0.8	± 0.4	± 0.5	± 0.2	± 1.0
		150	± 1.7	± 0.9	± 0.5	± 0.6	± 0.2	± 1.2
	Citric Acid:Glycerine NADES	25	± 1.1	± 0.6	± 0.3	± 0.4	± 0.1	± 0.7
		100	± 1.3	± 0.7	± 0.4	± 0.5	± 0.2	± 0.9
		150	± 1.5	± 0.8	± 1.3	± 1.4	± 0.2	± 1.0

**Table 2b**  
Standard Deviation of experiments conducted for 'sets' additives.

Set	Temperature (°C)	Linear Swelling (%) ± SD	Shale Recovery (%) ± SD	YP (lb/100 ft <sup>2</sup> ) (%) ± SD	PV (cP) ± SD	Mudcake Thickness (mm) ± SD	Filtrate Volume (mL) ± SD
SET A	25	± 0.4	± 0.3	± 1.0	± 0.6	± 0.1	± 0.7
	100	± 0.5	± 0.4	± 1.3	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.5	± 0.8	± 0.2	± 1.0
SET B	25	± 0.4	± 0.3	± 1.1	± 0.6	± 0.1	± 0.8
	100	± 0.5	± 0.4	± 1.4	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.6	± 0.8	± 0.2	± 1.1
SET C	25	± 0.4	± 0.3	± 1.0	± 0.6	± 0.1	± 0.7
	100	± 0.5	± 0.4	± 1.3	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.5	± 0.8	± 0.2	± 1.0
SET D	25	± 0.4	± 0.3	± 1.1	± 0.6	± 0.1	± 0.8
	100	± 0.5	± 0.4	± 1.4	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.6	± 0.8	± 0.2	± 1.1
SET E	25	± 0.4	± 0.3	± 1.0	± 0.6	± 0.1	± 0.7
	100	± 0.5	± 0.4	± 1.3	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.5	± 0.8	± 0.2	± 1.0
SET G	25	± 0.4	± 0.3	± 1.0	± 0.6	± 0.1	± 0.7
	100	± 0.5	± 0.4	± 1.3	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.5	± 0.8	± 0.2	± 1.0
SET AB	25	± 0.4	± 0.3	± 1.1	± 0.6	± 0.1	± 0.8
	100	± 0.5	± 0.4	± 1.4	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.6	± 0.8	± 0.2	± 1.1
SET ABC	25	± 0.4	± 0.3	± 1.0	± 0.6	± 0.1	± 0.7
	100	± 0.5	± 0.4	± 1.3	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.5	± 0.8	± 0.2	± 1.0
SET ABE	25	± 0.4	± 0.3	± 1.1	± 0.6	± 0.1	± 0.8
	100	± 0.5	± 0.4	± 1.4	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.6	± 0.8	± 0.2	± 1.1
SET ABCE	25	± 1.4	± 1.3	± 1.0	± 0.6	± 0.1	± 0.7
	100	± 1.5	± 1.4	± 1.3	± 0.7	± 0.2	± 0.9
	150	± 1.6	± 0.5	± 1.5	± 0.8	± 0.2	± 1.0
SET ABCEF	25	± 1.9	± 1.7	± 1.1	± 0.6	± 0.1	± 0.8
	100	± 1.5	± 1.4	± 1.4	± 0.7	± 0.2	± 0.9
	150	± 1.3	± 1.3	± 1.6	± 0.8	± 0.2	± 1.1
SET ACG	25	± 0.4	± 0.3	± 1.0	± 0.6	± 0.1	± 0.7
	100	± 0.5	± 0.4	± 1.3	± 0.7	± 0.2	± 0.9
	150	± 0.6	± 0.5	± 1.5	± 0.8	± 0.2	± 1.0

functionalized SiO<sub>2</sub> nanoparticles (Si-NP), polyetheramine (ATPE), and Okra mucilage (O.M) also contribute to shale inhibition through distinct mechanisms. [EMIM]Cl, an ionic liquid, interacts with clay minerals through neutralizing surface charge by cation exchange and surface

adsorption, disrupting the hydration process and reducing shale swelling. SiO<sub>2</sub> nanoparticles provide mechanical reinforcement to the shale matrix, preventing excessive swelling and improving overall stability and the functional group may also alter the surface properties of

**Table 3a**  
Standard Deviation of Zeta Potential and Surface Tension testing conducted for 'individual' additives.

Category	Additive/Set	Temperature (°C)	Zeta Potential (mV) ± SD	Surface Tension (mN/m) ± SD
Individual	Base	25	± 0.5	± 0.4
	KCl	25	± 0.6	± 0.5
	[EMIM]Cl	25	± 0.7	± 0.5
	SiO <sub>2</sub> Nanoparticles (Si-NP)	25	± 0.6	± 0.4
	Polyetheramine (ATPE)	25	± 0.7	± 0.5
	Okra Mucilage (O. M)	25	± 0.6	± 0.4
	Choline Chloride: Urea DES	25	± 0.8	± 0.6
	Citric Acid: Glycerine NADES	25	± 0.7	± 0.5

**Table 3b**  
Standard Deviation of Zeta Potential and Surface Tension testing conducted for 'sets' additives.

Set	Temperature (°C)	Zeta Potential (mV) ± SD	Surface Tension (mN/m) ± SD
SET A	25	± 0.6	± 0.5
SET B	25	± 0.7	± 0.5
SET C	25	± 0.6	± 0.4
SET D	25	± 0.7	± 0.5
SET E	25	± 0.6	± 0.5
SET G	25	± 0.6	± 0.4
SET AB	25	± 0.7	± 0.5
SET ABC	25	± 0.6	± 0.4
SET ABE	25	± 0.7	± 0.5
SET ABCE	25	± 0.8	± 0.6
SET ABCEF	25	± 0.8	± 0.6
SET ACG	25	± 0.7	± 0.5

the clay. Polyetheramine and Okra mucilage act as shale inhibitors by bonding with clay surface, preventing water intrusion and maintaining shale integrity.

Analyzing the results of surface tension, zeta potential, and d-spacing measurements further elucidates the mechanisms through which these additives inhibit shale instability. Lower surface tension values indicate modifying the capillary pressure, leading to decreased water penetration into the shale matrix. Additives that exhibit lower zeta potential values effectively reduce the electrostatic repulsion between clay particles, promoting closer packing and minimizing clay swelling. Additionally, additives that cause a reduction in d-spacing values indicate effective intercalation into the clay layers, further hindering water absorption and shale destabilization.

Some sets demonstrate superior performance in inhibiting shale instability compared to individual additives alone, which can be attributed to the synergistic effects of the combined additives within the sets. For instance, SET A (Base + KCl + [EMIM]Cl) and SET G (Base + KCl + ATPE) exhibit enhanced shale inhibition due to the complementary mechanisms of their constituent additives. In SET A, the inclusion of KCl and EMIM[Cl] contributes to shale inhibition through different mechanisms. KCl, known for its ability to reduce clay swelling by electrostatic forces, works by increasing the ionic strength of the drilling fluid, thereby decreasing the water activity and clay hydration. On the other hand, EMIM[Cl], an ionic liquid, interacts with clay minerals through surface adsorption and cation exchange, inhibiting clay swelling and hydration. The combined action of KCl and EMIM[Cl] in SET A synergistically enhances shale stability by effectively reducing water penetration and clay swelling.

The combined effects of KCl and ATPE in SET G result in improved shale inhibition compared to individual additives. When considering additive combinations, such as SETs, the synergistic effects of individual additives contribute to enhanced shale inhibition. In SET B, the synergistic mechanism is achieved by the complementary actions of KCl and ATPE. KCl promotes ion exchange and electrostatic adsorption on the clay surfaces, thereby reducing the hydration forces that lead to shale swelling. Concurrently, ATPE forms a protective barrier through hydrogen bonding interactions with the clay, which not only reinforces the structural integrity of the shale but also facilitates intercalation between the clay layers. In SET G, a similar dual mechanism is observed, where KCl again acts to neutralize the surface charges and mitigate hydration through cation exchange, while [EMIM]Cl further enhances shale stability by establishing hydrogen bonds with clay particles. The combination of these mechanisms electrostatic stabilization by KCl and the formation of a cohesive interfacial layer through hydrogen bonding by ATPE or [EMIM]Cl results in improved inhibition of shale swelling. This dual-action approach, supported by relevant literature and our experimental observations, underscores the enhanced performance of SET B and SET G in maintaining wellbore stability and optimizing drilling fluid properties.

In this study, certain mixed additive combinations, notably SET ABCEF, exhibited poorer performance relative to other formulations. It was hypothesized that the excessive number of additives and their higher combined concentration may lead to conflicting interactions among the components. In such over-saturated formulations, the intended synergistic effects are compromised by additive interference. Specifically, excessive additive loading can result in aggregation and micro-scale pore blockage, which obstructs the effective adsorption and intercalation processes necessary for optimal shale inhibition. The literature supports the notion that each additive has an optimal concentration range, and deviations from this range may trigger antagonistic interactions. Consequently, the failure mechanisms in SET ABCEF likely stem from the imbalance between synergistic and interfering effects, resulting in a reduced capacity to maintain wellbore stability.

In conclusion, the comprehensive analysis of additives and additive combinations provides valuable insights into their roles in shale inhibition during drilling operations. By understanding the mechanisms through which these additives interact with clay minerals, drilling fluid formulations can be optimized to achieve better shale stability, minimize borehole instability, and enhance overall drilling performance.

## 5. Conclusions

The following conclusions can be drawn from this study:

1. The research evaluated the effectiveness of different additives in inhibiting shale swelling and improving drilling fluid properties. Additives such as KCl, [EMIM]Cl, SiO<sub>2</sub> nanoparticles, polyetheramine, Okra mucilage, Choline Chloride:Urea DES, and Citric acid: Glycerine NADES representing wide categories of drilling fluids were studied individually and as combinations for their impact on shale stability.
2. Through comprehensive analysis, it was determined that certain additives exhibited superior performance in shale inhibition compared to others. Among the additives tested, Citric acid:Glycerine NADES showed superior performance by reducing mudcake thickness by 42.8 %, filtrate volume by 40.3 %, and linear swelling by 76.1 %. Moreover, shale recovery improved by 51.7 % compared to the base fluid.
3. SET B (0.5 % KCl + 0.5 % ATPE) and SET G (0.5 % KCl + 0.5 % [EMIM]Cl) significantly enhanced rheological properties, with YP/PV ratios approaching the optimal range. Their performance is attributed to the synergistic effects of KCl's ion exchange and the hydrogen bonding/intercalation capabilities of ATPE or [EMIM]Cl, which together improve cuttings transport and wellbore stability.

## Recommendations

Based on our findings, we recommend further exploration of optimized additive combinations, particularly focusing on the synergistic interactions that lead to improved shale inhibition. In future studies, we propose supplementing the current work with advanced experiments such as adsorption isotherms, SEM analysis for pore blockage, and molecular dynamics simulations to better elucidate the underlying failure mechanisms in underperforming formulations (e.g., SET ABCEF and SET ACG). These additional investigations will provide deeper insights into additive compatibility and optimize drilling fluid formulations for enhanced wellbore stability and operational efficiency. Moreover, future work should include a comprehensive cost-benefit and technoeconomic analysis to evaluate the economic viability of the optimized drilling fluid formulations.

## CRedit authorship contribution statement

**Muhammad Hammad Rasool:** Resources, Methodology, Investigation, Formal analysis, Conceptualization. **Maqsood Ahmad:** Supervision, Funding acquisition. **Numair Ahmed Siddiqui:** Funding acquisition, Formal analysis, Data curation. **Syahrir Ridha:** Investigation, Funding acquisition. **Azam Khan:** Writing – review & editing, Visualization, Validation. **Husnain Ali:** Writing – review & editing, Resources, Project administration.

## Declaration of Competing Interest

The authors do not have any conflict of interest to disclose.

## Acknowledgements

The authors would like to acknowledge YUTP Grant 015LC0-535 for providing financial assistance for this study.

## References

- M.H. Rasool, M. Ahmad, Revolutionizing shale drilling with potassium chloride-based natural deep eutectic solvent as an additive, *J. Pet. Explor. Prod. Technol.* 14 (1) (2024) 85–105.
- M.H. Rasool, M. Ahmad, Understanding Shale Instability through the Lens of Clay Mineralogy and Zeta Potential, *Geol. Earth Mar. Sci.* 5 (2023) 1–10.
- M.A. Abbas, et al., Characterization of nano based drilling fluid for shale swelling inhibition, *Pet. Sci. Technol.* 40 (22) (2022) 2710–2736.
- M.H. Rasool, M. Ahmad, A. Jawaad, N.A. Siddiqui, Perspective chapter: drilling fluid chemistry—tracing the arc from past to present, in: *Exploring the World of Drilling*, IntechOpen, 2024.
- M.H. Rasool, A. Zamir, K.A. Elraies, M. Ahmad, M. Ayoub, M.A. Abbas, Potassium carbonate based deep eutectic solvent (DES) as a potential drilling fluid additive in deep water drilling applications, *Pet. Sci. Technol.* 39 (15–16) (2021) 612–631.
- M. Wilson, L. Wilson, Clay mineralogy and shale instability: an alternative conceptual analysis, *Clay Miner.* 49 (2) (2014) 127–145.
- R. Gholami, H. Elochukwu, N. Fakhari, M. Sarmadivaleh, A review on borehole instability in active shale formations: Interactions, mechanisms and inhibitors, *Earth-Sci. Rev.* 177 (2018) 2–13.
- F. Liu, et al., Investigation of the inhibition mechanism of polymer/nano-silica composite as shale inhibitor in water-based drilling fluids, *Colloids Surf. A: Physicochem. Eng. Asp.* 636 (2022) 128099.
- T.A. Saleh, Advanced trends of shale inhibitors for enhanced properties of water-based drilling fluid, *Upstream Oil Gas Technol.* 8 (2022) 100069.
- S.Q.A. Mahat, I.M. Saaid, A. Sauki, A.A. Aja, N. Ridzuan, N. Ismail, From traditional to green: evolution of shale swelling inhibitors for sustainable drilling, *Malays. J. Anal. Sci.* 28 (2) (2024) 348–364.
- M.H. Rasool, M. Ahmad, N.A. Siddiqui, H. Ali, Novel application of citric acid based natural deep eutectic solvent in drilling fluids for shale swelling prevention, *Sci. Rep.* 14 (1) (2024) 25729.
- A. Khan, A.S.A. Shahid, M.K. Zahoor, Sustainable drilling fluid design; utilizing waste biomass as drilling fluid additive, in: *International Petroleum Technology Conference, IPTC, 2024* p. D021S032R002.
- T.A. Saleh, M.A. Ibrahim, Advances in functionalized Nanoparticles based drilling inhibitors for oil production, *Energy Rep.* 5 (2019) 1293–1304.
- X. Shen, et al., Application of carboxylated cellulose nanocrystals as eco-friendly shale inhibitors in water-based drilling fluids, *Colloids Surf. A: Physicochem. Eng. Asp.* 627 (2021) 127182.
- G. Chen, J. Yan, L. Lili, J. Zhang, X. Gu, H. Song, Preparation and performance of amine-tartaric salt as potential clay swelling inhibitor, *Appl. Clay Sci.* 138 (2017) 12–16.
- J. Zhang, W. Hu, L. Zhang, T. Li, D. Cai, G. Chen, Investigation of ammonium-lauric salt as shale swelling inhibitor and a mechanism study, *Adsorpt. Sci. Technol.* 37 (1–2) (2019) 49–60.
- R. Zhang, et al., Preparation and performance of ammonium-malic salts as shale swelling inhibitor and a mechanism study, *Inorg. Nano-Met. Chem.* 50 (10) (2020) 1027–1031.
- Z. Song, et al., Preparation and application of a novel polyammonium as potent shale hydration inhibitor, *J. Macromol. Sci. Part A* 57 (5) (2020) 326–331.
- W. Du, X. Pu, J. Sun, X. Luo, Y. Zhang, L. Li, Synthesis and evaluation of a novel monomeric amine as sodium montmorillonite swelling inhibitor, *Adsorpt. Sci. Technol.* 36 (1–2) (2018) 655–668.
- X. Zhao, Z. Qiu, Y. Zhang, H. Zhong, W. Huang, Z. Tang, Zwitterionic polymer P (AM-DMC-AMPS) as a low-molecular-weight encapsulator in deepwater drilling fluid, *Appl. Sci.* 7 (6) (2017) 594.
- W. Du, X. Wang, G. Chen, J. Zhang, M. Slaný, Synthesis, property and mechanism analysis of a novel polyhydroxy organic amine shale hydration inhibitor, *Minerals* 10 (2) (2020) 128.
- X. Bai, et al., Preparation and evaluation of amine terminated polyether shale inhibitor for water-based drilling fluid, *SN Appl. Sci.* 1 (2019) 1–9.
- H. Zhong, Z. Qiu, W. Huang, J. Cao, Shale inhibitive properties of polyether diamine in water-based drilling fluid, *J. Pet. Sci. Eng.* 78 (2) (2011) 510–515.
- H. Zhong, Z. Qiu, W. Huang, J. Cao, F. Wang, B. Xie, Inhibition comparison between polyether diamine and quaternary ammonium salt as shale inhibitor in water-based drilling fluid, *Energy Sources Part A: Recov. Util. Environ. Eff.* 35 (3) (2013) 218–225.
- Y. Tian, et al., Study of a polyamine inhibitor used for shale water-based drilling fluid, *ACS Omega* 6 (23) (2021) 15448–15459.
- X. Bai, Y. Xu, X. Zhang, X. Yong, Z. Li, Comparison on the inhibitive properties of different inhibitors in water-based drilling fluid, *Pet. Chem.* 61 (2021) 239–249.
- M.A. Abbas, A. Zamir, K.A. Elraies, S.M. Mahmood, M.H. Rasool, A critical parametric review of polymers as shale inhibitors in water-based drilling fluids, *J. Pet. Sci. Eng.* 204 (2021) 108745.
- H. Li, J.-s. Sun, K.-h. Lv, X.-b. Huang, Amine-terminated acrylamide polymer as a shale inhibitor for water-based drilling fluids, in: *International Field Exploration and Development Conference*, Springer, 2022, pp. 4553–4560.
- T. Zhou, et al., Development and mechanistic study of hydrophobic shale inhibitors for water-based drilling fluids, *J. Dispers. Sci. Technol.* (2023) 1–10.
- M.T. Rahman, B.M. Negash, D.K. Danso, A. Idris, A.A. Elryes, I.A. Umar, Effects of imidazolium-and ammonium-based ionic liquids on clay swelling: experimental and simulation approach, *J. Pet. Explor. Prod. Technol.* (2021) 1–13.
- R.A. Khan, M. Murtaza, H.M. Ahmad, A. Abdurraheem, M.S. Kamal, M. Mahmood, Development of novel shale swelling inhibitors using hydrophobic ionic liquids and gemini surfactants for water-based drilling fluids, in: *SPE Middle East Oil and Gas Show and Conference, SPE, 2021* p. D031S032R008.
- P. Huang, et al., Designing novel high-performance shale inhibitors by optimizing the spacer length of imidazolium-based bola-form ionic liquids, *Energy Fuels* 34 (5) (2020) 5838–5845.
- L. Yang, G. Jiang, Y. Shi, X. Yang, Application of ionic liquid and polymeric ionic liquid as shale hydration inhibitors, *Energy Fuels* 31 (4) (2017) 4308–4317.
- T.N. Ofei, C.B. Bavoh, A.B. Rashidi, Insight into ionic liquid as potential drilling mud additive for high temperature wells, *J. Mol. Liq.* 242 (2017) 931–939.
- Z. Luo, L. Wang, P. Yu, Z. Chen, Experimental study on the application of an ionic liquid as a shale inhibitor and inhibitive mechanism, *Appl. Clay Sci.* 150 (2017) 267–274.
- D. Zhao, Y. Liao, Z. Zhang, Toxicity of ionic liquids, *Clean. Soil Air Water* 35 (1) (2007) 42–48.
- T.P.T. Pham, C.-W. Cho, Y.-S. Yun, Environmental fate and toxicity of ionic liquids: a review, *Water Res.* 44 (2) (2010) 352–372.
- K. Kuroda, A simple overview of toxicity of ionic liquids and designs of biocompatible ionic liquids, *New J. Chem.* 46 (42) (2022) 20047–20052.
- H. Jia, et al., Investigation of inhibition mechanism of three deep eutectic solvents as potential shale inhibitors in water-based drilling fluids, *Fuel* 244 (2019) 403–411.
- M.H. Rasool, et al., Rheological characterization of potassium carbonate deep eutectic solvent (DES) based drilling mud, *J. Pet. Explor. Prod. Technol.* (2022) 1–11.
- J. Ma, S. Pang, W. Zhou, B. Xia, Y. An, Novel deep eutectic solvents for stabilizing clay and inhibiting shale hydration, *Energy Fuels* 35 (9) (2021) 7833–7843.
- M.H. Rasool, M. Ahmad, M.A. Abbas, A double action PD (polymer-deep eutectic solvent) based shale inhibitor in drilling mud, *J. Adv. Res. Fluid Mech. Therm. Sci.* 99 (1) (2022) 149–157.
- L. Benvenuti, A.A.F. Zielinski, S.R.S. Ferreira, Which is the best food emerging solvent: IL, DES or NADES? *Trends Food Sci. Technol.* 90 (2019) 133–146.
- A. Paiva, R. Craveiro, I. Aroso, M. Martins, R.L. Reis, A.R.C. Duarte, Natural deep eutectic solvents—solvents for the 21st century, *ACS Sustain. Chem. Eng.* 2 (5) (2014) 1063–1071.
- M.H. Rasool, M. Ahmad, M. Ayoub, M.A. Abbas, A novel ascorbic acid based natural deep eutectic solvent as a drilling mud additive for shale stabilization, *Processes* 11 (4) (2023) 1135.
- M.H. Rasool, M. Ahmad, N.A. Siddiqui, A.Z. Junejo, Eco-friendly drilling fluid: calcium chloride-based natural deep eutectic solvent (NADES) as an all-rounder additive, *Energies* 16 (14) (2023) 5533.

- [47] M.H. Rasool, M. Ahmad, Epsom Salt-based natural deep eutectic solvent as a drilling fluid additive: a game-changer for shale swelling inhibition, *Molecules* 28 (15) (2023) 5784.
- [48] M. Murtaza, H.M. Ahmad, X. Zhou, D. Al-Shehri, M. Mahmoud, M.S. Kamal, Okra mucilage as environment friendly and non-toxic shale swelling inhibitor in water based drilling fluids., *Fuel* 320 (2022) 123868.
- [49] A.K. Quainoo, B.M. Negash, C.B. Bavoh, T.O. Ganat, B.N. Tackie-Otoo, A perspective on the potential application of bio-inhibitors for shale stabilization during drilling and hydraulic fracturing processes., *J. Nat. Gas. Sci. Eng.* 79 (2020) 103380.
- [50] M.H. Rasool, M. Ahmad, A. Jawaad, N.A. Siddiqui, Perspective Chapter: Drilling Fluid Chemistry—Tracing the Arc from Past to Present, 2024.
- [51] J.-G. Xu, Z.-S. Qiu, X. Zhao, H.-Y. Zhong, G.-R. Li, W.-A. Huang, Synthesis and characterization of shale stabilizer based on polyethylene glycol grafted nano-silica composite in water-based drilling fluids, *J. Pet. Sci. Eng.* 163 (2018) 371–377.
- [52] X. Yang, Z. Shang, H. Liu, J. Cai, G. Jiang, Environmental-friendly salt water mud with nano-SiO<sub>2</sub> in horizontal drilling for shale gas, *J. Pet. Sci. Eng.* 156 (2017) 408–418.
- [53] M.H. Rasool, M. Ahmad, M. Ayoub, A. Zamir, M.A. Abbas, A review of the usage of deep eutectic solvents as shale inhibitors in drilling mud, *J. Mol. Liq.* 361 (2022) 119673.



**Dr. Hammad** is a dedicated Research Scientist with a strong background in drilling engineering, geochemistry, and material characterization. He holds a PhD in Petroleum Geosciences from Universiti Teknologi PETRONAS, Malaysia and two Masters in Petroleum Engineering where his research focused on developing eco-friendly Natural Deep Eutectic Solvents (NADES) for shale stability and hydrates in drilling operations. With expertise in drilling fluid rheology, CO<sub>2</sub> sequestration, and hydrogeological processes, his work spans both applied and fundamental sciences. Hammad has authored multiple peer-reviewed publications, contributed to book chapters, and has experience in academia, having taught chemistry, geosciences, and drilling engineering courses. His research inter-

ests include sustainable energy technologies, nanomaterials, distributed acoustic sensing and subsurface fluid interactions.



**Dr. Maqsood Ahmad** is a Senior Lecturer in the Department of Geoscience at Universiti Teknologi PETRONAS (UTP), Malaysia. He holds multiple master's degrees in Applied Geology (University of the Punjab, Pakistan), Information Technology (Queensland University of Technology, Australia), and Petroleum Engineering (University of New South Wales, Australia). He earned his Ph.D. in Petroleum Engineering from the University of Adelaide, focusing on unconventional gas reservoirs, particularly shale and tight gas. His research interests include unconventional oil and gas resources, geomechanics, hydraulic fracturing, drilling fluids, and petrophysics.



**AP Dr. Numair Ahmed Siddiqui** is an Associate Professor in the Department of Geoscience at Universiti Teknologi PETRONAS (UTP), specializing in field sedimentology, mapping, stratigraphy, petroleum geology, and static modeling. He holds a Ph.D. in Petroleum Geosciences from UTP, an M.Sc. in Petroleum Geoscience from Universiti Brunei Darussalam, and a B.S. in Petroleum and Gas Engineering from BUTEMS, Pakistan. With over nine years of teaching, research, and industry experience, Dr. Numair has expertise in siliciclastic rocks, sequence stratigraphy, Southeast Asian field sedimentology, reservoir characterization, outcrop modeling, and well logging. His research focuses on siliciclastic shallow-marine deposits in the Borneo region, reservoir characterization, and outcrop modeling with analogue studies. Currently, he teaches reservoir characterization and field sedimentology at UTP.



**AP Dr. Syahrir Ridha** is an Associate Professor in the Department of Petroleum Engineering at Universiti Teknologi PETRONAS (UTP) and the Director of the Institute of Sustainable Energy & Resources at UTP. He holds a Ph.D. and a Master's in Petroleum Engineering from UTP and a Bachelor's degree in Mining Engineering from Universitas Islam Bandung, Indonesia. His research expertise lies in drilling technology, well cementing, drilling fluids, extraction of rare earth elements and wellbore integrity. His work focuses on geopolymerization mechanisms for oilwell cementing, microstructure deformation in alternative cement systems during CO<sub>2</sub> flooding, and the application of graphene in drilling fluids. Dr. Syahrir has also contributed to studies on tsunami disaster awareness and the use of geopolymer-based cement in various engineering applications. His significant contributions to petroleum engineering research reflect his commitment to advancing sustainable energy solutions.



**Engr. Azam Khan** is an Assistant Professor at Petroleum and Gas Engineering department at University of Engineering and Technology Lahore, Pakistan with over 22 years of experience in teaching, research, and curriculum development. His expertise spans Drilling Engineering, Formation Evaluation, and Reservoir Engineering, with a research focus on Smart Drilling Muds and unconventional materials like nanoparticles. He has taught various petroleum engineering courses, including Drilling Engineering, Well Logging, Reservoir Simulation, and Properties of Reservoir Fluids. Dedicated to advancing drilling technologies, he continues to contribute to academia and industry through innovative research and education.



**Engr. Husnain Ali** is a PhD scholar in Chemical and Biomolecular Engineering at the Hong Kong University of Science and Technology (HKUST), where he has been studying since 2022. He earned his B.Sc. in Chemical Engineering from the University of Engineering & Technology, Lahore, Pakistan, in 2019, graduating first in his class and receiving a Gold Medal. He then completed an M.Sc. by Research in Chemical Engineering at Universiti Teknologi PETRONAS, Malaysia, in 2022. Husnain's research interests encompass Data Science, Process System Engineering, process monitoring and control, polymer processing, injection molding, process safety, and the application of multivariate statistical methods for industrial monitoring and diagnostics in chemical process systems.